Roll-to-Roll Embedding of Conductive Sintered Silver Grids

Ashish Kumar Pagudala

Master of Science Thesis
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**Abbreviations used:**

S2S – Sheet-to-sheet

R2R – Roll-to-roll

OLED – Organic light emitting diode

AMOLED – Active matrix organic light emitting diode

OPV – Organic Photovoltaic’s

UV – Ultra violet

SEM – Scanning electron microscopy

HOMO – Highest occupied molecular orbital

LUMO – Lowest unoccupied molecular orbital

ITO – tin doped indium oxide

PEN – Polyethylene naphthalate

PET – Polyethylene terephthalate

LED – Light emitting diode

NIL – Nano imprint lithography

PMOS- P-channel metal oxide field effect transistor

NMOS- N-channel metal oxide field effect transistor

Fig- Figure

RFID- Radio frequency distribution

LCD- liquid crystal display

CNT- Carbon nano tubes

EDX- Energy dispersive X ray spectroscopy

RUVNIL- Reverse ultraviolet nano imprint lithography

AFM- Atomic force microscopy

kV- Kilo volts
RPM- Revolutions per minute

$J_{sc}$- Short circuit current density

$V_{OC}$- Voltage at open circuit

FF%- Fill factor in %

$\eta$%- Efficiency in %
Aknowledgements

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Abstract:

R2R embedding of conductive sintered grids:

Within the organic light emitting diode (OLED) and organic photovoltaic (OPV) applications, to improve the efficiency of these devices, high conductive metal grids are required to replace tin-doped indium oxide (ITO) as an anode. High conductive metal grid, can be obtained by screen printing of the metal shunt lines which are several microns thick. In order to avoid the risk of electrical shorts, the height of the metal shunt lines should be minimized as much as possible, so that other active layers can be coated above it. But, the surface topology of the metal shunt lines greatly influences the active layers coated above it. New concept has been developed to address the problem of surface topology at Holst Centre.

New concept developed constitutes of embedding of the screen printed silver conductive sintered grids in the ultra violet (UV) sensitive transparent resist, which is cured on the polyethylene naphthalate (PEN) foil using the roll-to-roll (R2R) process. By doing so, we obtain the metal shunt lines in combination with the resist having more or less a flat surface profile. Characterization methods namely scanning electron microscopy (SEM), dektak profilometry, atomic force microscopy (AFM), optical interferometry and confocal microscopy measurements were performed on the samples.

Surface profile measurements were performed on the screen printed silver shunt lines and also on the embedded shunt lines. Height of the screen printed silver tracks was approximately 1.2μm, and of the embedded shunt lines was approximately 130 nm, which means that the percentage of the embedding of shunt lines is approximately 93%. R2R embedded shunt lines were integrated successfully into an organic light emitting diode (OLED) device.
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Chapter 1: Introduction.

1.1 Organic Electronics:

A major breakthrough in Organic Electronics came when Heeger, MacDiarmid and Shirakawa discovered and developed conductive polymers in the year 1977. Their efforts credited them with the Nobel Prize in the year 2000.1

For the past four decades, inorganic semi-conducting materials like silicon, gallium arsenide and insulators such as silicon dioxide have taken up the lead role in the electronics industry. From the recent past, research in the organic electronics domain is growing tremendously due to improvement of the conducting and semi-conducting properties of organic materials (oligomers, polymers) and hybrids like composite of organics and inorganics.3 Organic electronics has unique ability, where device processing can be done at a higher speed and potential to be mechanically flexible. Active layers can be processed over large areas on materials like plastics and papers, etc.

Resists and polymeric insulators are the two main passive organic materials which made it possible to reduce the cost and increase the performance. Recently, it was demonstrated that the use of polymer with a low dielectric constant instead of conventional oxide insulators, thereby improving overall wiring interconnect performance by approximately 37%.3 Organic materials have also provided improved reliability and performance for storage products. The ability of chemists to really tune the properties of organic materials has contributed to the growth in the electronics industry.3 In display technologies, the market share of organic electronics is increasing slowly. Active matrix organic light emitting diode (AMOLED) displays are replacing the conventional displays like liquid crystal displays (LCDs), etc. in mobile applications due to their wide viewing angle, efficiency and low cost.

After the first organic field effect transistor (FET) was introduced in 1986, a new era of electronics started i.e organic electronics. Even though the performances of organic devices are far lower than the conventional silicon devices, organic electronics can be used in the area which does not demand for high performance of a device, for example: radio frequency identification tags (RFID tags), sensors, etc. The advantage of organic electronics over silicon devices is that it can be manufactured at a lower cost using large-area printing technology.

The introduction of organic semiconductors has opened the way for fully printing of RFID tags. It will possibly, be a cheap production process from both an investment and a production point of view. In this methodology, an additive printing process is employed to make devices. Lithography and etching steps are eliminated by this method. p-channel metal oxide semiconductor field effect transistor (PMOS), n-channel metal oxide semiconductor field effect transistor (NMOS) and high Q passive components were also realized using printing technology of nanoparticle inks, organic semiconductors and insulators.4

Organic electronics is slowly making its way in the commercial market of electronics industry by providing the first electronic devices.
1.2 OLED (Organic light emitting diode):

An OLED is a solid-state semiconductor device made of carbon-based material that emits light when the current is applied across its electrodes. From the below figure 1.2 its principle of operation can be explained as follows:

When the current is applied across OLED electrodes, cathode pushes electrons into the emissive layer, while the anode removes electrons from the emissive layer, leading to the formation of holes in the conductive layer creating excitons.\(^5\)

The process of emission of light in an OLED can be classified in four steps as follows:

- Injection of holes and electrons into a device by supplying current to it.
- Transport of charge carriers.
- Exciton formation.
- Recombination of excitons under the emission of light.

![Fig 1.2) OLED fabricated with stack of different materials.\(^5\)](image)

1.3 Organic Photovoltaic (OPV):

The basic principle of organic solar cells is that excitons are formed in the active layer (organic layer) of OPV, when it absorbs light. These excitons can be referred to as tightly bound electron-hole pairs. Due to the strong binding energy between electrons and holes, excitons can be considered as a mobile excited state. These generated excitons will split into electrons and holes in order to generate current. At first organic photovoltaics were made of only one organic layer sandwiched between two electrodes. So, in order to overcome the binding energy of exciton, the following processes have to be facilitated:\(^5\):

1) To supply with higher value of thermal energy \(k_BT\).
2) Dissociation at metal contacts.
3) Higher degree of intrinsic electric field.

But unfortunately, all the above processes are not very efficient. Under ambient conditions, the temperature is not high enough and the active layer thickness will be usually higher than the exciton diffusion length \( L_D \). Furthermore, built-in-electric fields are too weak to split the strongly bound excitons.\(^8\) Thus, the largest part of the excitons recombine instead of dissociating.\(^9\) In order to overcome this, a new type of solar cell design has been proposed, which consists of two layers of different organic materials and is termed as hetero-junction solar cell. One is an electron donor, and other one is an electron acceptor.\(^8\)

In this hetero-junction solar cells, light is usually absorbed by the donor material (made of hole conducting small molecules). Now, photo generated excitons, can diffuse within the donor material to the interface between donor and acceptor material (relatively more electronegative). If the energy difference between the ionization potential of the \((I_{PD})\) of the electron donating material and electron affinity \((EA_A)\) of the electron accepting material is higher than the exciton binding energy \((E_B)\), exciton dissociation is energetically favorable.\(^8\) Below figures 1.3(a, b and c) illustrates the mechanism of generation of holes and electrons in the hetero junction organic solar cells.

\[ IP^{*}_{D} - EA_A - E_B < 0 \] (Condition for exciton dissociation)

Fig 1.3: a) b) c)

Fig 1.3a, b and c) The above figure illustrates the principle of organic photovoltaic: a) Shows the generation of an exciton when light is absorbed by the organic layer. b) Exciton dissociated at the interface of electron donor (Organic p-conductor) and electron acceptor materials (Organic n-conductor). c) Splitting and collection of charges at their respective electrodes.\(^8\)

Below fig 1.3d is the band diagram at donor-acceptor interface of the OPV, donor refers to the absorber which is in fig 13.a) and acceptor refers to the organic n-conductor which is in fig 1.3b.
Fig 1.3d) Schematic illustration of exciton dissociation at DA- interface. (1) Highest occupied molecular orbital of electron donor (HOMO$_D$) and lowest unoccupied molecular orbital of electron donor (LUMO$_D$) are the levels of not excited donor molecule. (2) An excited donor molecule is ionized by energy $IP_{D^*}$. (3) The electron is transferred to the LUMO of an electron acceptor by gaining energy $EA_A$. (4) Due to the Coulomb interaction between the electron on the acceptor and the remaining hole on the ionized donor an additional energy barrier $E_B$ has to be overcome for the spatial separation of the charge carriers. Thus exciton dissociation is energetically favorable if $IP_{D^*} - EA_A - E_B < 0$ (Condition for exciton dissociation).

1.4 Large area printing:

Improving the efficiency of OLED and OPV is a central goal for organic electronics research. There is a huge demand to further improve the techniques which are used for OLED and OPV fabrication. Many improvements are still necessary, to lower the fabrication costs. One possibility is implementing a roll-to-roll process. Some research institutes are focusing on in-depth studies of issues and parameters, which affect the roll-to-roll printing of organic electronics.

Typically, Indium tin oxide (ITO) is used as the anode material for OLED/OPV applications because of its transparency in the visible range of electromagnetic spectrum and also due to its electrical conductivity. But its conductivity is in the range of $1.5-5*10^3 \ \Omega^{-1} \ \text{cm}^{-1}$ which corresponds to sheet resistance of 20-80\Omega/square. Such high resistance limits the performance of an OLED device due to voltage drop along the long conducting ITO leads.\textsuperscript{18} Besides its high sheet resistance, ITO is brittle, scarcity of Indium which increases the cost, narrow process window, Indium migration into the active layers after lengthy operations (in OLED) which cause the device to deteriorate, absorbance of water and etch of ITO due to its acidic characteristics.\textsuperscript{19,20}

A possible alternative for ITO might be the use of metals with higher conductivity and higher work function in OLED devices.\textsuperscript{18} In this project, silver is used as a replacement for ITO in both OLED and OPV applications. An advantage of using silver is its high electrical conductivity.\textsuperscript{18}

There is a growing demand for R2R production of large area flexible OLEDs for lighting applications. ITO (conductive electrode) is a factor of concern for the commercialization of OLED technology because of difficulty in deposition and patterning of large area flexible OLEDs for cost effective production. Alternative solution for this is usage of large area printed conductive silver grids
(conductive electrodes) which can be deposited and cured in air unlike ITO which has to be deposited in inert conditions. Silver grids provide solution for low cost fabrication of R2R large area OLED devices instead of ITO.7

Thin film devices like OPV and OLED have a particular advantage of being made on flexible and cheap substrates. In both the cases anode plays, an important role and its fabrication is of high importance. For OPV, it is used for current collection and in OLED, it is used for emission of the light. So fabrication of the anode, in both the cases is of high importance. It has to be designed and fabricated carefully, so that overall efficiency of the device increases. Below is the figure (1.4a) showing the current collecting grid which is free from ITO. It consists of screen printed silver grid on the foil as anode for OPV.

![Figure 1.4a) 2*2 cm² with current collecting grid free from ITO.](image)

The alternative solution, for this is the use of printed conductive structures. The important factor which has to be considered is the conductivity of the shunting lines, which should be as close as to the bulk material properties as possible. The height of shunting lines should be minimized as much as possible, so that other layers above it can be coated homogenously. The risk of creating electrical shorts is minimized if the height of the shunting lines is reduced. In order to solve this problem embedded shunt lines can be used. Below figures 1.4(b and c) are the schematics of OPVs with ITO and embedded shunt lines as anode.

![Figures 1.4b and c) Schematic view of ITO based and embedded shunt line OPVs.](image)

From the above figures 1.4b and c, it can be observed that the main difference between ITO based device and embedded shunt lines based device is that, in ITO based device ITO is coated above the barrier layer
whereas in embedded shunt lines based device, the silver tracks are embedded in the barrier layer, thus reducing the overall thickness of the device by 100nm approximately.

Below is the table (1.4) comparing the characteristics between ITO based (anode) and embedded shunt line (anode) based OPVs.

