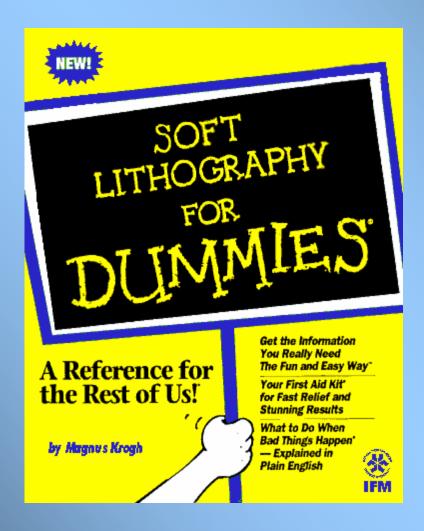
# My Little Guide to Soft Lithography

or



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# Soft lithography

Why? Soft lithography is an umbrella term for many methods designed to pattern materials like polymers. Soft lithography should be regarded as a complement to common lithography and has some possible advantages:

- Possibility to pattern UV sensitive materials without degrading the performance
- Possibility to pattern non-UV reacting materials directly
- Possibility to pattern non-planar surfaces
- Possibility to pattern large areas
- Possibility to control the chemistry during the patterning in a better way
- Possibility to generate 3D structures
- Ideally does not have any diffraction limit
- Short time between idea and prototype
- Possibility to clean room free operation
- Low cost
- Etc.

All the soft lithography methods will not be explained in detail here, since others have already done that much better than I could ever do. I suggest you read Xia and Whitesides soft lithography review. (G. M. Whitesides et al, "Soft Lithography", Angewandte Chemie Int. Ed., 1998, vol. 37, p. 550-575) The full review and a compressed one can be downloaded as a pdf-files at these URLs:

http://www.ifm.liu.se/~petas/mikrosystem/Links/Material\_files/SoftLithography\_AngewChem(1998).pdf

http://www.ifm.liu.se/~petas/mikrosystem/Links/Material\_files/SoftLithography\_AnnRenMaterRes(1998).pdf

As mentioned earlier there are quite a few different methods that are regarded as soft lithography methods. They often have an abbreviation that is not always obvious what it means. To simplify, here are some of them listed and also written in full text. Methods explained later in more detail are marked:

lift-up

μCP MicroContact Printing
 μTM MicroTransfer Molding

• MIMIC MicroMolding In Capillaries

• put-down

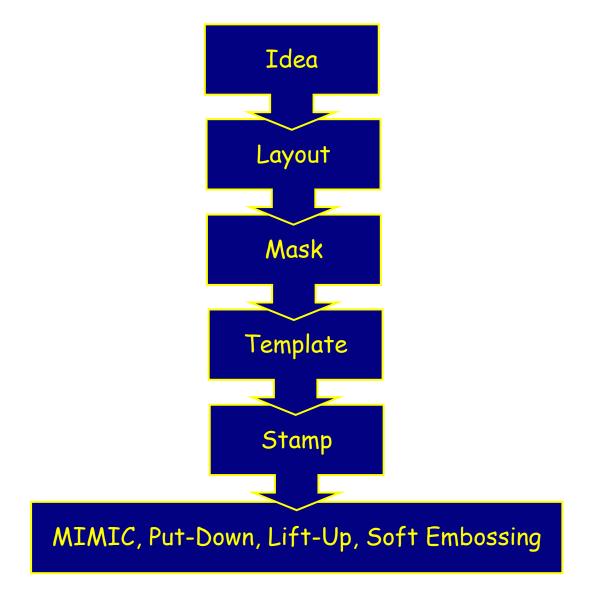
• REM REplica Molding

SAMIM Solvent-Assisted MIcroMolding

• SE Soft Embossing

Well... At least a few words have to be said about the methods used at the Biomolecular and Organic Electronics Group at Linköping University. A short description of the operating procedures from an idea to a finished pattern will be given.

