Sample Preparation Basics:

The following are some general notes, which apply to typical cases and may differ significantly from your sample requirements.

Substrates:

We typically use Si, Si-SiO_2, Mica, and glass as substrates. Si is what we use most of the time and Si has a thin native oxide layer, Si-SiO2 has a thick layer of SiO_2 evaporated on it and is useful for some dewetting studies. Most of the time we use Si - the Si with the thick oxide layer is rather expensive and should only be used if you need that for a specific reason. Mica is great for floating films onto water (to be picked up later when making multilayers or free-standing films). Glass microscope slides are also a good substrate for floating films, but in less critical applications. Many polymers will float off of glass slides, which are nice and cheap, but mica is the best for floating and of course, it costs more.

When cleaving Si, make sure you use the [100] type. This means that it has been cut along the 100-axis and will cleave easily into squares. In some applications the [111] Si is best, but this is cut along the 111-axis and must be cleaved into triangles! When preparing to cleave, it is crucial that you keep your work environment as clean as possible. So clean your tools – tweezers, scribe, ruler, using a kimwipe or lens tissue with some acetone. Also clean off your workbench, and *always* wear gloves. Pay attention to things like hair hanging over your samples.

We cleave into 9 mm x 9 mm squares since most of our sample holders are based on a 10 mm x 10 mm sample size (there is some paper with an appropriate grid for this). The cleaving should be done in the laminar flow hood while wearing gloves and using tweezers. Take a Si wafer out of the box very carefully, place with the good side down onto a lens tissue (NOT kimwipes). It is best to handle the lens tissue using tweezers. Close the box immediately to avoid contamination of all the wafers. Place the Si wafer onto a piece of paper with a 9 mm grid on it, still retaining the clean lens tissue in between. This should be done so that the flat edge of the Si is lined up along the grid – the flat edge designates the [100] direction. Make sure that you do not slide the wafer over the lens tissue since this may cause tiny scratches on the surface of the Si. Now you can begin to scribe lines on the back surface of the Si with the diamond pen and a ruler so that you match up with the grid. If you do not scribe exactly parallel and perpendicular to the grid (and the [100]) then it is very hard to cleave the Si. Apply gentle pressure: enough to scribe, but not so much that you break the Si (getting this right takes some practice). Once the grid has been scribed, place the Si on a different lens tissue. Note that the lens tissue is covered by very fine Si dust – this is horrible for the samples and the reason that you need to replace lens tissue frequently. The Si should *never* be placed onto lens tissue (or part of the lens tissue) that has been previously used. Now you are ready to cleave the Si. There are several ways, but I find it easiest to place a straight metal wire under the tissue and Si, directly under a scribed line. Then a bit of pressure on either side will cleave the Si. Because each cleave results in some Si dust, place the

cleaved strip onto a new lens tissue out of the way, while you continue cleaving the other bit which must also be placed onto a new tissue. Continue this way until you have a bunch of Si strips, always ensuring that you either use fresh lens tissue or that you use a clean part of the lens tissue that has not been contaminated.

The strips are easy to cleave in the same manner into small squares. For this part you can use the same lens tissue as long as you place the strip onto a part of the tissue that has not been used before. At each stage of the cleaving, it may be helpful to gently remove the dust from the Si pieces, using the air gun, which is attached to clean argon or nitrogen gas. Keep in mind that you are blowing dust around, so make sure that you direct it away from your clean Si, lens papers, tools, and work area, and off the bench. Be careful not to blow your Si pieces over, or off the table (I have done this many times!)

Take all the Si squares and place them into a tray with the good side up and make sure that you write your name on the tray so that you know which samples are yours. Cleaving the Si cleanly is tedious and time consuming – however, trying to clean the Si afterwards because of a poor cleaving job is impossible. (Give yourself a few hours, at least at until you are practiced).

Right before you are ready to use the Si, you need to clean the Si surface. The minimal *typical* procedure we use for cleaning the substrates is to clean with the CO2 snowjet and then place the samples into the UV ozone chamber for 30 minutes. Both these procedures will remove organic contaminants and the snowjet will remove particulate matter as well. Now you are ready to spincoat onto the sample.

Solutions:

Because the thickness of a film is dependent on the polymer concentration in the solution, it is crucial that you make your concentration measurements very carefully. The following is a typical excerpt from my lab notebook. You should have the same information in your notebook:

Need solution of PS (1246k) @ 2% in toluene (Polymer Source # P974-St)

	need (g)	actual (g)
M(PS)	~ 0.2	0.1915
M(Toluene)	M(Tol) = M(PS)*0.98/0.02 = 9.384	9.3844

actual concentration: C = 2.00% Solution # 12090101

As you can see, I noted the M_w of the polymer as well as the batch number from the company from which the polymer is purchased. I use the chart because that way I can focus on my measurements while I am making the solution. First add the polymer to the vial and get roughly the quantity you need. It is the actual value that matters. Calculate

the mass of the solvent required to get the concentration you need, and then add the solvent with a pipette. We add the solvent last because adding solvent can be done more accurately than adding polymer (i.e. drop by drop at the end).

The labelling is important: notice that in my lab notebook I wrote the solution number 12090101. This means that the solution was made on the September 12th in 2001 and it was the first solution I made that day (day/month/year/#). On the label for the solution I wrote the following and you must do the same:

PS(1246k) in Toluene
P974-St
2%
#12090101KDV

The advantage of this is that anyone can look at the vial, see what is in it, who made up the solution, and we can easily go to the right lab notebook to find the relevant information. Please follow this convention.

The solution should stir gently overnight. Make sure that the stirring is gentle enough that no air bubbles get into the solution.

Spincoating:

Best results are obtained with 2000-4000 rpm – the speed used will affect the thickness of the film, so this is the kind of detail that should certainly appear in your notebook. You can also set the acceleration at the start of the spincoating run. This should be on maximum all the time. If you work with a very viscous solution, then decreasing the acceleration (say to 12 o'clock) will improve the uniformity of your samples – just *make sure that you turn acceleration back to maximum after you are finished* and note in your lab notebook that you are using a slow acceleration because it affects the final film thickness.

There are two methods of spincoating, you can spin and then place a *single* drop of solution on the sample (make sure you hit the middle), or you can place a few drops on the sample and then spin. The method used ('spin then drop' or 'drop and spin') changes from solution to solution and you should use whatever gives you the most uniform samples (and note this in your lab-book).

After spinning for about 30 seconds, you can hit the reset button to stop spinning and place your sample into a tray. One caution, on the bottom of your sample you may still have a drop of polymer and solvent – if you place the wet sample into your tray, the solvent will dissolve the top layer of the tray and glue your sample into the tray!!! (Again, I know this because I have done it several times!)