

1. Overview

In this lab we will fabricate a model organic optoelectronic device, with the goal of developing the basic concepts of organic device fabrication that are general to most devices. We will demonstrate the techniques of ITO patterning, substrate dicing, substrate cleaning, thermal evaporation, shadow masking, and packaging, and finally show you that the device does indeed glow with a 9V battery! (If you are really nice to your TA, you may even get to take one home with you...) More extensive and quantitative testing of similar devices will be left to Lab 3.

2. Sample Preparation

The device structures are shown in Figure 1. All of the chemical structures and complete names are given in Figure 2. We will make this device starting with a 14" square piece of ITO coated glass, and processing all the way to a complete packaged OLED substrate with as many as 10 (mega)pixels.

a) Creation of Patterned ITO substrates (1" squares)

Scribe

We buy glass sheets, 14" squares, that are precoated with ITO (Indium Tin Oxide, our transparent conducting anode), rated at 20 Ohms/square. However, all of our processing techniques can only handle 4.25" squares, and so we must scribe and dice the 14" pieces by hand. A practiced hand can get very good at this, but we will see how well our **first volunteer** does.

Pattern

Our goal is to pattern individual 1" squares, but for ease of processing, we actually first pattern a 4.25" sheet, and dice it down to 1" squares afterwards. The patterning process that we currently use in our lab is to use plating tape as an etch mask, and to pattern the tape using the dicing saw (limited to 1D patterning). Since this step is not very interesting, and time consuming, we will just show you the end result, and demonstrate the acid etch. **Volunteer 2** will place a thin strip of plating tape on a piece of scrap ITO. This taped over area will be the only non-etched portion, and the rest of the ITO will be stripped away to bare glass.

Etch

The etching itself is a simple one step process. We take the ITO coated glass, protected by plating tape, and dip it into the acid etch. Our etch mixture is 300mL deionized water, 225mL hydrochloric acid (HCl), and 75mL nitric acid (HNO₃). This is not a particularly nasty etch compared to other standards, but I would get into trouble if you all burned yourselves, so I will handle this step. The etch time for our ~150nm thick ITO is 15 minutes under vigorous stirring at room temperature. We then rinse off the acid under DI

water, and a **third volunteer** can peel off the plating tape, and measure the resistance of our ITO strip using an ohmmeter.

Dice

We are now ready to dice our 4.25" patterned square into 1" substrates. To do this we will use a Microautomation Dicing Saw (which is fun to note that we bought at auction from a going out of business start-up company a year or so ago). The process here is complicated, though the concept is simple. A thin diamond grit resin blade is used to literally slice through the glass. Precise motor controls allow this machine to have 0.0005" resolution in the vertical direction (half of one mil, 13um), a similar resolution in the glass plane. Most important to us is the z-resolution, since we need to cut entirely through the glass (43mil thick), but only half way through the backing tape (3mil thick) so as not to shatter the fragile blade on the vacuum chuck.

The procedure is to apply the backing tape, program the saw, perform alignment to our ITO patterns, and then press "GO". To save this half hour, I will simply show you the before and after shot of the taped glass, and then the diced glass, and possibly demonstrate a single cut just so that you believe me that this is possible.

Cleaning

Next we must clean off our 1" squares of patterned ITO, to insure repeatable, low resistance ITO/organic interfaces. This process takes about an hour, though it is possible to clean as many slides as you may need in that amount of time. The substrates are first soaked in a water based detergent while undergoing sonication for 5 minutes; rinsed with DI water; sonicated in DI water; rinse and sonicate again; sonicate in Acetone; wash in boiling trichloroethylene (TCE) twice; wash in sonicating acetone twice; wash in boiling isopropanol twice; sheet off remaining solvent with clean wipe and nitrogen blowgun; UV ozone ash for 5 minutes. I will set all of this up for you, and a **fourth volunteer** will use tweezers to quickly run through the entire process so that we can see the sparkling clean results.

b) Transfer

Substrate Loading

As soon as we have UV ozone cleaned our substrates, we want to protect them from absorbing too much water vapor and oxygen on the surface. We therefore to all subsequent handling in either nitrogen or vacuum. A **fifth volunteer** will pass in a substrate through the left glove box antechamber, cycling the pressure three times.