The sections in this paper is ordered chronologically from idea to finished result:

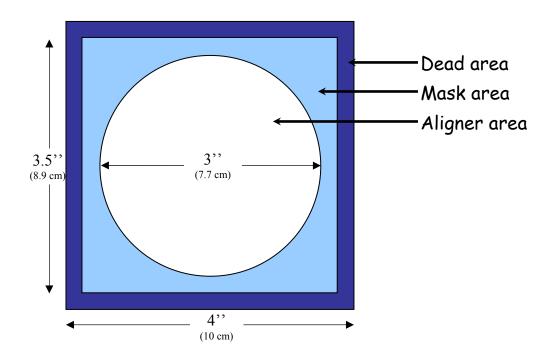


# Mask Cr

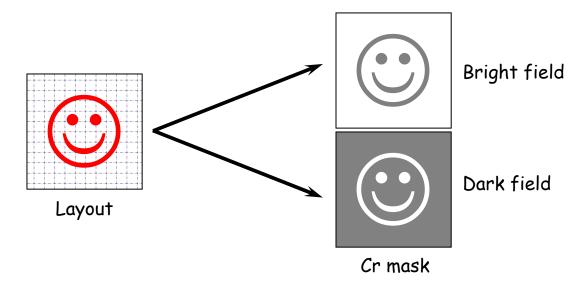
Why? The shadow mask consists of a glass substrate with a patterned layer of Cr. Cr is not transparent to UV light which makes it is possible to illuminate chosen areas of a substrate through the mask, e.g. common photolithography.

The first thing you need is an idea of what your structures should look like. Once you have an idea you simply draw it in a layout program like CleWin from WieWeb (www.wieweb.com) or a CAD program of your choice. However, if you are *not* using CleWin, make sure that *cif* (Caltech Intermediate Fileformat) or GDSII file format is supported, as this is the format that you need when ordering the mask. Programs like linkCAD from BayTech (www.bay-technology.com) can however convert from common formats to cif. Cr masks can be ordered from DeltaMask in Holland (www.deltamask.nl) and it will take one to two weeks to get it. When making your layout there are a few useful things to remember:

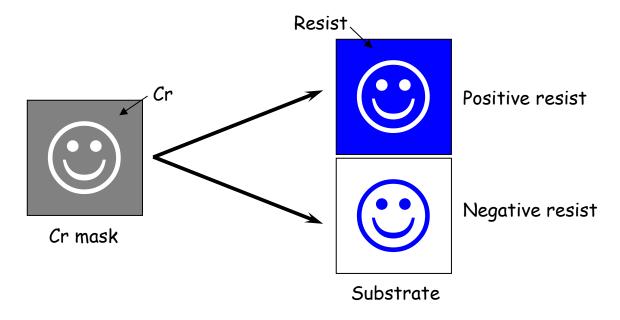
- Include your name, i.e. a label, and year (or date). If you for some reason misplace your mask, it is then possible to trace the owner you! It can also be useful to know how old it is if you have several similar masks.
- Alignment marks are often useful. An alignment mark does not have to look like a +. In fact, it is better to make an L or preferably even a bit more inventive than that, since then it is harder to place the mask upside-down or have it 180° rotated in the mask aligner. Make sure that he alignment marks are not too far from the center since the aligner microscope can only move over a certain area and make as many as possible, e.g. every 15 mm.
- Scaling. If you are going to use the mask to make silicone stamps, you have to scale up your structures since the stamp material will shrink a few percent during the cure. How much it will shrink you can find in chapter "Stamp".
- The Cr mask is in general a 4 inch square, but the useable area is a bit smaller. Further, the aligner can in general only use a circular area that is even smaller.



• When you order the mask you can specify it as bright field or dark field (order from Wim Bouwens). Bright field means that the Cr mask will look exactly like your layout, while dark field means that you will get the negative. This can simplify the drawing remarkably. Help lines/squares could also be of help when making the layout. Just remember to remove them when the layout is finished. Tip: Designate one layer for help figures.



• You should also remember to check if the photo resist you will use is a positive or negative resist. In a positive resist areas exposed to light will be removed in the development and in a negative resist they will be the ones remaining. SU-8 is for instance a negative resist, meaning you should in general have a dark field mask. It is possible to make a negative mask of the original mask, but it will be mirrored and it will take some time. In other words, check twice that you have everything the right way.



Useful links:

Download a cif layout template here:

http://www.ifm.liu.se/~petas/mikrosystem/Links/Material files/TEMPLATE.CIF (Remove layer 15 when finished!)