<table>
<thead>
<tr>
<th>Anode</th>
<th>$J_{sc}$ (mA/cm$^2$)</th>
<th>$V_{OC}$ (Volt)</th>
<th>FF (%)</th>
<th>$\eta$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ITO</td>
<td>6.59</td>
<td>0.489</td>
<td>29.60</td>
<td>0.95</td>
</tr>
<tr>
<td>Silver lines/HC PEDOT</td>
<td>6.25</td>
<td>0.540</td>
<td>57.10</td>
<td>1.93</td>
</tr>
</tbody>
</table>

Table 1.4) Comparison of different parameters of the OPVs using ITO and silver as anode.$^{21}$

From the above table 1.4 we can infer that by replacing ITO with high conductive embedded silver metal grids in combination with high conductive Poly(3,4-ethylenedioxythiophene) (PEDOT), there is a significant improvement in the performance of OPV.

$J_{sc}$: Short circuit current density.

Open circuit voltage ($V_{OC}$): It is the potential which is developed across the terminals when the external load resistance is very large.

Fill factor (FF): It is defined as the ratio of maximum power ($P_{max}$) at finite load resistance to the product of $V_{OC}$ and short circuit current ($I_{SC}$).

$$FF = \frac{P_{max}}{V_{OC} \times I_{SC}}$$

Short circuit current ($I_{SC}$): Current which flows with zero external resistance.

Efficiency ($\eta$ %): It is the ratio of electrical power delivered to the load to the optical power incident of the cell.

The main objectives of organic electronics are to be able to produce low-cost and stable devices. It can be achieved by using low-cost materials (cheap and inexpensive substrates and, etc.) in combination with fast roll-to-roll process technologies. Applications based on organic semiconductors have strict requirements of layer thickness and uniformity, etc. due to their material properties. OPV ink for fabricating OPV at large scale using roll-to-roll processing was being formulated.$^2$

Typical methods which include patterning on substrates are micro-contact printing, ink-jet printing, offset printing and screen printing. All these patterning/printing methods can be employed on inexpensive large area substrates like metal, glass, and plastic, etc.$^{10}$

The main challenge which exists for using large-area printing is the design of a set of chemical materials compatible with large-area printing. At present, strong research focus is on improving the
Material properties; so that it can be compatible to these printing methods. Du-Pont developed a technique called thermal imaging, which allows the patterning of a range of organic materials on large areas with micron scale resolutions at high speed.

Ink-jet printing can be used to pattern active materials or to etch masks in order to obtain a desired pattern. There are many advantages of ink-jet printing technology, they are (i) the patterning process originates from the electronic image file which is not fixed to the rigid substrate and can be used to align the flexible substrates, (ii) it is a non-contact process, (iii) layer to layer registration is accompanied by digital image processing. Due to these advantages, ink jet printing is considered as one of the candidates to be applied as the main technology for industrial large area printing. Replacing the conventional patterning process by digital lithography is the first step in processing TFTs.

It was recently demonstrated that using screen printing technology, wireless sensors can be made on flexible substrates. The best part of screen printing and ink-jet printing is that they do not require any wet or dry etching to pattern the substrates. If this is extended to roll-to-roll printing, there is the possibility to produce some low-cost flexible wireless sensors. Below are the images (1.4d and 1.4e) of the wireless sensors made on foil.

Fig 1.4d) Sensor units fabricated on a PET film via screen printing and via interconnection. A 4*7 array of sensors fabricated on 15*15 cms of PET foil. Fig 1.4e) Close up view of one sensor.

Fabrication of high mobility plastic transistors through screen printing has been demonstrated. They used ITO coated PET foil (commercially available) as substrate and ITO acts as a gate electrode. On the other side of the PET foil, a pair of electrodes was printed and the fabrication of the transistor was finished using screen printing.

Experimentation was done to utilize the properties of silicon and apply it by screen printing to attain low fabrication costs. The material used to produce a diode in this novel approach was silicon powder. In the near future, this concept has a huge potential to make devices using silicon powder on flexible substrates at very low cost with the help of roll-to-roll screen printing technology.

Some attempts were also made to print carbon nano tubes on polyester sheets, so that it can be used as transparent electrodes replacing ITO. The basic idea used in screen printing of CNTs is to use multi walled carbon nano tubes dispersed in polymers, so that they can be screen printed. The most interesting part of this attempt was that thermal shock measurements on both ITO samples, and CNT samples revealed a minimal change in sheet resistance, while for ITO samples sheet resistances increased.
drastically for about five times.\textsuperscript{17} Below figure 1.4f is the TEM image of the multi-walled carbon nanotubes.

Fig 1.4f) TEM image of MWCNT’s (Multi walled carbon nanotubes).\textsuperscript{17}

From all the above experimentations and attempts in various research institutes across the world using printing technology, it can be inferred that by printing technology, devices can be made not only in an efficient way without using any etching steps but also can be implemented on a large scale production using roll-to-roll process. Thus, there is a huge potential for this printing technology to be applied for different materials and very low fabrication costs can be achieved by focusing on large-area printing.

In this report, screen printing technology is used and the process of screen printing is explained in chapter 3.

1.5 Embedding Technology:

A new highly promising technique named ‘\textit{Embedded shunt lines}’ is developed within Holst Centre to increase the efficiency of ITO-free OPV/OLED anode manufacturing.

In this project an R2R approach is demonstrated, and is termed as ‘\textit{Embedded shunt lines}’. This method will be discussed detailed in the chapter 2.

Electrode fabrication can be done in many ways. Here in this project an attempt is made to use the combination of printing and imprinting together, to increase the efficiency of OLEDs and OPV cells.

Typically shunting lines can be deposited with the different techniques like electroplating, evaporation, sputtering and patterning, achieved using shadow masks or photolithography. By using shadow mask patterning it is quite difficult to achieve the necessary heights of the shunting lines of tens of $\mu$m range, since this will require long sputtering times. By embedding technology necessary height of the shunting lines of tens of $\mu$m can be deposited with very less time.\textsuperscript{6}

1.6 Importance and objectives of the present work:

The important motive of this research work are (i) to develop a R2R embedding technology which is compatible to OLED and OPV device processing.
The objective of this work is to understand the process of embedding shunt lines. To approach this project goal, experimental work was started on S2S scale. Various parameters affecting the S2S process will be studied and optimized. The influence of the surface energy of the substrate, the metal and the resist will be studied. Experiments with different resists, substrates like polyethylene naphthalate(PEN), polyethylene terephthalate(PET) etc. will be carried out and the quality of embedding is analyzed. Different types of inks such as silver nano/ micro particle ink pastes and a carbon paste will be tested for their potential to produce embedded shunt lines. Furthermore, possible solution to silver migration in OLED devices is also addressed.

After optimization of the S2S process, the concept of embedding shunt lines will be transferred to a R2R set-up. Additional parameters, affecting the process of embedding will be analyzed and optimized in the R2R process. Surface profile measurements were done on the sample using various profilometry techniques.
References:


Chapter 2: Embedding/Imprinting methods.

2.1 Introduction.

There are different types of imprinting techniques available at nano scale such as ‘Nano imprint lithography’ (NIL) and reverse nano imprint lithography (RNIL), etc. Nano imprint lithography can be classified in three categories: (1) Hot embossing nano imprint lithography (HE-NL), (2) UV type nano imprint lithography and (3) soft lithography. This technique is applied in the fabrication of sensors, data storage devices and organic electronics, etc.\textsuperscript{1,2}

The basic concept of nano imprint lithography (NIL) is to make structures by pressing the mold in the resist, which is spin coated on the substrate. The structures which are made by pressing the mold can be used further to fabricate devices. The only difference between NIL and reverse NIL is the mold. In NIL, structures are made within the mold but in reverse NIL, structures are made on the mold using metals. Below figures 2.1(a, b and c) shows the schematic illustration of reverse NIL.

![Figures 2.1a, b and c) Before RUVNIL (reverse ultra violet nano imprint lithography), during the process of RUVNIL and after RUVNIL.\textsuperscript{3}](image)

Before starting the process of the RUVNIL, the resist is spread uniformly on the substrate by spin coating or by droplet dispensing the resist (figure 2.1a). Once the resist is spread uniformly on the substrate, mold is pressed against the substrate (figure 2.1b), while the resist is sandwiched in between them and then the resist is allowed to cure under the UV light. Once the resist is cured completely the mold is released against the substrate, while leaving the negative replica of the mold in the resist (figure 2.1c).

In this research work, method similar to reverse ultraviolet nano imprint lithography (RUvNIL) is used, and it is termed as ‘Embedding shunt lines’. The main difference between RUvNIL and embedding shunt lines is that in the RUvNIL, metal structures on the mold are used to pattern the UV resist, but in the embedding shunt lines, metal structures are directly transferred into the UV resist. Either the mold or acceptor substrate or both should be transparent.

The main advantages of using this embedding technology are roll-to-roll compatibility, low material cost and scalability, when compared to the other methods like sputtering and evaporating. The
acceptor substrate in combination with the high conductive PEDOT can be used, when compared with the combination of expensive tin doped indium oxide (ITO) and low conductive PEDOT. Combining the technologies of printing and embedding makes this ‘embedding shunt lines process’ time effective and more importantly it does not constitute any conventional lithographic steps.

Using the concept of embedding shunt lines, an acceptor substrate in combination with electrode (typically anode) which has more or less flat surface profile is obtained. This acceptor substrate can be used for roll-to-roll coating of PEDOT and other organic layers to complete the device processing using slot die coating, etc. Other applications of embedded shunt lines include organic devices like optical electric devices, which require a high degree of conductivity and transparency.¹

2.2. Embedding shunt lines.

2.2.1. Sheet-to-sheet (S2S) embedding of shunt lines:

Metal structures are obtained on the mold using screen printing technology, and it is termed as ‘donor substrate’. Structures from the donor substrate are transferred into the resist, which is droplet dispersed on another substrate, and this substrate is termed as ‘acceptor substrate’.

In the S2S process, a resist is droplet dispersed on the donor substrate, and then the acceptor is pressed against resist and sealed in a vacuum bag. Vacuum bag helps in spreading the resist uniformly. The whole setup is placed under UV lamp for UV curing. After the resist is cured completely, Vacuum bag is opened, and the donor substrate is delaminated from the acceptor substrate, leaving the screen printed silver tracks embedded in the cured resist. In this method, both acceptor substrate and donor substrate is the PEN foil. Below figure 2.2.2 (a, b and c) are the schematics of S2S process of embedding shunt lines.

![Fig a, b and c: S2S embedding process](image)

**Fig 2.2. a, b and c) S2S embedding process:** a) a resist is droplet dispersed on the donor substrate. b) An acceptor substrate pressed against the donor substrate substrate, while resist is sandwiched in between them and cured. c) An Acceptor substrate with embedded shunt lines is delaminated from the donor substrate.

2.2.2. Roll-to-roll embedding of shunt lines:

The R2R process of embedding shunt lines can be explained clearly from the figure 2.2d. The acceptor substrate moves in the forward direction, while the resist dispenser (D) dispenses a resist on the acceptor substrate. When the acceptor substrate is on the roller 1, it comes in contact with the donor
substrate placed on the drum. Then, the acceptor substrate is pressed against the donor substrate, while a resist is sandwiched between them. This whole setup gets UV treatment when it is exactly above the UV setup (U). When the acceptor substrate and donor substrate reaches the roller 2, the donor substrate is delaminated from the acceptor substrate, leaving the screen printed silver tracks embedded in the cured resist, which is on the acceptor substrate. Thus in this way the embedding of shunt lines works on the R2R tool. All the three rollers are synchronized with each other, rotated at a speed of 0.1 metre/minute (typical settings). Rollers 1 and 2 are rotated in the opposite direction to the main imprint drum.

Main setup of the R2R tool:

Fig 2.2d) R2R imprint setup: D is the resist dispenser; acceptor substrate(C) placed on the web of the tool; M is the main imprint drum; two rollers 1 and 2 which are very important to vary the resist thickness; donor substrate is placed on the main imprint drum and UV curing setup (U).