A **sixth volunteer** will now load the substrates into the substrate holder. This will be done in the glove box, and so requires some skill. A vacuum pen can be used to handle the backside of the substrates without affecting the device performance. Once the substrates are loaded (maximum of nine in this holder), a backing plate is screwed on to hold them in place. The substrate holder can now be loaded into vacuum. To do this we must vent the load lock to atmospheric pressure of nitrogen. This involves playing with some neumatic valves (**volunteer seven**) that close off the pumps and the rest of the

transfer line, and then open up a nitrogen line. For more detailed instructions on any system use, see the additional instruction sheets. Once the loadlock is at atmosphere, our **sixth volunteer** can place the substrate holder (I call this the puck sometimes) onto the loadlock caboose and reseal the door.

Loadlock pumpdown

A **eighth volunteer** will now rough out the loadlock, and then switch over to high vacuum and pump it all the way down to 10^{-7} torr, over 9 orders of magnitude lower pressure than was there just minutes ago. We then open the loadlock to the rest of the transfer line (more pneumatic valves), and are ready to perform linear transfer.

Linear Transfer

This system is designed to allow linear transfer of our puck to any of up to eight chambers, of which there are only two in place now. The concept is simple engine and caboose running along a set of parallel rails. Operating the system requires a series of very simple mechanical manipulation from outside of the chamber, and will be done by a **ninth volunteer**, listening carefully to my instructions.

Lateral Transfer

Once the puck is positioned in front of the thermal evaporator, a **tenth volunteer** will follow my instructions, and manipulate the puck into the evaporator chamber. Once the puck is installed and rotating, and the chamber is closed off from the transfer line, we are ready for organic growth to begin.

c) Growth

Organic Growth

Each lab group will grow one of the three structures of figure 1, but in the end, everyone will have the opportunity to see each of the devices in operation. Each layer will be grown by a separate **volunteers** (with the exception of doped layers), listening carefully to my instructions. Detailed instructions on the use of the thermal evaporator are provided in the attached instruction sheets. The basic organic growth procedure involves selecting the appropriate boat to grow from, and then increasing the current driven through that boat slowly while monitoring the thickness monitors. When a steady rate of 1-3 Angstroms/s is achieved, the substrate shutter is opened, and deposition begins. When the full thickness is achieved, the shutter is closed, and the source current is shut off. The growth of the next layer may then begin.

Shadow Masking

Before depositing the metal cathodes, a shadow mask must be applied to define their pattern (see Fig 4 for mask patterns). Our system allows for this to be done in vacuum, again with the simple manipulation of a few external mechanical knobs. A **volunteer** will attempt this, though I expect that I will have to finish the procedure as it is somewhat tricky. The masked substrates are then returned to the thermal evaporator for metal deposition.

Metal Deposition

This follows in the exact same way as the organic growth, except that much higher temperatures are required (you can actually see the silver boat glow red-hot if the windows are opened during growth). I will deposit these layers, to keep things moving along. The transfer procedure then proceeds in reverse (again, I'll do it to show you how quick this can be), and we move the substrates over to the right glove box for packaging.

d) Packaging

A **final volunteer** will perform this simple operation. A syringe loaded with epoxy is already in place in the box. Two foot pedals control the pressure and vacuum that is used to push the epoxy out onto our cover slip (this was cleaned using the same cleaning procedure as our substrates went through). A square of epoxy is drawn around the edges of the cover slip, and then the device substrate is lowered onto this, and pressed tight to make sure that a complete seal is achieved. The sample is then pushed under the ultraviolet curing lamp, with is turned on in three 30 second blasts to fully cure the epoxy. The sample may then be removed from the nitrogen environment, and tested with a 9V battery. Under forward bias, your devices should emit blue, green and red light!

3. Discussion Questions

All of the quantitative device testing will take place during lab #3; as such the following questions are purposefully qualitative. As always, simple answers are usually the best answers.

- a) Why did we use thermal evaporation to make these two and three layer devices, rather than any of the other growth techniques that we have discussed (and demonstrated)?
- b) What color should each of the devices (a-c) emit?
- c) Are the devices transparent, and why?
- d) What color light do you expect to be useful in creating a photovoltaic effect in these devices?
- e) Why did we need to pattern the ITO to make these devices? The metal? Why didn't we pattern the organic?
- f) What improvements would you suggest to such a procedure? This could be in terms of future labs, or my own experimental setup...