# Template SU-8

Why? To be able to mould a stamp you need a master/template to mould it from. A cheap and quite fast way is to provide a Si wafer with epoxy structures by common lithography.

In our lab the template usually consists of a 4 inch Si wafer as carrier substrate with epoxy structures on top. The epoxy is called SU-8 or XP SU-8 which stands for ??? (= tell me). The process of creating the structures is not that hard and will take about one hour. The steps are resist spin, pre-exposure bake, exposure, post-exposure bake and development. I suggest you do as follows:

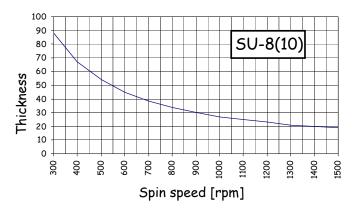
# Substrate preparation/dehydration bake

To get good adhesion between the Si wafer and the SU-8 resist the wafer needs to be clean. Cleaning is done by a TL1 wash, which means that the Si wafer is boiled in a mixture of  $H_2O$ ,  $H_2O_2$  and  $NH_3$  (5:1:1 by volume) for 10 minutes and then rinsed in de-ionized water a few times. Before spin coating the wafer with SU-8, it is placed in an oven (110°C) for 15 minutes. This is done to remove any water on the surface that would reduce the adhesion. It is good idea at this point to turn on the hot plate (100°C) and the UV lamp, since it takes ~15 min for them to heat up.

# Resist spin

SU-8 photoresist is manufactured in different grades, determined by the amount of solids with respect to the solvent. A number behind the 'SU-8' indicates what grade a certain SU-8 has, e.g. SU-8(2), SU-8(5), SU-8(10)... 2 has the lowest viscosity and 500+ the highest. Since the viscosity differs, the spin speed has to be adjusted accordingly. MicroChem Corp. (www.microchem.com) has a bulletin board where characteristic thickness at different spin speeds (example below) and some other information about SU-8 can be found. Thickness from less than 1 μm (ask Tobias Nyberg, tobny@ifm.liu.se) to several hundred μm can be achieved. As a rule of thumb, use about 1

ml of SU-8 per inch diameter of the wafer. The volume of the dispense will effect the film thickness somewhat. The resist is dispensed at the center of the wafer and spread at 500 rpm for ~15 s. If thicker grades are used it is necessary to spread the film longer. After spreading comes the actual spin with spin speed according to a tabulated value. (3 ml SU-8(10), 500rpm/15 s + 2000 rpm/15s  $\rightarrow$  ~20  $\mu$ m). Ask other group members for start parameters.



# Pre-exposure bake / soft bake

The wafer with the SU-8 film needs to be baked prior to the exposure. This is done to remove any remaining solvent and stabilize the film. The pre-exposure bake also makes the surface non-sticking, which prevents it from leaving remains on the Cr mask. The pre-exposure bake is done on a hot plate at  $100^{\circ}$ C for ½ to ½ min per micrometer of film thickness. For really thick films this can take several hours. After the bake the resist needs to cool off before exposure. Minimum cooling time is ~5 min (10-30  $\mu$ m film thickness). From this point and forward; avoid using tweezers and sharp objects, since they may scratch the film.

#### **Exposure**

SU-8 is a negative resist, meaning exposed areas will cross-link and remain after development. The exposure time/dose is depending on resist thickness. SU-8 can be over-exposed although it is not common. A  $\sim$ 30 s exposure is in most cases sufficient in the 10-30  $\mu$ m range of film thickness. Remember to put the Cr side of the mask *towards* the film.

# Post-exposure bake

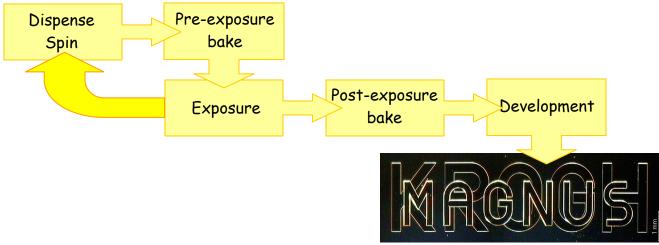
The post-exposure bake continues the polymerization process and is done on a hot plate at 100°C for 15 min. During the bake the pattern starts to stand out and the film seems to become "bubbly". Don't worry! The bubbles are never *in* your structures, always between them. After the post-exposure bake, let the film cool off. Minimum cooling time is (again) ~5 min.