Dancer pressures (operated by the hydraulic pressure) are present on either side of the rollers 1 and 2 which are responsible for tuning the tension in the web (where the foil is present). The un-winder is present on the side of roller 1 which unwinds the foil, after embedding process is completed; at the other side (side of the roller 2), the re-winder is present, which is responsible for collecting the foil which has embedded tracks in it.

2.3 Resist shrinkage:

Resist shrinkage is one of the important factors, which is to be considered for embedded shunt lines technology. The flatness of the surface profile after embedding shunt lines depends on the resist shrinkage. Resist shrinkage of any resist depends on its chemical properties. If shrinkage of the resist is more, then quality of embedded shunt lines will be very poor. This might be one of the limitations for this process of embedding due to which; it is very difficult to get high quality of embedded shunt lines.
Reference:


Chapter 3: Sample preparation.

3.1 Preparation of the donor substrate and the acceptor substrate:

DuPont Q65FA, thickness 125µm PEN foil, is used for screen printing the donor substrate and is also used as an acceptor substrate. The PEN foil has two sides; one is an adhesive side (smooth side is used as the acceptor substrate) and other one is an anti-adhesive side (rough side used for preparing the donor substrate). The below figure 3.1a, shows the microscopic image of screen printed InkTec-PA-010 silver ink on the PEN foil. The rough surface topology of the ink after sintering is due to escape of solvents during sintering process.

Figure 3.1a) Screen printed Ink Tec 010 silver nano ink, on the PEN foil sintered at 130\degree C for 20 minutes.

   (i) Silver Ink:

A silver ink (InkTec-PA-010) is used for screen printing on the donor substrate. The InkTec ink consists of nano particles with a size distribution around 80 -100nm. The of ink are an organic silver compound which is less than 50% by weight, silver powder less than 45% by weight and a-terpineol which is less than 5% by weight.

SEM measurements of the sintered silver nano ink (Ink Tec 010) are as follows:

Fig 3.1b) Top view of the sintered silver nano ink.

Fig 3.1c) Side view of the sintered silver nano ink.
From the above figures 3.1b and 3.1c, it can be clearly observed that after oven sintering, the silver nanoparticles are sintered well by re-orienting their grain boundaries.

(ii) Screen printing of Silver ink:

A DEK Horizon 03i flat-bed screen print tool is used to screen print silver ink on the donor substrate. The PEN foil with its anti-adhesive side is taken, wiped with isopropanol and dried completely, before printing the silver material. The reason for pre-treatment of the substrate is to remove any dust particles and to define a uniform surface energy throughout the surface.

Working principle:

From the figure 3.1d, it can be explained that the PEN foil is positioned on a vacuum chuck, as a support for screen printing, exactly under the design present on the mesh. Silver ink is placed on the top side of the design with the help of syringe. Based on the settings of force and speed, squeegees move accordingly along the direction of the design present on the mesh, pushing the silver ink through the screen. After this process is completed, it is observed that the silver ink is deposited on the PEN foil, and the design on it resembles the negative replica of the mesh design.

The screen printed PEN foil is placed in an oven at a temperature of $130^\circ C$ for 15 minutes, to remove the solvents present in the silver ink and to sinter the nanoparticles. Dektak profilometry measurements were done on this donor substrate to develop a reference.

3.2 UV resists:

In this research work, UV resists are used because of its compatibility with the embedded shunt lines technology. Low viscosity, less resist shrinkage value and transparency are few critical parameters, which are necessary for this technology.

A UV curable solvent free resist, mr-UVCur21sf is used as a base line resist. Density and viscosity of this resist are $1.089 \text{ g.cm}^{-3}$ and $30 \text{ mPa.s}$ respectively. (Technical data sheet can be found in the appendix#).

Sartomer customized resist (Irga 819): Six different types of resins were mixed together to make this customized resist. Different types of resins used are as follows:
1) CN991: 30% (Low viscous oligomer).
2) CN963B80: 10% (barrier property (protection from water and oxygen)).
3) SR341: 15% (Adhesion and decrease of viscosity).
4) SR285: 15% (Adhesion and decrease of viscosity).
5) SR420: 10% (Adhesion and low surface tension).
6) SR833S: 15% (Scratch resistance and barrier property).
7) Irgacure 819: 5% (Photo initiator from BSAF).

Density and viscosity of customized resist are 1.07752 g.cm$^{-3}$ and 42.4282 mPa.s respectively.

The above resist is also a solvent free resist. It is observed that customized resist takes 40 seconds on the S2S imprint setup, when compared to mr-UVcur21sf which takes around 90 to 100 seconds to cure. From the above data, it is clear that the selection of resist play a very important role in the process of embedding shunt lines. For example: difference in curing time between mr-UVcur21sf resist and customized resist.

3.3 UV lamp:

**For sheet to sheet embedding:**

In this project different kinds of UV setups were used for S2S and R2R embedding.

For sheet to sheet embedding, Delolux 03 S is used (Technical data sheet is attached). Energy received per cm$^2$ is calculated using a sensor which had an aperture of 9.5mm. Around 72 mW of power was recorded by the sensor (in the UV range) with its aperture being 9.5 mm. So the total intensity is

\[ 72\text{mW}/((1/4)*\pi*(9.5\text{mm})^2)=101\text{ mW/cm}^2. \]

Total energy (mJ/cm$^2$) = Total intensity (mW/cm$^2$)*curing time in s.

Hence total energy is= 101(mW/cm$^2$)*90(s) = 9090 mJ/cm$^2$.

**For R2R tool:**

On the R2R tool, a set of UV LED’s are fixed exactly under the main imprint drum. Pro light PG1C-3LLx 3W UV power LED is used.

The measured total energy reaching the sample during curing is approx 1680 mJ/cm$^2$. (Technical datasheet can be found in the appendix#).
Chapter 4: Measurements, analysis and results.

In order to understand and analyze the concept of embedding shunt lines various measurement techniques were used for characterization. mr-UVCur21sf resist is used as a base line resist for embedding shunt lines for both S2S and R2R process.

4.1 S2S embedded shunt lines:

In order to analyze the quality of embedded shunt lines, measurement techniques namely profilometry, confocal microscopy and SEM were used.

4.1.1. Embedding analysis.

4.1.1.1. Profilometry measurements:

A dektak profilometer is used to quantify the surface roughness. It operates by lightly dragging the stylus above the sample according to the user programmed scan length, speed and stylus force. As the stylus rides over the sample, the surface variations on the sample cause the stylus to move in the vertical direction. This vertical movement of the stylus is represented in the form of graph using a software. Radius of the stylus used for measurement is 12.5µm; force used by stylus is 3mg-10mg.

Definitions of few important terms which are used to analyze the results are as follows:

**Average peak height**: It is the average of peak heights measured at three different points on the screen printed silver track. It is measured in nanometers (nm).

**Trench depth** (height \(h_{trench-depth}\)): It is the depth of trench measured in the resist layer after etching the silver which was embedded in it. Trench depth measurement indicates the actual height of the silver embedded in the resist. It is measured in nanometers (nm).

**Height of the silver track above resist** (height \(h_{silver-resist}\)): It is the height of the silver track which is sticking out of the resist layer after embedding the silver in the resist. It is measured in nanometers (nm).

**Percentage of embedding (%)**: It is the ratio of depth of the trench measured in the resist layer after etching the silver which was embedded in the resist (height \(h_{trench-depth}\)) to the height of the silver track which is sticking out of the resist layer after embedding the silver track in the resist.

\[
\text{Percentage of embedding} \% = \left(\frac{h_{trench-depth}}{h_{silver-resist} + h_{trench-depth}}\right)\times 100
\]

For Embedded samples, percentage (%) of embedding is calculated by measuring the surface profile after embedding silver tracks in the resist and then etching the silver tracks using selective etching of the silver, resulting in the formation of trenches in the resist. After this, trench depth is measured to get the percentage of embedding. The below graphs (4.1.1a and 4.1.1b) represent embedding analysis of S2S process.
Average peak height and width of the screen printed silver is 3000 nm and 198\(\mu\m\) (represented by black line in fig 4.1.1.1a). From the above graph it can be observed that surface profile of the screen printed silver track is very rough which is due to the evaporation of the solvents when silver ink is sintered in an oven.

Average peak height of the screen printed silver after embedding is around 180 nm (represented by blue line in the graph 4.1.1.1b), the surface profile after embedding silver is relatively very smooth when compared to the compared to the screen printed silver on the PEN foil (donor substrate). This is because the base part of silver on the PEN foil (donor substrate) is smooth, after embedding in the resist it becomes the top part of the embedded sample. The rough part of the silver which is actually the top part on the PEN foil (donor substrate) is embedded in the resist after embedding process.

Height of the silver embedded in the resist is measured by a trench after etching the silver from the resist and it is around 1800 nm (represented by red line in the graph 4.1.1.1b). Percentage of the silver embedded in the resist is 91% approximately.

### 4.1.1.2 Confocal microscopy measurements:

Confocal microscopy is used to extract more information regarding the surface profile of samples by generating 3D images of it.

Basic principle: Confocal microscopy uses a point illumination and pinhole in an optically conjugate plane front of the detector to eliminate out of focus information. It detects only the light within the focal plane so that, the image quality is better than wide-field imaging. Thickness of the focal plane is defined by square of the numerical aperture of the objective lens, the optical properties of the specimen and ambient index of refraction.\(^1\) Below are the measurement results using confocal microscopy for S2S embedded samples.
Donor substrate Measurements are as follows:

From the above figure 4.1.1.2a it is clear that the screen printed silver on the PEN foil (donor substrate), has very rough surface topology which cannot be seen clearly using profilometer due to 2D graphs generated by it. Approximate height of the screen printed silver track on the PEN foil is around 3000nm. Using dektak profilometry it is also difficult to observe the surface of the PEN foil or the resist layer. Due these limitations of dektak profilometry, confocal microscopy measurements are preferred. Rough surface topology is due to evaporation of solvents, when the screen printed silver is heated in an oven.

From the above figure 4.1.1.2a it is clear that the screen printed silver on the PEN foil (donor substrate), has very rough surface topology which cannot be seen clearly using profilometer due to 2D graphs generated by it. Approximate height of the screen printed silver track on the PEN foil is around 3000nm. Using dektak profilometry it is also difficult to observe the surface of the PEN foil or the resist layer. Due these limitations of dektak profilometry, confocal microscopy measurements are preferred. Rough surface topology is due to evaporation of solvents, when the screen printed silver is heated in an oven.

Fig 4.1.1.2a) Screen printed silver track on PEN foil 50x, 3D view. Height of the silver track is 3000nm approximately.

Fig 4.1.1.2b) Line scan of the screen printed silver track on the PEN foil. Height of the silver track is 3000nm approximately.

Fig 4.1.1.2c) 50X, 3D view S2S embedded silver track in the resist. Height of the silver track sticking outside the resist is 170 nm approximately.

Fig 4.1.1.2d) Line scan of embedded silver track in the resist. Height of the silver track sticking outside the resist is 170nm approximately.
It is observed from the above figure (4.1.1.2c) that, after embedding the silver tracks in the cured resist; bottom part of the screen printed silver on the PEN which is very smooth, now becomes the top part of an embedded sample. Due to this reason, we see that the surface topology of silver track in the embedded sample is very smooth. Resist layer is not flat; this might be because of resist shrinkage, while curing of resist.

4.1.1.3. SEM Measurements:

After analyzing the surface profile of the embedded sample, for better understanding at the cross-section of the embedded sample, SEM measurements were done. Accelerating voltage used is 10kV, source: secondary electron imaging is used. SEM measurement technique was used because cross-section of the embedded sample cannot be analyzed using profilometry and confocal microscopy measurement techniques.

For sheet to sheet imprinting:

![Cross-section view of embedded silver in the resist using S2S embedding process, 250x, accelerating voltage 10 kV.](image1)

![Cross-section view of embedded silver in the resist using S2S embedding process, 1000x, accelerating voltage 10 kV.](image2)

From the above fig 4.1.1.3a, it can be inferred that the thickness is of the PEN foil (acceptor substrate) is 135µm and 8.32µm is of the resist thickness. From the fig 4.1.1.3b) it is observed that the silver track embedded in the resist by 1.74µm and the rough side of silver is now facing downwards into the resist which is actually the top part of the screen printed silver on the PEN foil.
4.2. R2R embedded shunt lines.

