Advance Manufacturing Techniques
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Replication part 2
Lab Session – 9

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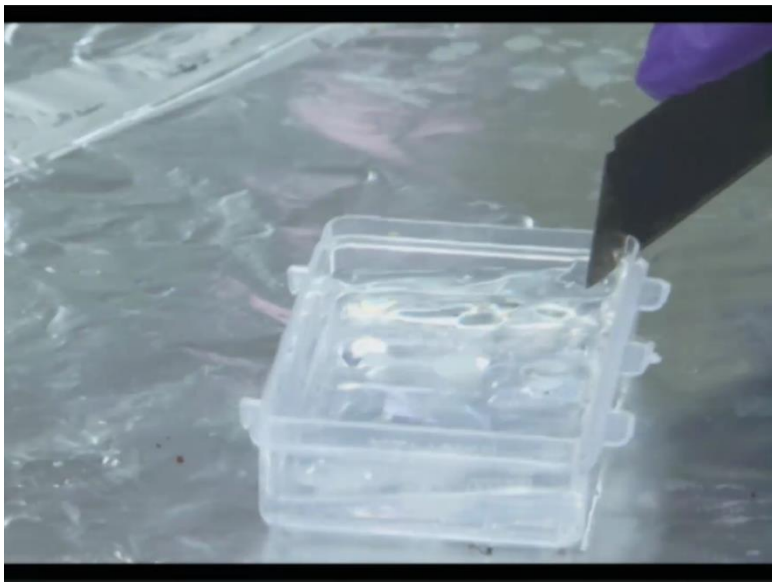

So after this whole heating process is carried out for about 85 to 90 degrees for about close to 45 minutes to 1 hour. You have a box kept here, so this was labeled in this particular leveling plate inside the oven, and you have quite uniform layer of PDMS which is solidified on the top of this these structures here, which is made up of the laser machine PMMA surface.

And so you have now what you are looking for you know actually we are trying to build two different layers of PDMS actually a thinner layer on the top of this mould and both are equally important for the field of micro fluidics, so this would be thin layer PDMS. So subsequently I will show you another step, where you have to do a thick layer. Use the normal paper cutter for the purpose of cleaving the mould box and all the sides. Remember this box actually is a sacrificial mould box, it has to be at the end of the day, it has to be a use and through kind of a platform, so we cleave all the corners of this box. And one thing which is important for me to tell you is that the PDMS is we sticking to any surface acquired firmly, especially when it's get cured etcetera.

And in this case, because we did not treat the surface of the mould box with this molder releasing agent, there is a tendency of the PDMS side to stick to the side walls of the mould box. So one has to be extremely careful about removing this mould box and removing or extracting the molded PDMS along with the polymeric structure which is inside, the PMMS structure laser machine which is inside. So that you can have the sides or the edges of the whole stamp un-cleaved. And this is very it has to be done very carefully to prevent any kind of cleaving action or sticking action of the sides of the mould box with reference to the or with respect to the PDMS inner.

So you can see here the PDMS being extracted very carefully and wherever the additional cutting to be done, and in fact you know one can because that's why the box is designed in a manner which is always oversize than the size of the individual dye, which is going to replicate the device. So if need to be some sides and edges can be cleaved off, in any event there are going to be cleaved off later. Once the extraction of the mould and subsequently the retrieval of the replicator part happens from this particular setup. There is always possibilities that there is some kind of a infiltration of PDMS on the lower side of the PMMA mould.

You remember when we did this fitment of the PMMA mould, although we put a sort of adhesive tape at the bottom double-sided tape on the bottom, still there is some gap which are there. There is possibilities of the PDMS to go on the backside of that mould. And when we rescue there is (( )) membrane developed there also. So one has to be careful about cleaving that so you have to really know very well which side is the pattern side. And then we careful by removing every other thing and all the different sides, like on the other surface for example of the PMMS, where there is no pattern, ok so that surface whatever film is formulated, you should be typically cleave off. Those are all sacrificially used for realizing the most important which is what the film that is formulated on the top of the pattern side of the PDMA. So this is the complete PDMS chunk, which is now cured, you can see that it is quite thick. And the surface on which we wanted this micro fluidic device to be embedded is actually the thinner surface side. And so what we have to do is this is the slow retrieval of these PMMA, laser micro machine PMMA from the PDMS the lower side. One has to be extremely slow and removing it, because otherwise there maybe a (( )), it has a irreversible shield which is developed on the surface. And this sticktion maybe able to cause damage to the device structure.

So this is how now the device looks like, there is a thin film o f the PDMS on the top of the surface. This is the thin device, we will also subsequently show a thicker version later on. You can see, you can see the mould not being separated, so the mould is on one side, and the other side is integrated with the PDMS on top of that featured side or pattern side of the mould, there is a rubberized PDMS, which is left over. So you can now separate the mould from that PDMS in a very careful manner; and the thin layer of PDMS which is now replicated can come or can be retrieved from the top surface of the mould, which is the patterned surface.