#### Development

The final step is to develop the film. This is done by placing the wafer with the film in a SU-8 developer (ethylene glycol monomethyl ether acetate) bath for a few minutes. You will see when the pattern appears (it is hard to over-develop). The template is then rinsed in developer and blow-dried. Note: the developer is highly toxic (www.niwl.se/ah/1999-13.pdf) and should be kept in a well-ventilated area at all times. Finally check for remains in a microscope and measure the thickness. It is possible to remove small remains in a descum procedure and the film can also be made more solvent resistant by a final hard bake, however SU-8 is in general very solvent resistant.

### Multi layer structure

It is possible to stack several SU-8 layers on top of each other. Simply stop after the exposure and spin the next layer on top.



#### Silanisation

PDMS (polydimethylsiloxane) will stick to SU-8 unless the template is silanised. The silanisation *must* be done in the designated room and equipment otherwise it could contaminate large areas of the building. The silanisation is done like this:

- Put the template in a mixture of 50 ml xylene and 1-2 pipette tip of dimethyl-dichlorosilane (DDS) for ~5 minutes. Note: Trichloroetylene is highly cancerogenic.
- Rinse in xylene three timesl
- Blow-dry or let it dry in the hood.

#### Useful links

IBM research info on XP SU-8: www.research.ibm.com/journal/rd/411/shaw.html

General info on SU-8: aveclafaux.freeservers.com/SU-8.html

Possibilities of SU-8: www.maxlab.lu.se/beamlines/bld811/results.html

Medical safety data sheet on SU-8 developer: camd.lsu.edu/msds/x/xp\_su8\_developer.htm

# Stamp silicone

Why? Well... you need it if you want to do soft lithography!

The stamp is the key in soft lithography since it is used to generate the pattern. The stamp is made of silicone, also called PDMS (polydimethylsiloxane), Sylgard 184 or rubber. PDMS is it's chemical name while Sylgard 184 its commercial name. Rubber is however a bit misleading. Dow Corning Sylgard 184 (www.dowcorining.com) is transparent and has low viscosity as uncured, both favorable features when making stamps and using them for soft lithography. Sylgard 184 has a very low resistance to most non-polar solvents. The stamp will in general not be destroyed by the solvents, rather deformed by swelling and will regain its original shape once the solvent has evaporated. Well, it can at least withstand water for some time. ;-) If you like to have a stamp material that will not swell by solvents I suggest you try fluorosilicone instead. To mould a stamp from a template you do like this:

### Weigh

Sylgard 184 is a two-component heat-curing system, i.e. it consist of a base part and a curing agent part. Take a common plastic cup and fill it with *one* part curing agent and *ten* parts of base (by weight). Start with the curing agent, since it is harder to pour the right amount of it! 7-10 g material will be sufficient for covering one template, e.g. 0.7 g curing agent and 7 g base. A small error in the amounts will not effect the final result though.



#### Mix

Use a plastic spoon to mix the base and the curing agent. Mix it rather carefully for at least a few minutes, depending on the amount of material. When you mix it you will incorporate a lot of air in the solution. Don't worry – it will be removed in the next step. Use a plastic spoon to mix it.



# Degas

After the mixing the silicone mixture will be full of air bubbles and needs degassing. This is done in an exsicator using vacuum. During the degassing the silicone expand and start to look like foam, this means that you can only have a small (<5 g) amount in each plastic cup else it will overflow. Also remove the spoon. When the silicone is completely clear and transparent it is finished.



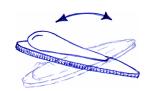
#### **Dispense**

Dispensing the silicone on to the template can be a bit tricky, as you do not want to trap air in the process. Sylgard 184 has relatively low viscosity, so the flow is no problem. It is also possible to make the viscosity even lower by mixing in silicone oil or hexane, but this is seldom necessary. Dispensing the material at the center of the template from a low altitude minimizes the risk of trapped air. Keep the template horizontal during dispense.