4.2.1. Embedding analysis.

Profilometer measurements:

Average peak height of the screen printed silver track is 3000 nm in 4.2.1a.

Peak height of the screen printed silver track after embedding is around 140 nm (represented by blue line in the graph 4.2.1b), and height of the silver track embedded in the resist is measured by a trench after etching the silver from the resist which is around 1800 nm (represented by red line in the graph 4.2.1b). The percentage of silver embedded in the resist is 93% approximately.

Using R2R process better quality of the embedded sample can be prepared when compared to S2S process. This is because the pressure can be tuned according to our requirement during the embedding process using pressure rolls which is not possible with S2S process, because vacuum is used as a source of pressure in S2S process which has constant pressure during the process.

4.2.2 Resist-silver interface:

To understand more in detail at the interface of silver and resist optical interferometry and atomic force microscopy measurements were done.

4.2.2.1. Optical interferometry measurements:

Using the technique of optical interferometry, we can extract information at the interface of PEN foil (bad adhesion side) - screen printed silver and interface of screen printed silver – resist after embedding process:
(i) Interface of PEN-screen printed Silver:

The minimum height difference between PEN and screen printed silver is approximately 550nm (fig 5.4c). From the above figure a), it is clear that PEN foil has very smooth surface, and the interface between silver and PEN can be easily differentiated.

(ii) Interface of silver-resist after embedding process:

From the optical interferometry measurements it can be inferred that, the minimum height difference between embedded silver track and resist is 20nm approximately (fig 4.2.1f) whereas the minimum height difference between PEN and screen printed silver is approximately 550nm (fig 5.4c). This difference clearly indicates the degree of flatness after embedding of silver in the resist. The height difference between embedded silver track and resist at their interface varies locally. This might be due to variation in the filling of the resist at different places or due to the stress introduced in the resist layer while curing. Variation in the height of the resist thickness locally, is approximately ±10nm.
4.2.2.2 Atomic Force Microscopy measurements:

AFM tool is used to understand more in detail at the interface between resist and embedded silver tracks which is not possible with optical interferometry:

From the above figures 4.2.2.2 (a, b and c), it can be observed clearly that there is a trench with a depth of 100 nm and width approximately 5µm at the interface between resist and silver, it is more clearly visible in figure 4.2.2c. Measuring the dimensions of the trench at the interface between silver and resist was not possible with the optical interferometry due to this reason atomic force microscopy measurements were preferred. Trench formation might be because of incomplete filling of the resist up to 100 nm depth, or might be a crack due to stress introduced in the resist while curing.

4.3. Resist thickness on the R2R tool:

Resist thickness plays a crucial role in the quality of embedding. If the resist thickness is too low when compared to the thickness of the silver tracks, then embedding quality will be very poor. So, it is essential to know the resist thickness on the R2R tool as a function of different applied pressures on rolls 1 and 2. Confocal microscopy is used to get the measurements of resist thickness as a function of different pressures on the rolls 1 and 2 of R2R tool. Below is the measurement method regarding, how the resist thickness was measured.
Measurement method:

Below is the schematic view of the measuring procedure of resist thickness using R2R process.

![Resist Tape](image1)

4.3a) Schematic Cross-section view of the resist laminated on the tape which is placed on the acceptor substrate using R2R process.

![Resist thickness](image2)

4.3b) Schematic Cross-section view of the resist laminated on the acceptor substrate after removing the tape, which gives the resist thickness using R2R process.

From the above figure 4.3a, it can be explained that the tape is placed at the center of the acceptor substrate. After this, acceptor substrate is laminated with a resist (whose thickness has to be measured), in a similar fashion of embedding process on R2R tool. After the lamination process is completed, the tape is delaminated (figure 4.3b) and clear interface is obtained between the acceptor substrate, and the resist layer.

Confocal microscopy is used for resist thickness measurements because of its sensitivity in measuring the surface profile. Below is the line scan of the cured resist on the acceptor substrate after removing the tape. Thin film of gold around 20 nm is deposited at the interface between the acceptor substrate and resist, to differentiate surface topology between resist and the acceptor substrate using confocal microscopy.

![Line scan](image3)

Fig 4.3c) Line scan: Surface of the acceptor substrate (right side) after removing the tape which was covered by the cured resist on the top of it. Surface of the cured resist layer on the top of the PEN foil (left side) after lamination of resist on R2R tool is completed. Pressure on both the rolls (1 and 2) for the above graph is 2 bar.

It is observed from the above figure 4.3c that, the resist layer is not uniform; it is more like a wave. Exactly at the interface of acceptor substrate and resist layer, the thickness of the resist layer is higher.
when compared to the thickness of resist layer at 5µm on x-axis. Higher value of the resist thickness at
the interface of the resist layer and the acceptor substrate is due to the bump formed at the edges of the
tape during lamination process. In order minimize errors, the minimum height difference between the
acceptor substrate and resist layer is taken into consideration.

Similar measurements were performed for the pressures 3 bar, 4 bar and 5 bar. Results are discussed
below.

Settings used in R2R tool: Dancer pressure (it is the pressure responsible for tuning the tension in the web)
in both sides of the rolls (1 and 2) is 15% and speed of rotation of the rolls is 0.1metre/minute and resist
used is mr-UVcur21sf.

<table>
<thead>
<tr>
<th>S.no</th>
<th>Pressure on both rolls 1 and 2 (bars)</th>
<th>Minimum resist thickness approximately (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>20</td>
</tr>
<tr>
<td>2</td>
<td>3</td>
<td>7</td>
</tr>
<tr>
<td>3</td>
<td>4</td>
<td>2</td>
</tr>
<tr>
<td>4</td>
<td>5</td>
<td>3</td>
</tr>
</tbody>
</table>

Table 4.3a) Resist thickness as a function of pressure on both the rolls 1 and 2.

From the above table 4.3a) we can observe that, resist thickness decreases as the function of an
increase in pressure on both the rolls 1 and 2. Non-linearity is observed for the pressures 4 bar and 5 bar.
This error might be due to the measurement artifacts, of confocal microscope or might be due to non-
uniformity of the resist layer.

**4.4 Stress analysis:**

Stress analysis is important for the polymers because mechanical properties of the polymers depend on
the stress and strain. Stress is observed in the PEN foil and in the embedded samples with the help of the
polarizers in the figures 4.4a and 4.4b. But this observation is not enough to get a complete idea of stress
introduced during the embedding process. Qualitative analysis has to be done, using microscopes, which
can give us some data regarding optical birefringence from the samples. With the help of optical
birefringence, we can calculate the stress in the sample indirectly.3

Polarizers are used, to get first impression of stresses in the foils before and after the embedding
process.
For stress analysis, samples (figure 4.4a and 4.4b) were placed in between two polarizers and observed under white light. Basic principle of this analysis is, when light passes through these samples, each color in the visible light is polarized with its orientation. The structural stress is present at the locations where there is large concentration of colored bands and this can be observed in both the above figures 4.4a and 4.4b. The location of the stress is usually the location where there is a structural failure. When top polarizer is turned, the color pattern is changed as new colors are blocked and colors formerly blocked are visible.\(^2\)

### 4.5 Electrical resistance measurements:

The base line pattern used for R2R embedding of shunt lines is as follows in figure 4.5:

<table>
<thead>
<tr>
<th>Sintering type</th>
<th>Temperature (°C)</th>
<th>Design no</th>
<th>Resistance (Ω) (Between positive and negative terminals)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oven sintering</td>
<td>130</td>
<td>1</td>
<td>2.5</td>
</tr>
<tr>
<td></td>
<td>130</td>
<td>2</td>
<td>1.6</td>
</tr>
</tbody>
</table>

Fig 4.4a) Stress observed in PEN foil (before embedding).

Fig 4.4 b) Stress observed in the embedded sample.

Fig 4.5) Design of the donor substrate.
<table>
<thead>
<tr>
<th></th>
<th>130</th>
<th>3</th>
<th>2.1</th>
</tr>
</thead>
<tbody>
<tr>
<td>R2R photonic sintering</td>
<td>-</td>
<td>1</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

Table 4.5) Resistance measurements on different designs using different sintering techniques.

From the above table 4.5 in can be inferred that, in principle design 3 should have a higher resistance than design 1 and 2. Because electrons take the shortest path for travelling, which is possible in design 1 and 2 but not in 3. It has to travel through the length of the bus bar. But for design 3, resistance value is lower when compared to design 1. This measurement error might be because of non uniform application of pressure by squeegees while screen printing. It results in deposition of more ink on one side of the donor substrate. In this case towards design 3, more ink has been deposited.

Settings used for R2R Photonic sintering are:

Speed of the roll: 1.4 (number on the tool), intensity 60%, frequency : 10 Hz.

The resistance measurements were done before and after embedding the shunt lines. There is no change in the resistance values.

**4.6 Parameters effecting embedded shunt lines:**

From the above analysis of the results, several critical parameters affecting the process of embedding shunt lines have been identified based on the different characterization techniques used to analyze the samples are as follows:

(1) **Resist shrinkage:** It is one of the important factors, which affects the quality of embedding. The lower the value of the resist shrinkage, the better will be surface profile (flatness), thus leading to a better quality of the embedded samples. Selection of the resist plays an important role in the quality of embedding shunt lines.

(2) **Stress:** Localized stress present at the interface between silver and resist is very critical for the quality of embedding. If high stresses are present at the interface between silver and resist, then there might be the possibility of opening of a crack at their interface. It is also critical within the resist layer.

(3) **Adhesion mechanism:** Differences in the strength of adhesion present on the surfaces of donor and acceptor substrates are crucial for the concept of embedding of shunt lines to work. The larger the difference of adhesion between acceptor and donor substrate (acceptor substrate>donor substrate), the better will be the quality of embedding.
(4) **Peeling direction**: If the donor and acceptor substrates are peeled off randomly without any systematic manner, then this affects the quality of the embedded sample. Also, the direction of the design with which it is peeled off affects the quality of embedded sample.

![Image of R2R embedded sample](image)

By careful observation of the above figure 4.6, it can be analyzed that peeled direction is the direction in which the sample was embedded. It means that donor substrate is delaminated against the acceptor substrate in this direction. For this reason, it is observed that at the right side of silver lines, the transfer is not perfect. Some residue is left over on the donor substrate for both the bus bar and the grid line, because initially, some force is required to separate silver from the donor substrate. If the direction of embedding was along the peel direction, then only the residue of the bus bar would be left over on the donor substrate, and the grid lines would have been embedded completely. In this way, the direction of design along which the sample is embedded plays a critical role in the quality of embedding.

All the above results and analysis were performed for the embedded sample consisting of cured resist (mr-UVcur21sf) which is the baseline resist in this research work. If other resist is used, then some baseline parameters might be varying depending on the chemical and physical properties of the resist.

**Overview of S2S and R2R embedding process results are as follows:**

<table>
<thead>
<tr>
<th>Imprint type</th>
<th>Donor substrate type</th>
<th>Resist type</th>
<th>Resist quantity (ml)</th>
<th>Trench depth (height trench-depth in nm)</th>
<th>Height of the silver track above resist (height silver-resist in nm and % of embedding)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-</td>
<td>PEN with silver tracks</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>3000 nm</td>
</tr>
<tr>
<td>S2S embedding</td>
<td>- same-</td>
<td>Mr-UV Cur 21sf</td>
<td>1.5</td>
<td>883.62</td>
<td>180nm and 91%</td>
</tr>
<tr>
<td>Polymer</td>
<td>- same-</td>
<td>Mr-UVCur</td>
<td>-</td>
<td>-</td>
<td>±50nm (surface variation of...</td>
</tr>
</tbody>
</table>
Table 4.6) Overview of embedded shunt lines results using S2S and R2R process. Height profile of the silver tracks and trench depth were measured using dektak profilometry.

In the next chapter more detailed analysis regarding the adhesion mechanisms and its importance will be discussed.
References:


Chapter5: Adhesion mechanisms at polymer-metal interfaces.