### **Spread**

Pick up the template with a pair of flat tweezers and start tilting it at a low angle. The material will now start to spread. By tilting it in different directions it is possible to cover the whole template by silicone. Try to make the shape as circular as possible. When you think it is ok, leave it for a minute in order to get a flatter top surface. Stamps thickness can range from ~0.1 to 5 mm or more.



#### Curing

Sylgard 184 is heat curing. It is curable from less than room temperature to over 150°C. Sylgard 184 also has temperature dependant shrinkage as seen below. Curing in 140°C (~15 min) will make the stamp shrink almost exactly 3 %. Take the template and place it in a pre-heated oven. The time is not that critical, it is almost impossible to cure it a too long time.



# Sylgard 184

### As supplied

Mix ratio A:B 10:1

Viscosity (mixed) 3900 centipoise

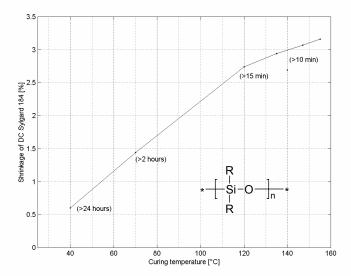
Specific gravity 1.05 Pot life (25°C) 2 h

#### As cured

Appearance Transparent
Durometer hardness 40 Shore A
Tensile strength 6.2 MPa
Elongation 100%
Surface state Hydrophobic

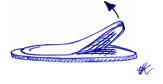
Water adsorption 0.1%, (7 d immersion)

Refractive index 1.430 Dielectric constant 2.65-2.7



# Peal off

The final step is to peal off the stamp from the template. Use a sharp and curved pair of tweezers to do this. Start by releasing all borders and then continue at a low speed in a direction parallel to most structures and peel off the remaining parts. Store the stamp in a flat plastic container with the pattern facing upwards, i.e. away from the container. Finished!



### Negative stamp

It is possible to make a negative stamp of an existing stamp. Simply use the original stamp instead of the template and cure it at high (e.g. 140°C) temperature. If you cure at a low temperature the two parts will cross-link and you will not be able to separate them later on. Note that the stamp will shrink once more, e.g. leaving a negative stamp that is for instance 3 % + 3 % smaller than the original template.

#### Making PDMS hydrophilic / Bonding PDMS

It is simple to make PDMS hydrophilic, simply run it in the plasma preparation chamber for  $\sim 15$  s. The plasma preparation will incorporate oxygen atoms in the PDMS surface, leaving it hydrophilic. This can also be used to bond PDMS to for instance glass, Si or even another PDMS stamp. What you should do is to plasma prepare *one* of the substrates (*not* both!) and place them in contact for a few days. The bond is very strong and if you try to separate them you most certainly will break the stamp. A tip if you like to remove them again after they have bonded is to place it in water for a while. This will usually break the bond. PDMS can also be made hydrophilic by a mixture of  $H_2SO_4$  and  $KMnO_4$ . (Note: It works poorly!)

#### Useful links:

All about silicone chemistry: http://www.ifm.liu.se/~petas/mikrosystem/Links/Material.htm

Sylgard 184 data sheet: http://www.ifm.liu.se/~petas/mikrosystem/Links/Material files/DC Sylgard 184.pdf

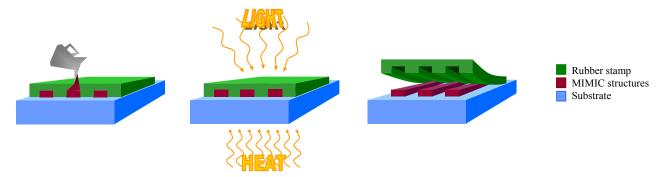
Some pdf files: www-personal.engin.umich.edu/~yens/project.html

Applied silicone: www.baywatch.com

# MIMIC polyurethane, epoxy...

Why? MIMIC is used when you want really high and sharp structures, e.g. insulating cathode separators.

MIMIC is an abbreviation for MIcro Molding In Capillaries and are capable of generating structures down to a few micrometer. What you do is that you place the stamp on a substrate with the relief facing towards the substrate. This way capillaries are formed. Next step is to fill these capillaries with a material like polyurethane or epoxy. The capillary force will then try to fill the capillaries.

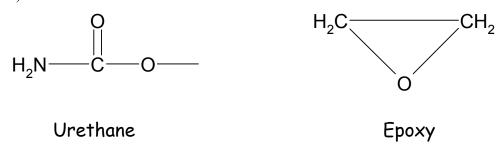


The fill rate is dependent on quite a few parameters as seen in the equation below (E. W. Washburne, "The dynamics of capillary flow", Phys. Rev, **1921**, Vol. 17 (No. 3), p 273-283). This equation is valid for cylindrical and horizontal capillaries rather than the quadratic ones in MIMIC. The behavior is however similar. t is the fill time,  $\mu$  viscosity, r radius,  $\Delta P$  pressure difference at the capillary openings,  $\gamma$  surface tension,  $\theta$  contact angle and l is the fill length.

$$t = \frac{4\mu l^2}{r^2 (\Delta P + \frac{2\gamma \cos \theta}{r})}$$

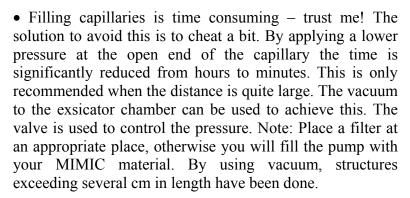
The third step is to cure the material inside the capillaries either by UV or heat. It can also be done chemically by using a curing agent. Finally the stamp is removed. If possible, try to remove the stamp in the same direction as the main part of the structures.

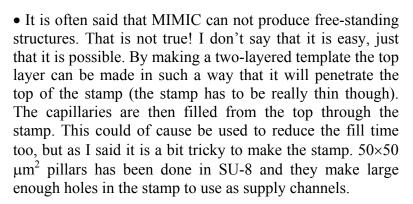
Two materials that work good are polyurethane and epoxy. Polyurethanes has the advantage of being able to fill longer distances while epoxies have in general good adhesion to the substrate and are usually a bit harder. Both material types generate high and sharp structures down to <5 µm. Curing is done by either UV, heat or as 2-component cure system. The curing time is product dependent but is in most cases longer inside the capillaries then outside. Two polyurethanes with good properties are NOA83H and NEA121 from Norland Products (www.norlandprod.com). (Note: You need a special permit to keep urethane in the lab but not polyurethane as far as I have understood)

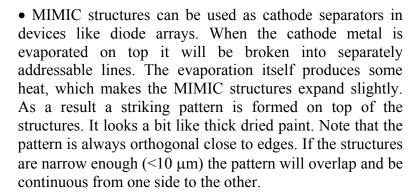


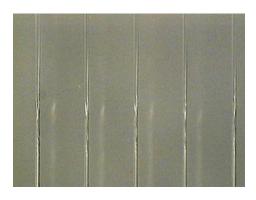
# A few useful MIMIC tip:

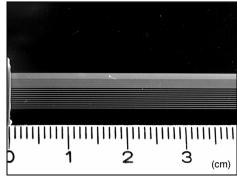
- Let the stamp settle. By leaving the stamp on the substrate for a while trapped air has time to diffuse through the stamp and the stamp will be better bonded to the substrate. This means that the risk of leakage is reduced. This is especially effective on polymer substrates like P3HT and PEDOT-PSS.
- UV curing polyurethane, e.g. NOA83H, has a problem with catalyst diffusion into the stamp. This means that it is only possible to cure a short distance (top part in figure), the rest will remain soft. One solution is to saturate the PDMS stamp by the catalyst *prior* to the filling by placing polyurethane on top of the stamp for a few hours. Another way is to change material to a 2-component system, where the curing agent hopefully will not diffuse into the stamp. However, the best way to deal with this problem is to reduce the fill time by using vacuum aided filling.

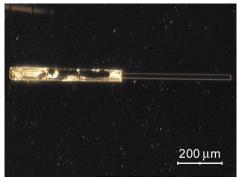


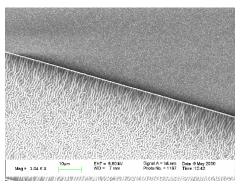












# Put-down PEDOT-PSS

Why? Put-down has mainly been used to pattern a polymer called PEDOT-PSS. It is possible to pattern large areas on for instance glass and ITO.