5.1 Introduction:

The word adhesion is derived from Latin, originating from the word adhaerere or “stick to.” Today, the word adhesion is defined by ASTM D 907 as “The state in which two surfaces are held together by the interfacial forces which may consist of valence forces or interlocking forces or both.” The main conclusion which can be drawn from the above definition is adhesion refers to the connection strength of two materials against each other by different forces, which are acting on the contact area between those two materials at the molecular level. Energetic state of two surfaces brought into contact will determine the magnitude of adhesion. Energetic state of a surface is quantified by the surface energy. Thus, as a rough guide line, the empirical rule of adhesion states: adhesion between two surfaces will be low if they show large difference in surface energy and high if they have similar surface energy.\(^1\)

Interface: An interface can be described between two phases, a polymeric interphase which can be described as a transition zone between surfaces of the resist and second one is the bulk of the resist, which is cured or solidified against the substrate.\(^2\)

Besides the surface energy which is one of the important factors for adhesion between two different materials, influence of roughness on either of the surfaces in contact is another important factor, which will lead to large contact area and mechanical interlocking of the materials, thus leading to a better adhesion.\(^1\)

5.2 Surface roughness:

It is observed that the surface on which the silver ink is screen printed (bad adhesion side of PEN, donor substrate) is very smooth (see in fig 5.2b), when compared to the surface of acceptor substrate (Good adhesion side of the PEN foil) in figure 5.2a. Good adhesion on the acceptor substrate might be due to an increase in the contact area between the PEN foil and the material which is deposited or cured on it. In this way degree of adhesion on the surface of the substrate can be tuned by varying the roughness of surface.
(i) Dektak profilometry measurements:

Fig 5.2a) Surface profile measurement on the adhesive side of the PEN foil. Peaks can be observed on the PEN foil which promotes adhesion to the resist. This is used as the acceptor substrate.

(ii) SEM measurements on the PEN foil:

SEM measurements were performed on either sides of the PEN foil, to get more information regarding the surface roughness and the surface chemistry (using Energy-dispersive X-ray photo spectroscopy) to analyze exactly which factor is contributing to the surface adhesion.

From the above figures 5.2 (a, b, c and d), it can be observed that, the surface is very rough on the acceptor substrate (5.2(a, c)), when compared to the surface of the donor substrate (4.2 (b, d)) which helps the resist to cure and adhere well on the acceptor substrate but not on the donor material. This surface variation is very crucial for embedding shunt lines using R2R process.
5.3 Surface chemistry:

Analysis of the surface chemistry on the either sides of PEN foil (donor and acceptor substrate) is important, to understand the embedding shunt lines process more in detail. If the surface chemistry is on either sides of the PEN foil is different, then we can understand that the surface chemistry plays an important role in the process of embedding shunt lines apart from surface energy and surface roughness. If surface chemistry is similar on the either sides of PEN foil, then we can assume that surface energy and surface roughness plays more critical role in the process of embedding shunt lines. EDX spectroscopy is used to see the different chemical elements present on the either sides of a PEN foil.

The ratio, in which different chemical elements present on the surface of good (acceptor substrate) and bad adhesion side of the PEN foil using EDX spectroscopy, is explained in the table 5.3 below as follows:

Surface Chemistry on either sides of the PEN foil (using EDX) as follows:

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt %</th>
<th>At %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesive side of the PEN foil</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CK</td>
<td>55.64</td>
<td>61.83</td>
</tr>
<tr>
<td>NK</td>
<td>9.85</td>
<td>9.39</td>
</tr>
<tr>
<td>OK</td>
<td>34.5</td>
<td>28.78</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Anti-adhesive side of the PEN foil</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CK</td>
<td>56.57</td>
<td>62.71</td>
</tr>
<tr>
<td>NK</td>
<td>9.7</td>
<td>9.22</td>
</tr>
<tr>
<td>OK</td>
<td>33.72</td>
<td>28.06</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 5.3) Ratio of different elements present on the either sides of the PEN foil.

From the above table 5.3, it can be inferred that the ratio in which different chemical elements present on either side of the PEN foil are almost identical in terms of wt% (weight percentage) and At% (atomic percentage). Presence of nitrogen on either sides of the PEN foil indicates that the PEN foil contains some impurities. Different measurement techniques should be used further, in order to know the chemical structures on the surface of the PEN foil, to get better understanding of variation in adhesion on both sides of the PEN foil. But from this measurement of quantitative analysis, no conclusion about variation in the chemical composition on the surface of either side of the PEN foil can be drawn.
Good adhesion on the adhesive side of the PEN foil might be due to irradiating it by KrF excimer laser, (248nm) in Vacuum with different influences and seven pulses. By treating it this way on the smooth side of the PEN foil, which is semi crystalline in nature, will change into an amorphous form at the surface zone within few hundreds of nanometers, leaving the bulk properties of the semi crystalline PEN foil unaltered. This process of laser treatment reduces the polymer chain length and introduces chain scission, which might play an important role in enhancing the adhesion.\(^3\)

Decrease in the chain length will lead to enhancement in the mobility of the polymer chains in the amorphous region, which might support the lamination process. There is also a possibility that new chain ends, which may consist of other chemical groups than that irradiated polymer, which may act as chemical reaction centers for promoting adhesion.\(^3\)

**Polymer-metal interaction:**

The interaction between metal and polymer strongly depends on the functional group of a polymer and the type of metal used. Oxidation of polymer surfaces is one among the important techniques used to promote adhesion of polymers to metals, and it can be done prior to or during the formation of the interface. Chemical modification of the interfacial polymer chains with polar groups like carbonyl and hydroxyl, etc. is also used.\(^1\)

Strong interaction at the metal-polymer interface may also originate from chemisorption, which eventually leads to molecular bonding. Chemisorption can be classified between weak physical interactions and chemical reactions. In few cases, molecular bonding may lead to chemical reaction.\(^2\)

Most of the polymer-metal interfaces are fractal in nature; boundaries are not smooth microscopically. Adsorption of polymers on fractal surfaces is reported recently\(^2\). Below figures 5.3c and 5.3d representing the fractal nature of the polymer and metal interfaces (computer generated) which indicate that there are very rough in nature. This is one of the reasons which promotes mechanical interlocking between metal and polymer and helps in embedding of silver in the resist.

Figures 5.3 c and d) Represents computer generated the fractal nature of the polymer Interface (5.3 c) and the metal interface (5.3 d).\(^2\)
Selectivity of adsorption is not only dictated by geometric accessibility, but also by chemical heterogeneity of a surface. There is also a possibility that electrons are transferred from HOMO of the polymer to the conduction band edge of the metal atom or from the valence band edge of the metal atom to the LUMO of the polymer. This kind of molecular interaction is also known as acid-base interaction.²

Molecular interaction between a discrete molecule (molecules which interact via Van der Waals forces), and a metal has an additional interaction due to internal transition of the electrons between the bulk and surface of the metal. Below figure 5.3e representing the charge transfer between HOMO, LUMO of the polymer and valence band of the metal atoms.

![Figure 5.3e) Interaction between metal atoms and polymer.²](image)

### 5.4 Mechanical peel test measurements.

**(i) Sample preparation and setup:**

Mechanical peel test measurements were performed in order to calculate the interface adhesion energy between different materials like resist-silver (embedded), resist-PEN (acceptor substrate), PEN (donor substrate)-screen printed silver and PEN (acceptor substrate)-screen printed silver.

For all the peel test measurements, typical sample dimensions were length 3 cm and width 1 cm approximately. Width is important for this peel test, because it defines the contact area at the time of peeling. Before conducting tests, samples are fixed using double-sided tape onto the glass. One side of the tape is fixed to the sample and other side of the tape is fixed to the glass. Samples used are embedded silver in the resist cured on the PEN foil, screen printed silver on the PEN foil and only resist cured on the PEN foil.

The whole setup along with glass is clamped to the peel test tool. Actual length of the sample which is fixed is around 2 cm, which consists of embedded shunt lines or screen printed silver track or resist depending upon the type of the sample. Remaining 1 cm is just a foil which is fixed to the tool, and it is peeled slightly so that it is positioned perfectly, at the interface of silver-resist or silver-PEN or resist-PEN depending upon the type of the sample. This is done to ensure that the total stress is acting at the interface of the two materials where test has to be performed. Below are the figures 5.4a and 5.4b representing peel test measurement setup.
Once everything is arranged, peel test is started and the force with which two materials are separated from their interface is measured and the data is plotted (on y-axis), against the time required to peel of the entire sample (on x-axis).

For instance, if we consider a sample of embedded silver in the resist, which is cured on the PEN foil, taken for the peel test measurement in the above figure (5.4b), then embedded silver, which is actually the top side of the sample, is fixed to the double sided tape and other side of the tape is fixed to the glass. There are three interfaces which have to be taken into account (i) interface between tape-silver, (ii) silver-resist and (iii) resist-PEN. Once the peel test measurement is started, the weakest interface of the above three starts delaminating, and the interface adhesion energy is calculated for it. In the above case, it was observed that weakest interface was of silver-resist. This interface started delaminating and the force with which the silver and the resist were separated was measured and plotted on the y-axis. The time taken to delaminate the entire sample was plotted on x-axis.

(ii) Peel test analysis:

Interface Adhesion Energy: \( f^* (1-\cos \Theta)/w \), units: N/m. (1)

\( f \) – Peeling force (average value of y axis is taken into account).

\( w \) – Width of the sample.

\( \Theta \) - 180 deg.\(^5\)

Resist used: mr-UVcur21sf.
By substituting the values of width of the sample (in our case it is 1 cm) and average load in the above equation (1) we can obtain interface adhesion energy.

It is observed from the above figures 5.4c and 5.4d that, the interface adhesion energy between resist-PEN (acceptor substrate) is much better than the interface adhesion energy between resist-embedded silver. Due to very low interface adhesion energy between resist-embedded silver, both materials were separated easily at minimum load and it is the maximum of interface adhesion energy present between the above two materials.

But the interface adhesion energy between resist-PEN (acceptor substrate) was very high, even at maximum load, separation of resist and PEN (acceptor substrate) was not possible (figure 5.4c). The weakest interface was of tape and resist, and this interface got separated but not the interface of resist and PEN (acceptor substrate). It means that the interface adhesion energy which is calculated for resist-PEN (acceptor substrate) is of the minimum value (value of the weakest interface i.e resist and tape) and it was not possible to determine the maximum interface adhesion energy, the value at which resist and PEN (acceptor substrate) gets separated. Tape used for the test was the strongest tape available for the measurement. Due to this reason, for resist-PEN (acceptor substrate) interface it is quantified as the minimum interface adhesion energy.

The same principle of quantification of minimum and maximum interface adhesion energy holds for below measurements of interface adhesion energy between silver-PEN (acceptor substrate) and silver-PEN (donor material)

Interface adhesion energy measurements were performed at the interface of silver-PEN (acceptor substrate) and silver-PEN (donor material) as shown below.
Maximum interface adhesion energy between silver-PEN (donor material) is 20 N/m approximately.

Minimum interface adhesion energy between silver-PEN (acceptor substrate) is 2000N/m approximately.

Interface adhesion energy between silver-PEN (acceptor substrate) is much better than the interface adhesion energy between silver–PEN (donor material).

The above peel test measurements clearly indicate that interface adhesion energy between PEN (acceptor substrate) and resist or silver is very high.

(iii) Measurement results:

Significant difference in the interface adhesion energy is observed between either sides of the PEN foil with both the resist and the silver. Further investigation regarding the composition of the PEN foil has to be done, to get much better understanding regarding the concept of embedding.

From the above measurements, it is clear why embedding works, when we make use of either sides of the PEN foil effectively. One side as acceptor substrate and other side for making donor substrate (taking an advantage of different surface treatments on either side of the PEN foil), due to which resist gets cured on the acceptor substrate but not on the donor substrate. The roughness present on the acceptor substrate promotes adhesion for both the resist and the silver due to its enhanced surface energy.

5.5 Surface energy analysis:

(i) Surface energy: Energy required to form an unit area of the new surface at an interface between liquid and solid.6
If the surface energy is higher, then resist wets thoroughly on the surface on which it has to be bonded, so that it maximizes its contact area and increases the molecular interaction between the resist and the bonding surface.

**Surface Energy (Disperse interactions):** Interactions are because of the time fluctuations of the charge distribution within the molecules.\(^7\)

**Surface Energy (Polar interactions):** It is described by Coulomb interactions between permanent dipoles and the ones between permanent and induced dipole.\(^7\)

**(ii) Theory:**

Under isothermal conditions, minimum free energy of a system corresponds to equilibrium state. Under these conditions, we can find only one value of equilibrium contact angle \((\theta)\). Such calculation yields the known equation first derived by Young\(^8\): 

\[
\sigma_s = \sigma_{sl} + \sigma_L \cos \theta
\]  

where \(\sigma_s\)=Overall surface tension of the wetting liquid, \(\sigma_{sl}\)=interfacial tension between the solid and liquid and \(\theta\)=contact angle between liquid and solid.