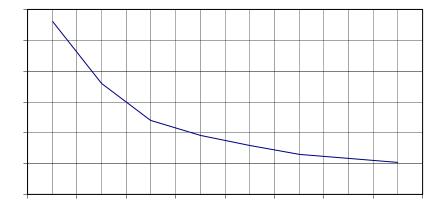
PEDOT-PSS is generally the material used with put-down. Some information about PEDOT-PSS:

Content – PEDOT-PSS is a water dispersion with two parts PEDOT (poly(3,4-ethylenedioxy thiophene)) and PSS (poly(4-styrenesulfonate)). PEDOT (i.e. PEDOT<sup>+</sup>) is a conducting part (0.8 % solid content by weight), while PSS (0.5 %) is more or less insulating. Since it is a water dispersion, the surface that it should be applied to must be hydrophilic. The solution has a very low pH value of 1-2. As a result PEDOT-PSS will etch some metals, e.g. Cu.

Chemical properties – PEDOT is a polythiophene derivate that can be doped by the polymer PSS, meaning a sulfur atom in the PEDOT will oxidize and give one electron to PSS. As a result some of the PEDOT monomers (up to ~25 %) on the chain will become PEDOT<sup>+</sup>. PEDOT-PSS has a very high workfunction (5.2 eV) and a band gap of 1.6 eV. The high workfunction makes it suitable as a hole injecting/extracting layer in LED:s/photo diodes, e.g. between ITO and the semiconducting polymer.

Conductivity – PEDOT-PSS is a conducting polymer with a conductivity of ~50 S/m, but it can be increased by a factor of over 400 by adding glycerol or sorbitol. More information about this phenomena can be found in "Optimising the Conductivity in PEDOT PSS by adding glycerol and sorbitol" by M. Karlsson and M. Krogh (IFM, Linköping University, 1999) and "An investigation of ways to improve the electrical conductivity of a conjugated polymer, PEDOT" by S. Jönsson (IFM, Linköping University, 2000). Note: This have shown to degrade the performance of for instance diodes.

PEDOT-PSS thickness vs. spin speed



The put-down process is quite straightforward and consists of basically three steps:



# Applying the film

The film is applied on to the PDMS stamp by some method, e.g. dip, spin or spray coating. If PEDOT-PSS is used the stamp must be hydrophilic. This is done by a 15 s plasma preparation. The thickness is highly dependent on the pattern size and the pattern fill factor, at least in dip and spin coated stamps. Small structures/low fill factor gives thinner films compared to larger structures/higher fill factor. This problem should be possible to avoid by using spray coating.

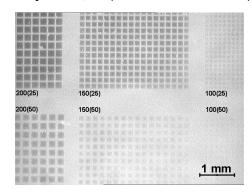
# Applying the stamp

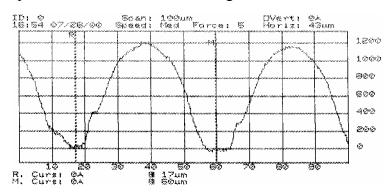
The film quickly dries and when it is dry it will not adhere to glass or ITO. Moisturizing the film/substrate is necessary before the stamp is applied. This can be done several ways. A good way is to place the substrate (hydrophilic surface) in a fridge for a while, taking it out and letting some water condense. This will give an even water film that will last longer than one made by hot steam. A quicker way is simply to breathe on the stamp. The amount of water is the critical point; too much and the PEDOT-PSS will smear out, too little and you will not stick to the substrate. The stamp is than placed on to the substrate making conformal contact. When in contact at one place, the contact area will spread, making the whole stamp come in contact with the substrate. If possible, do not put any pressure on the stamp, the weight of the stamp is usually sufficient.

### Removing the stamp

When the stamp has been applied you have to wait. 20 min should be sufficient. If it takes more than 30 min to dry the amount of water has to be reduced, otherwise your structures will be poor. A common defect with a too wet film is high walls of PEDOT-PSS surrounding the actual pattern. "Snap" sounds when removing the stamp is an indication of this problem. The stamp is removed with a constant speed at a low opening angle ( $\sim 30^{\circ}$ ).

Put-down structures are in general quite sharp when seen from above, the cross-section is however far from sharp. It is rounded; shaped more or less like a part of a circle. This can perhaps be avoided by spray coating a number of ultra thin layers on top of each other. The surface roughness of the PEDOT-PSS put-down structures is rather rough. Put-down structures are capable of making small structures but works better with larger ones. The separation between the structures can be very small; 40 µm structures with 5 µm separation have been done on a large area.





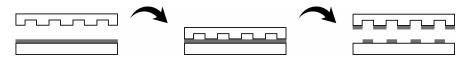
Useful links:

Conformal contact: www.ifm.liu.se/~magkr/Conformal\_Contact\_and\_Pattern\_Stability\_of\_Stamp\_(IBM\_Zurich).pdf PEDOT-PSS data sheet: Ask Magnus Krogh (No computer file available)
Optimization: www.ifm.liu.se/~magkr/Optimising the Conductivity in PEDOT PSS (Krogh Karlsson).pdf

# Lift-up PEDOT-PSS

Why? Another method for patterning PEDOT-PSS that gives nicer structures than put-down but is more difficult to perform. Lift-off would perhaps be a better name.

In lift-up one basically do everything the opposite way compared to put-down. The stamp have an inverted pattern, the film is applied on the substrate instead of on the stamp and removing unwanted parts rather than adding them forms the pattern. There are however still three main steps:



# Applying the film

After plasma preparing the substrate and the stamp (~15 s) a PEDOT-PSS film is spin cast on to the substrate, e.g. glass. At this point it is possible to manipulate the film to achieve preferred properties.

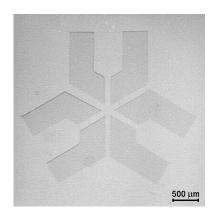
# Applying the stamp

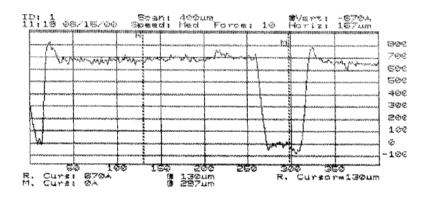
The film is left to dry a few minutes and than remoisturized. As in the case of put-down this can be done several ways, but the crude method of breathing on the substrate seems to work best. The water content when using lift-up has to be rather low, lower than with put-down. If the film is too wet droplets of PEDOT-PSS will form around the structures rather than follow the stamp. If the film is to dry, nothing will happen or the stamp will merely leave an imprint in the film.

# Removing the stamp

The time between applying the stamp and removing it again is short, about 10-20 seconds. The film may not be completely dry when the stamp is removed since than the PEDOT-PSS will remain on the substrate. As a rule of thumb can be said that PEDOT-PSS adheres best to the stamp when it is wet and to glass when it has dried out. Note: Lift-up on ITO has never worked properly.

Put-down structures are in general both sharp when seen from above and in cross-section. Sometimes a small tip will remain at one of the edges as seen in the left part of the cross-section figure below. A pattern with large separations is in general easier to produce than with a small separation. The outcome does not seem to be significantly dependent on the actual structure size. 20 µm separation has been produced a few times.





# Soft embossing Semiconducting polymers...

Why? Soft embossing is used when you need to pattern a surface with structures with dimensions around one micrometer (or less). Used to make diffraction grids, make ordered polymers...

Soft embossing has mainly been used on semiconducting polymers, but also some on SU-8 and polyurethane. Note that soft embossing always leaves a thin layer between the actual structures. In some applications this is not a problem, but often it is. The description of soft embossing will be quite short since I basically never have used it and the different operation procedures are highly material dependent. The three basic steps:



# Applying the film

The film is applied and treated appropriately. The material used has to have such properties that it is possible to make it soft by some means, usually heat.

# Applying the stamp

In soft embossing the stamp generally does not need to be plasma treated. The stamp is placed in conformal contact with the film. The substrate is heated to make the film soft while a uniform pressure is applied on the stamp. Most of the material will then be pushed into the relief of the stamp.

# Removing the stamp

Next step is to cool the film, still applying the pressure. The material will then "freeze" in its current position and when the stamp is removed a pattern will remain.

Soft embossing only works with small dimensions with a rather uniform fill factor around 0.5. Sub micrometer structures has been done at Linköping University using soft embossing.