**Owens/Wendt theory (two component model for solid surface energy):**

This theory is used in calculating the surface energies of a solid. It is developed to know specific (polar types) interactions between solid surfaces and liquids. It states that the surface energy of a solid consists of two components namely polar and disperse. Dispersive component theoretically accounts for van der Waals and other non-site specific interactions, which a surface is capable of having with applied liquids. Polar component accounts for dipole-dipole, hydrogen bonding, dipole-induced dipole and other site specific interactions which a surface is capable of having with applied liquids.\(^4\)

This theory is based on two fundamental mathematical equations as follows:

Young’s equation:

\[
\sigma_s = \sigma_{sl} + \sigma_L \cos \theta \quad (1)
\]

Goods equation:

\[
\sigma_{sl} = \sigma_s + \sigma_L - 2\sqrt{\sigma_L^D \cdot \sigma_s^D} - \sqrt{\sigma_L^P \cdot \sigma_s^P} \quad (2)
\]

Equations (1) and (2) are combined to get the following equation:

\[
\frac{(1 + \cos \theta)\sigma_L}{2\sqrt{\sigma_L^D}} = \sqrt{\sigma_s^P \cdot \sigma_L^P} + \sqrt{\sigma_s^D \cdot \sigma_L^D} \quad (3)
\]
The contact angle, \( \theta \), is determined experimentally, \( \sigma_L = \sigma_L^P + \sigma_L^D \) (polar and disperse parts of the liquid) is known from literature, thus \( \sigma_L^P \) and \( \sigma_L^D \) are the two unknowns in this expression. By using liquids with known disperse and polar energy contributions, we can extract the \( \sigma_L^P \) and \( \sigma_L^D \) by plotting \( \frac{(1 + \cos \theta) \sigma_L}{2\sqrt{\sigma_L^D}} \)
as function of \( \sqrt{\frac{\sigma_L^D}{\sigma_L^P}} \). The slope gives the polar enthalpy of the surface, \( \sqrt{\sigma_L^P} \), and the intercept with the y-axis gives the disperse contribution, \( \sqrt{\sigma_L^D} \). It is essential to use liquids with significantly different enthalpy contributions to get an accurate value for the values of the surface.\(^4\)

Three different known liquids used and their corresponding polar and disperse parts are as follows:

<table>
<thead>
<tr>
<th>S.no</th>
<th>Liquid (mN/m)</th>
<th>Polar Part (mN/m)</th>
<th>Disperse Part (mN/m)</th>
<th>Total surface energy (mN/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Water</td>
<td>21.8</td>
<td>51</td>
<td>72.8</td>
</tr>
<tr>
<td>2</td>
<td>Ethylene Glycol</td>
<td>19</td>
<td>29</td>
<td>48</td>
</tr>
<tr>
<td>3</td>
<td>Diodomethane</td>
<td>58.8</td>
<td>0</td>
<td>58.8</td>
</tr>
</tbody>
</table>

Table 5.5a) Polar and disperse parts of three known liquids.

Using three known liquids, polar and disperse parts of resists (cured and liquid form), PEN foil and silver are calculated and their corresponding values are as follows:

<table>
<thead>
<tr>
<th>S.no</th>
<th>Sample type</th>
<th>SE (Disperse) (mN/m)</th>
<th>SE (Polar) (mN/m)</th>
<th>Total SE (mN/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Anti-adhesive PEN</td>
<td>38.89</td>
<td>4.32</td>
<td>43.21</td>
</tr>
<tr>
<td>2</td>
<td>Adhesive PEN</td>
<td>36.36</td>
<td>10.71</td>
<td>47.07</td>
</tr>
<tr>
<td>3</td>
<td>Screen printed Silver (Embedded)</td>
<td>38.38</td>
<td>1.32</td>
<td>39.71</td>
</tr>
<tr>
<td>4</td>
<td>mr-UVCur21sf (cured)</td>
<td>37.62</td>
<td>10.16</td>
<td>47.49</td>
</tr>
<tr>
<td>5</td>
<td>Customized resist (cured)</td>
<td>35.34</td>
<td>6.58</td>
<td>41.9</td>
</tr>
<tr>
<td>7</td>
<td>mr-UVCur21sf (liquid)</td>
<td>-</td>
<td>-</td>
<td>36.75 (surface tension)</td>
</tr>
<tr>
<td></td>
<td>Customized resist (liquid)</td>
<td>-</td>
<td>-</td>
<td>34.35 (Surface tension)</td>
</tr>
<tr>
<td>-----</td>
<td>---------------------------</td>
<td>------------------------</td>
<td>------------------------</td>
<td>-------------------------</td>
</tr>
</tbody>
</table>

Table 5.5b) Measurement results of polar and disperse part of solids and surface tension of uncured resists.

From the above table 5.5b, it can be observed that the polar part of acceptor substrate is much higher than polar part of donor substrate. It means that acceptor substrate, wets well and has much better adhesion compared to donor substrate.

Polar part of cured resist (4 and 5) is higher than polar part of donor substrate. This might help in curing of resist on the acceptor substrate but not on the donor substrate, when substrates are pressed against each other while resist is sandwiched between them during curing.
References:


[4] Christopher Rulison. “So you want to measure surface energy?” Kruss USA.


Chapter 6: Integration and alternatives.

6.1 Integration:

The embedded shunt lines were integrated into an OLED device to verify the proof of concept.

Cross section of an OLED device fabricated is as follows:

![Cross section of OLED](image)

Fig 6.1a) Cross section of OLED fabricated. PEN foil 135μm, anode: embedded silver, high conductive PEDOT 100nm, LEP 80nm, cathode: barium 5nm and aluminum 100nm.

Fabrication steps:

The high conductive PEDOT was spin coated on the embedded shunt lines at 1000 rpm to achieve 100 nm thicknesses and placed on a hot plate for 6 minutes at 130°C. In next step, LEP (light emitting polymer) was spin coated at a speed of 810 rpm to achieve 80 nm thicknesses and placed on a hot plate for 6 minutes 130°C. After this step, at first barium was sputtered deposited to achieve 5nm thickness and at last aluminum was sputtered deposited to achieve 100 nm thicknesses.

OLED testing:

After fabrication of an OLED without any encapsulation, it was tested under inert conditions as follows in figure 6.1b and 6.1c:

![OLED tested under inert conditions](image)

![OLED tested in external environment](image)

Fig 6.1b) OLED tested under inert conditions.

Fig 6.1c) OLED tested in external environment.
OLEDs worked for about 3 minutes after removing from the inert condition. OLEDs were not encapsulated after removing from inert conditions which protects them from moisture.

6.2 Embedding experiments of carbon stacked with silver:

Introduction:

To avoid the problem of sliver migration in the OLED devices which make use of silver for fabrication of electrodes, the concept of shielding silver with other conductive layer was experimented. Carbon was used as a shielding agent.

![Fig 6.2a) Resist sandwiched between donor substrate (with carbon stacked silver pattern) and acceptor substrate or carrier substrate.](image)

Fig 6.2a) Resist sandwiched between donor substrate (with carbon stacked silver pattern) and acceptor substrate or carrier substrate.

![Fig 6.2b) Embedded carbon stacked silver sample.](image)

Fig 6.2b) Embedded carbon stacked silver sample.

Procedure:

At first carbon ink is screen printed on the donor substrate and sintered in an oven at $130^\circ$ C for about 15 minutes. After this step, using alignment marks silver ink is screen printed exactly above the carbon ink and again sintered in an oven at $130^\circ$ C, for about 15 minutes. After screen printing process is completed, next step is embedding carbon stacked silver tracks. For this, resist is droplet dispensed on the donor substrate, and then it is pressed against the acceptor substrate. Then, the whole setup of resist, the acceptor substrate and the donor substrate is placed under UV lamp, to allow the resist to cure. After complete curing of the resist, the acceptor substrate or carrier substrate is delaminated from the donor substrate. This results in, leaving the carbon stacked silver tracks embedded in the cured resist on the acceptor substrate.

Inks used: Dupont 5025 silver micron particle paste and Dupont 7102 carbon $\mu$-particle paste.
From the above figure 6.2c it silver beneath the carbon clearly visible in the form of shiny particles. This is due to the pin holes present in the carbon ink after sintering and problem of pin holes has to be solved.

Analysis of embedded carbon stacked with silver, using dektak profilometry are as follows:

<table>
<thead>
<tr>
<th>S.no</th>
<th>Type</th>
<th>Average height (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td><strong>Bus bar measurements</strong></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>Screen printed Carbon bus bar</td>
<td>5.21</td>
</tr>
<tr>
<td>2</td>
<td>Micro particle silver bus bar</td>
<td>5.61</td>
</tr>
<tr>
<td>3</td>
<td>Carbon-silver screen printed bus bar</td>
<td>9.96</td>
</tr>
<tr>
<td>4</td>
<td>Embedded carbon-silver bus bar</td>
<td>0.995</td>
</tr>
<tr>
<td></td>
<td><strong>% Embedding</strong></td>
<td>90%</td>
</tr>
<tr>
<td></td>
<td><strong>Gridline Measurements</strong></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>Screen printed carbon gridline</td>
<td>4.68</td>
</tr>
<tr>
<td>2</td>
<td>Screen printed micro particle silver</td>
<td>3.97</td>
</tr>
<tr>
<td>3</td>
<td>Carbon-silver screen printed gridline</td>
<td>5.42</td>
</tr>
<tr>
<td>4</td>
<td>Embedded carbon-silver gridline</td>
<td>0.166</td>
</tr>
<tr>
<td></td>
<td><strong>% Embedding</strong></td>
<td>96%</td>
</tr>
</tbody>
</table>

Table 6.2) Profilometry measurements of the screen printed carbon stacked silver lines and also of the embedded samples.
Challenges:

- To achieve, control on the uniformity and thickness of the resist.
- Pin holes were observed in screen printed carbon ink. Solution for the pin holes in the screen printed carbon ink should be addressed, or else silver migration will occur.
- More, detailed understanding of the adhesion between carbon and silver ink has to be gained, in order to get stabilized embedding results.
Conclusion:

Successful embedding of the screen printed silver shunt lines was done using S2S, and R2R process in a UV sensitive resist. Approximately 93% of the silver shunt lines were embedded in the resist using R2R process. Relative variation in the degree of adhesion on the surface of donor and acceptor substrate is very important for embedding of the shunt lines (degree of adhesion: Acceptor substrate > Donor substrate). The larger the difference (degree of adhesion between donor and acceptor substrate) between them, the better will be the quality of embedded shunt lines. Resist shrinkage is also one of the important factors, which affects the quality of embedding. Resist, which has least shrinkage properties must be selected for embedding of the shunt lines. Successful integration of R2R embedded shunt is done into an OLED device.

In brief concept of the embedding shunt lines (method 2) works as follows:

The donor substrate and the acceptor substrate are pressed against each other while the resist is sandwiched between them. Due to surface roughness on the acceptor substrate, resist is cured on the acceptor substrate but not on the donor substrate. While curing of the resist, it gets mechanically interlocked with the silver tracks present on the donor substrate and also there might be chemical interaction between resist and silver. Mechanical interlocking/chemical interaction between silver and resist, has a greater degree of adhesion when compared to the adhesion between donor substrate and silver. When the acceptor substrate is delaminated from the donor substrate after the resist is cured completely, due to variation in the degree of adhesion, resist is cured on the acceptor substrate, and it takes silver tracks along with it from the donor substrate. Thus, in this way the concept of embedding shunt lines in the resist works.

Future work:

Alternative materials (for example: different foils, different silver inks) apart from the materials used in this research work have to be explored which constitutes low cost and better functionality. Modification of R2R imprint tool from the current state of art which is compatible for full scale R2R embedding of shunt lines. Epoxy based resists and other available acrylate based resists which has less shrinkage properties should be tried, which can give better quality of embedded samples. In depth investigation of localized stress at the interface between resist-silver and within the resist layer has to be done.
Appendix:
— Processing guidelines —

mr-UVCur21SF – UV-curable Polymer for UV-based Nanoimprint Lithography

Characteristics

mr-UVCur21SF is a liquid UV-curable polymer system with low viscosity and high curing rate designed for UV-based nanoimprint lithography. It is provided as a ready-to-use liquid and can be spin-coated or dispensed on the substrate. Optimum imprint results on Si or SiO₂ substrates are achieved by applying adhesion promoter mr-APS1 before coating mr-UVCur21SF.

mr-UVCur21SF is deposited on the substrate by spin coating or droplet dispensing.

UV-transparent mould is pressed into the liquid polymer system.

Polymer system is cured by UV exposure at room temperature.

Mould is detached.

Residual polymer layer is removed by anisotropic plasma etching.

Physical data

<table>
<thead>
<tr>
<th></th>
<th>mr-UVCur21SF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colour, appearance</td>
<td>colourless, clear</td>
</tr>
<tr>
<td>Film thickness after spin coating [1] [μm]</td>
<td>1.7 ± 0.2</td>
</tr>
<tr>
<td>Density [2] [g cm⁻³]</td>
<td>1.089 ± 0.005</td>
</tr>
<tr>
<td>Dynamic viscosity [2] [mPa s]</td>
<td>30 ± 1</td>
</tr>
<tr>
<td>Refractive index [nD] [2]</td>
<td>1.487 ± 0.002</td>
</tr>
</tbody>
</table>

[1] Liquid film after spin coating at 3000 rpm for 60 s and subsequent softbake at 80 °C for 1 min

Processing

Best patterning results are obtained at temperatures of 20 – 25 °C and a relative humidity of 40 – 46 %.
Processing conditions

<table>
<thead>
<tr>
<th>Process step</th>
<th>mr-UVCur21SF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Substrate preparation</td>
<td>(1) Oven 200 °C, 30 min (2) Adhesion promoter pretreatment (mr-APS1)</td>
</tr>
</tbody>
</table>
| Coating | Spin coating 
  3000 rpm for 60 s Droplet dispensing: |
| Softbake (optional) | |
| Hotplate | 60 s at 80 °C |
| Film thickness (liquid film) | 1.6 ± 0.2 |

Imprinting and UV exposure

| Temperature | room temperature |
| Pressure | > 100 |
| Exposure dose | > 700 |

1) Uncovered spin coater
2) The volume to be dispensed depends on the mould layout, on the pattern density, and on the targeted residual layer thickness
3) Dependent on the pattern density of the mould
4) Referring to a UV intensity integrated in the wavelength range 320–420 nm, curing in vacuum

Substrate preparation

The substrates have to be free of impurities and moisture in order to achieve optimum adhesion. The substrates should be baked at 200 °C and cooled to room temperature immediately before coating. Alternatively, oxygen or ozone plasma cleaning is recommended. For improving the polymer film adhesion to Si or SiO2 substrates it is advisable to apply an adhesion promoter. We recommend to use mr-APS1 (micro resist technology). Omnicoat (MicroChem Corp., USA) works as an adhesion promoter, too. HMDS (hexamethyl-disilazane) is not suitable.

Coating

The substrate can be coated with mr-UVCur21SF by spin coating or droplet dispensing.

1) Spin coating

Uniform coatings are obtained by spin coating of the liquid polymer in the thickness range indicated in the spin curve in Fig. 1. Please select the appropriate spin speed required for the desired film thickness and application. A spin time of 60 s is recommended, since lower film thicknesses and a higher film quality can be achieved compared to 30 s.
Fig. 1: Spin curve, 60 s spin time (liquid film after spin coating and softbake)

The refractive indices of the polymer films depending on the wavelength and the Cauchy equation are given in Fig. 2. This information is needed for ellipsometric or other optical thickness measurement.

\[ n(\lambda) = n_0 + \frac{n_1}{\lambda^2} + \frac{n_2}{\lambda^4} \]

\( n_0 = 1.473 \)
\( n_1 = 16 \)
\( n_2 = 88 \)

Fig. 2: Refractive indices vs. wavelength, Cauchy coefficients of liquid mr-UVCur21SF after spin coating and subsequent softbake

(2) Dispensing
A drop of mr-UVCur21SF or an array of droplets is applied to the substrate. The volume to be dispensed depends on the mould layout, on the pattern density, and on the targeted residual layer thickness.
Softbake

The spin-coated films can be baked optionally at 80 °C for 60 s on a hot plate before UV exposure. This step is not necessary, but can improve film quality and uniformity.

Imprint and UV exposure

Main factors determining the imprint conditions are the viscosity of the polymer system, the mould layout (feature size, density of the patterns etc.), the residual layer thickness to be attained and the imprinting tool. mr-UVCur21SF can be imprinted in any tool suitable for doing UV-based nanoimprint lithography. Commercial nano-imprinters as provided e.g. by EV Group (Austria), Obducat (Sweden), and Suss MicroTec (Germany) may be used.

Mould

It is highly recommended to treat the UV-transparent mould with a release agent like F₃₅-OTCS or other ones before using it for nanoimprinting. F₃₅-OTCS (trichloro-(1H,1H,2H,2H-perfluorooctyl)-silane, CAS number [78560-45-9]) is commercially available from many suppliers of laboratory chemicals.

Imprint conditions

The mould with nanometer-scale patterns is pressed into the films with a pressure of > 100 mbar to transfer the pattern. The imprint pressure and time necessary to get complete filling of the mould cavities depends on the pattern density and pattern width. Since mr-UVCur21SF has a very low viscosity, the time necessary to build up the imprint pressure is sufficient to completely fill the patterns.

UV exposure

The polymer has to be exposed to UV light for curing. Sufficient curing is attained at doses of approximately 700 ml/cm² (curing under vacuum) using broad band UV light (320 – 420 nm). Applying higher doses or broader UV wavelength ranges do not affect the imprint quality or the properties of the cured polymer. Exposure applying a smaller UV range of e.g. 350 – 400 nm or applying the single 365 nm line works as well, but will require higher doses.

The degree of shrinkage of mr-UVCur21SF during the UV exposure is approximately 3 – 4 % (linear shrinkage). Reproducible exposure conditions will lead to reproducible shrinkage values. After UV exposure the mould is detached.

Residual polymer layer removal

The residual layer remaining in the recessed areas of the polymer film after the imprint is removed by oxygen reactive ion etching (RIE) in order to open the window to the substrate.

Removal

After the whole process residue-free removal of mr-UVCur21SF is preferably achieved applying oxygen plasma. Since mr-UVCur21SF does not contain any inorganic components like silicon, no residues are left on the substrate after plasma treatment with pure oxygen.
Storage

Storage at temperatures of 15 – 25 °C is recommended. Storage in a refrigerator is not required. mr-UVCur21SF is light-sensitive and should not be exposed to direct daylight. mr-UVCur21SF bottles have to be handled under yellow light. Keep the bottles closed when not in use. Under these conditions, a shelf life of 12 months from the date of manufacture is ensured.

Disposal

Dispose of as halogen free solvent.

Environmental and health protection

Ensure that there is adequate ventilation while processing the polymer solution. Avoid contact of the solution with skin and eyes and breathing solvent vapours. Wear suitable protective clothing, safety goggles and gloves.
ProLight PG1C-3LLx
3W UV Power LED
Technical Datasheet
Version: 1.8

Features
● Industry best moisture sensitivity level - JEDEC 2a
  4 week floor life without reconditioning
● Low-temp. & lead free reflow soldering
● RoHS compliant
● Instant light (less than 100ns)
● Cool beam, safe to the touch
● Superior ESD protection

Typical Applications
● UV gluing, UV curing, UV marking
● UV drying of printing inks and lacquers
● Currency inspection
● Forensic analysis - urine, protein stains
● Leak detection using fluorescent dyes
● Detects fluorescing minerals and gems
Emitter Mechanical Dimensions

Notes:
1. The cathode side of the device is denoted by a hole in the lead frame.
2. Electrical insulation between the case and the board is required --- slug of device is not electrically neutral. Do not electrically connect either the anode or cathode to the slug.
3. Drawing not to scale.
4. All dimensions are in millimeters.
5. All dimensions without tolerances are for reference only.
6. Please do not bend the leads of the LED, otherwise it will damage the LED.
7. Please do not use a force of over 3kgf impact or pressure on the lens of the LED, otherwise it will cause a catastrophic failure.

*The appearance and specifications of the product may be modified for improvement without notice.
Star Mechanical Dimensions

Notes:
1. Slots in aluminum-core PCB for M3 or #4 mounting screw.
2. Electrical interconnection pads labeled on the aluminum-core PCB with "+" and "-" to denote positive and negative, respectively. All positive pads are interconnected, as are all negative pads, allowing for flexibility in array interconnection.
3. Drawing not to scale.
4. All dimensions are in millimeters.
5. All dimensions without tolerances are for reference only.
6. Please do not use a force of over 3kgf impact or pressure on the lens of the LED, otherwise it will cause a catastrophic failure.

*The appearance and specifications of the product may be modified for improvement without notice.
Flux Characteristics at 700mA, $T_J = 25^\circ$C

<table>
<thead>
<tr>
<th>Radiation Pattern</th>
<th>Color</th>
<th>Part Number Emitter</th>
<th>Part Number Star</th>
<th>Radiometric Power (mW) Minimum</th>
<th>Radiometric Power (mW) Typical</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lambertian</td>
<td>UV</td>
<td>PG1C-3LLE</td>
<td>PG1C-3LLS</td>
<td>635</td>
<td>780</td>
</tr>
</tbody>
</table>

- ProLight maintains a tolerance of ± 10% on flux and power measurements.
- Please do not drive at rated current more than 1 second without proper heat sink.

Electrical Characteristics at 700mA, $T_J = 25^\circ$C

<table>
<thead>
<tr>
<th>Color</th>
<th>Min. $V_F$ (V)</th>
<th>Typ. $V_F$ (V)</th>
<th>Max. $V_F$ (V)</th>
<th>Thermal Resistance Junction to Slug ($^\circ$C/ W)</th>
<th>Thermal Resistance Junction to Board ($^\circ$C/ W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UV</td>
<td>2.85</td>
<td>3.5</td>
<td>4.1</td>
<td>5.5</td>
<td>8</td>
</tr>
</tbody>
</table>

Optical Characteristics at 700mA, $T_J = 25^\circ$C

<table>
<thead>
<tr>
<th>Color</th>
<th>Min. $\lambda_p$ (nm)</th>
<th>Typ. $\lambda_p$ (nm)</th>
<th>Max. $\lambda_p$ (nm)</th>
<th>Total included Angle (degrees) $\theta_{0.90V}$</th>
<th>Viewing Angle (degrees) $2 \theta_{1/2}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>UV</td>
<td>390 nm</td>
<td>400 nm</td>
<td>410 nm</td>
<td>160</td>
<td>140</td>
</tr>
</tbody>
</table>

- ProLight maintains a tolerance of ± 1nm for dominant wavelength measurements.
### Absolute Maximum Ratings

<table>
<thead>
<tr>
<th>Parameter</th>
<th>UV</th>
</tr>
</thead>
<tbody>
<tr>
<td>DC Forward Current (mA)</td>
<td>700</td>
</tr>
<tr>
<td>Peak Pulsed Forward Current (mA)</td>
<td>1000</td>
</tr>
<tr>
<td>Average Forward Current (mA)</td>
<td>700</td>
</tr>
<tr>
<td>ESD Sensitivity (HBM per MIL-STD-883E Method 3015.7)</td>
<td>±4000V (Class III)</td>
</tr>
<tr>
<td>LED Junction Temperature (°C)</td>
<td>120</td>
</tr>
<tr>
<td>Aluminum-core PCB Temperature (°C)</td>
<td>100</td>
</tr>
<tr>
<td>Storage &amp; Operating Temperature (°C)</td>
<td>-40 to + 100</td>
</tr>
<tr>
<td>Soldering Temperature (°C)</td>
<td>235°C</td>
</tr>
</tbody>
</table>

### Radiometric Power Bin Structure

<table>
<thead>
<tr>
<th>Color</th>
<th>Bin Code</th>
<th>Minimum Radiometric Power (mW)</th>
<th>Maximum Radiometric Power (mW)</th>
<th>Available Color Bins</th>
</tr>
</thead>
</table>
| UV    | S        | 635                           | 755                           | 2, 3, 4 [1]
|       | T        | 755                           | 875                           | 2, 3, 4 [1]          |

- ProLight maintains a tolerance of ± 10% on flux and power measurements.
- The flux bin of the product may be modified for improvement without notice.
- *[1]* The rest of color bins are not 100% ready for order currently. Please ask for quote and order possibility.

### Peak Wavelength Bin Structure

<table>
<thead>
<tr>
<th>Color</th>
<th>Bin Code</th>
<th>Minimum Peak Wavelength (nm)</th>
<th>Maximum Peak Wavelength (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UV</td>
<td>1</td>
<td>390</td>
<td>395</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>395</td>
<td>400</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>400</td>
<td>405</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>405</td>
<td>410</td>
</tr>
</tbody>
</table>

- ProLight maintains a tolerance of ± 1nm for peak wavelength measurements.
Color Spectrum, \( T_J = 25^\circ C \)

1. UV

![Graph showing relative spectral power distribution vs. wavelength.]

Light Output Characteristics

Relative Light Output vs. Junction Temperature at 700mA

![Graph showing relative light output vs. junction temperature.]

ProLight
Forward Current Characteristics, \( T_J = 25^\circ C \)

Fig 1. Forward Current vs. Forward Voltage for UV.

Fig 2. Relative Luminous Flux vs. Forward Current for UV at \( T_J = 25^\circ C \) maintained.

Ambient Temperature vs. Maximum Forward Current

1. UV (\( T_{J\text{MAX}} = 120^\circ C \))

- \( R_{\theta JA} = 30^\circ C/W \)
- \( R_{\theta JA} = 25^\circ C/W \)
- \( R_{\theta JA} = 20^\circ C/W \)
- \( R_{\theta JA} = 15^\circ C/W \)
Typical Representative Spatial Radiation Pattern

Lambertian Radiation Pattern

![Lambertian Radiation Pattern Graph](image-url)
Moisture Sensitivity Level - JEDEC 2a

<table>
<thead>
<tr>
<th>Level</th>
<th>Floor Life</th>
<th>Soak Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Standard</td>
</tr>
<tr>
<td></td>
<td>Time</td>
<td>Conditions</td>
</tr>
<tr>
<td>2a</td>
<td>4 weeks</td>
<td>( \leq 30^\circ C / 60% \text{ RH} )</td>
</tr>
</tbody>
</table>

- The standard soak time includes a default value of 24 hours for semiconductor manufacture's exposure time (MET) between bake and bag and includes the maximum time allowed out of the bag at the distributor's facility.
- Table below presents the moisture sensitivity level definitions per IPC/JEDEC's J-STD-020C.

<table>
<thead>
<tr>
<th>Level</th>
<th>Floor Life</th>
<th>Soak Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Standard</td>
</tr>
<tr>
<td></td>
<td>Time</td>
<td>Conditions</td>
</tr>
<tr>
<td>1</td>
<td>Unlimited</td>
<td>( \leq 30^\circ C / 85% \text{ RH} )</td>
</tr>
<tr>
<td>2</td>
<td>1 year</td>
<td>( \leq 30^\circ C / 60% \text{ RH} )</td>
</tr>
<tr>
<td>2a</td>
<td>4 weeks</td>
<td>( \leq 30^\circ C / 60% \text{ RH} )</td>
</tr>
<tr>
<td>3</td>
<td>168 hours</td>
<td>( \leq 30^\circ C / 60% \text{ RH} )</td>
</tr>
<tr>
<td>4</td>
<td>72 hours</td>
<td>( \leq 30^\circ C / 60% \text{ RH} )</td>
</tr>
<tr>
<td>5</td>
<td>48 hours</td>
<td>( \leq 30^\circ C / 60% \text{ RH} )</td>
</tr>
<tr>
<td>5a</td>
<td>24 hours</td>
<td>( \leq 30^\circ C / 60% \text{ RH} )</td>
</tr>
<tr>
<td>6</td>
<td>Time on Label (TOL)</td>
<td>( \leq 30^\circ C / 60% \text{ RH} )</td>
</tr>
</tbody>
</table>
# Qualification Reliability Testing

<table>
<thead>
<tr>
<th>Test Condition</th>
<th>Stress Conditions</th>
<th>Stress Duration</th>
<th>Failure Criteria</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Room Temperature Operating Life (RTOL)</strong></td>
<td>25°C, ( I_F = \text{max DC} ) (Note 1)</td>
<td>1000 hours</td>
<td>Note 2</td>
</tr>
<tr>
<td><strong>Wet High Temperature Operating Life (WHTOL)</strong></td>
<td>85°C/60%RH, ( I_F = \text{max DC} ) (Note 1)</td>
<td>1000 hours</td>
<td>Note 2</td>
</tr>
<tr>
<td><strong>Wet High Temperature Storage Life (WHTSL)</strong></td>
<td>85°C/85%RH, non-operating</td>
<td>1000 hours</td>
<td>Note 2</td>
</tr>
<tr>
<td><strong>High Temperature Storage Life (HTSL)</strong></td>
<td>110°C, non-operating</td>
<td>1000 hours</td>
<td>Note 2</td>
</tr>
<tr>
<td><strong>Low Temperature Storage Life (LTSL)</strong></td>
<td>-40°C, non-operating</td>
<td>1000 hours</td>
<td>Note 2</td>
</tr>
<tr>
<td><strong>Non-operating Temperature Cycle (TMCL)</strong></td>
<td>-40°C to 120°C, 30 min. dwell, &lt;5 min. transfer</td>
<td>200 cycles</td>
<td>Note 2</td>
</tr>
<tr>
<td><strong>Non-operating Thermal Shock (TMSK)</strong></td>
<td>-40°C to 120°C, 20 min. dwell, &lt;20 sec. transfer</td>
<td>200 cycles</td>
<td>Note 2</td>
</tr>
<tr>
<td><strong>Mechanical Shock</strong></td>
<td>1500 G, 0.5 msec. pulse, 5 shocks each 6 axis</td>
<td></td>
<td>Note 3</td>
</tr>
<tr>
<td><strong>Natural Drop</strong></td>
<td>On concrete from 1.2 m, 3X</td>
<td></td>
<td>Note 3</td>
</tr>
<tr>
<td><strong>Variable Vibration Frequency</strong></td>
<td>10-2000-10 Hz, log or linear sweep rate, 20 G about 1 min., 1.5 mm, 3X/axis</td>
<td></td>
<td>Note 3</td>
</tr>
<tr>
<td><strong>Solder Heat Resistance (SHR)</strong></td>
<td>260°C ± 5°C, 10 sec.</td>
<td></td>
<td>Note 3</td>
</tr>
<tr>
<td><strong>Solderability</strong></td>
<td>Steam age for 16 hrs., then solder dip at 260°C for 5 sec.</td>
<td></td>
<td>Solder coverage on lead</td>
</tr>
</tbody>
</table>

**Notes:**
1. Depending on the maximum derating curve.
2. Criteria for judging failure

<table>
<thead>
<tr>
<th>Item</th>
<th>Test Condition</th>
<th>Criteria for Judgement</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Forward Voltage ( V_F )</strong></td>
<td>( I_F = \text{max DC} )</td>
<td>Min. Max.</td>
</tr>
<tr>
<td><strong>Luminous Flux or Radiometric Power ( \Phi_V )</strong></td>
<td>( I_F = \text{max DC} )</td>
<td>Initial Level x 0.7 --</td>
</tr>
</tbody>
</table>

* The test is performed after the LED is cooled down to the room temperature.

3. A failure is an LED that is open or shorted.

---

**ProLight**

10
Recommended Solder Pad Design

- All dimensions are in millimeters.
- Electrical isolation is required between Slug and Solder Pad.
Reflow Soldering Condition

<table>
<thead>
<tr>
<th>Profile Feature</th>
<th>Sn-Pb Eutectic Assembly</th>
<th>Low-Temp. &amp; Pb-Free Assembly (58Bi-42Sn Eutectic Alloy)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Preheat &amp; Soak</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Temperature min (T_{smin})</td>
<td>100 °C</td>
<td>90 °C</td>
</tr>
<tr>
<td>Temperature max (T_{smax})</td>
<td>150 °C</td>
<td>120 °C</td>
</tr>
<tr>
<td>Time (T_{smin} to T_{smax})</td>
<td>60-120 seconds</td>
<td>60-120 seconds</td>
</tr>
<tr>
<td>Average Ramp-Up Rate (T_{smax} to T_P)</td>
<td>3 °C / second max.</td>
<td>2 °C / second max.</td>
</tr>
<tr>
<td>Liquidous temperature (T_L)</td>
<td>183°C</td>
<td>138°C</td>
</tr>
<tr>
<td>Time at liquidous (t_L)</td>
<td>60-150 seconds</td>
<td>20-50 seconds</td>
</tr>
<tr>
<td>Peak package body temperature (T_P)</td>
<td>235°C</td>
<td>185°C</td>
</tr>
<tr>
<td>Time (T_P) within 5°C of the specified classification temperature (T_C)</td>
<td>20 seconds</td>
<td>20 seconds</td>
</tr>
<tr>
<td>Average ramp-down rate (T_P to T_{smax})</td>
<td>6 °C/second max.</td>
<td>3 °C/second max.</td>
</tr>
<tr>
<td>Time 25°C to Peak Temperature</td>
<td>6 minutes max.</td>
<td>4 minutes max.</td>
</tr>
</tbody>
</table>

- All temperatures refer to topside of the package, measured on the package body surface.
- Repairing should not be done after the LEDs have been soldered. When repairing is unavoidable, a heat plate should be used. It should be confirmed beforehand whether the characteristics of LEDs will or will not be damaged by repairing.
- Reflow soldering should not be done more than two times.
- When soldering, do not put stress on the LEDs during heating.
- After soldering, do not warp the circuit board.
Notes:
1. Emitter 50 pieces per tube and Star 20 pieces per tube.
2. Drawing not to scale.
3. All dimensions are in millimeters.
4. All dimensions without tolerances are for reference only.

**Please do not open the moisture barrier bag (MBB) more than one week. This may cause the leads of LED discoloration. We recommend storing ProLight's LEDs in a dry box after opening the MBB. The recommended storage conditions are temperature 5 to 30°C and humidity less than 40% RH.
Precaution for Use

- **Storage**
  Please do not open the moisture barrier bag (MBB) more than one week. This may cause the leads of LED discoloration. We recommend storing ProLight’s LEDs in a dry box after opening the MBB. The recommended storage conditions are temperature 5 to 30°C and humidity less than 40% RH. It is also recommended to return the LEDs to the MBB and to reseal the MBB.
  - The slug is is not electrically neutral. Therefore, we recommend to isolate the heat sink.
  - Any mechanical force or any excess vibration shall not be accepted to apply during cooling process to normal temperature after soldering.
  - Please avoid rapid cooling after soldering.
  - Components should not be mounted on warped direction of PCB.
  - Repairing should not be done after the LEDs have been soldered. When repairing is unavoidable, a heat plate should be used. It should be confirmed beforehand whether the characteristics of the LEDs will or will not be damaged by repairing.
  - This device should not be used in any type of fluid such as water, oil, organic solvent and etc. When cleaning is required, isopropyl alcohol should be used.
  - When the LEDs are illuminating, operating current should be decide after considering the package maximum temperature.
  - The appearance, specifications and flux bin of the product may be modified for improvement without notice. Please refer to the below website for the latest datasheets.
    http://www.prolightopto.com/

Handling of Silicone Lens LEDs

Notes for handling of silicone lens LEDs
- Please do not use a force of over 3kgf impact or pressure on the silicone lens, otherwise it will cause a catastrophic failure.
- The LEDs should only be picked up by making contact with the sides of the LED body.
- Avoid touching the silicone lens especially by sharp tools such as Tweezers.
- Avoid leaving fingerprints on the silicone lens.
- Please store the LEDs away from dusty areas or seal the product against dust.
- When populating boards in SMT production, there are basically no restrictions regarding the form of the pick and place nozzle, except that mechanical pressure on the silicone lens must be prevented.
- Please do not mold over the silicone lens with another resin. (epoxy, urethane, etc)