So the idea is whatever features are embedded within this dye side, (( )) that you want to remove the PDMS, the replicated PDMS from the surface of the mould. And I just like to illustrate here that in the laser micro machine PMMS surface, there was a feature which was actually like a channel, in craved channel on the top of that PMMA. And we are now replicating that channels, so whatever we will be getting on the top of this PDMS surface will be a ridge. It will not be a channel, but will be a ridge, because now the PDMS has infiltrated into the pattern, and we are separating the rubber and we got rubberized. So we are separating the rubberized PDMS from the pattern, so it will have the same shape, embedded like a ridge on the top of this PDMS.

So you can see this layer of PDMS being retrieved of the mould, which has actually the embedded feature the, I would say the (( )) feature of whatever was like in a embedded inside the PMMA or on the top of the PMMA. So it is exactly the inverse of that embedded channel. So this film has now something coming out of the surface, projected out of the surface like a ridge pattern of which is the replica of the pattern which was there on the PDMS ok. So again, reiterating, PMMA surface which was laser machine earlier had a channel like depth in this particular shape and orientation. And the PDMS surface which has come out has the exact ridge like projected part of the surface coming out in those region.

As you can see here, ok so this ridge is actually being further replicated now in another layer and that is why this process is called micro replication by double inversion, so the first layer is PMMA is the channel, the replication or replicator layer is the ridge. And this ridge is now again being replicated into a channel by using this ridge pattern on the PDMS, after solidifying and heat curing PDMS for sometimes. As a ridge which would be finally eventually trying to take you know or create a replica of a ridge which is again a back to channel.

So, we are actually inverting a pattern, once building a negative pattern on the PDMS intermediate. And then on the next step, we are bringing another inversion of the pattern on the PDMS, so we are essentially replicating a channel onto that surface. So we will now demonstrate another very interesting you know the portion where we will be doing the secondary level replication by the PDMS film which has been extracted earlier with the ridge like pattern on the surface.

You can see the ridge like pattern of the PDMS on the surface in this particular area. This again coming again, this is the channel, it is the embedded that on the laser micro machine surface. This is the replica of the channel, which is like a ridge or projected out feature on the PDMS. On the next layer would be again replicating this particular ridge which would again the channel on the PDMS surface. So the first thing now what we have to do is to sort off heat this PDMS using a hot plate to a temperature of above about close to two hundred degrees Celsius, where it becomes glassy. Because you know it highly porous medium, otherwise it is porous on the surface.

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And we want that to become glassy, so that this mould which is being a use for the next step for the micro replication by double inversion. This mould should be able to again you know we able to get separated very fast from the surface which it itself replicate. So we setup the temperature

all the way to about close to 200 degree to 200 two Celsius, and allow it to heat cure the the PDMS for this amount of time for about close to you know tens of minutes, so that the PDMS achieves a glass liked structure on the top. So now you have not only a ridge like structure on the PDMS, but you are making the ridge like structure with very glassy, so that you can actually we able to replicate the glassy structure onto another layer of PDMS.

So we now in the next step would be using the same decicator for whatever glassy surfaces been obtain will like to replicate that surface that we have to use a mould release agent on that surface. So now we will be actually taking on the PDMS structure, and using the decicator as we showed before in the last experiment when we are doing this replication of the PMMA mould. This is that PDMS with ridge like pattern as you are seeing here, and we have to this is glassy also, because you have heat cured this whole surface.

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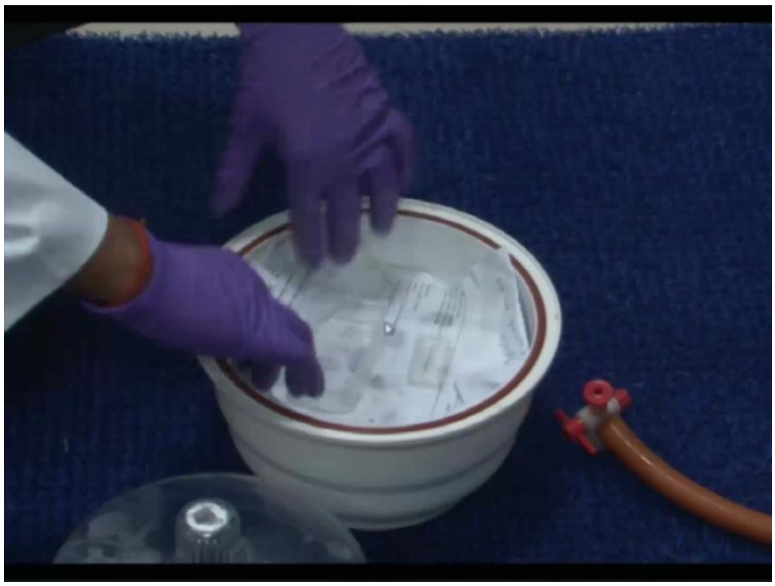

So it is becomes glassy. So now you can actually put the glassy pattern, inside the decicator and heat cured this with sorry the and chemically treat this with HMDS- hexamethyl diasolusin, so that again the glassy surface of the PDMS that has been created can be coated with suitable fluidic, layer.

So the HMDS normally comes in small ampoules and the very good idea for the process to take place mostly the process has to be in sort of you know vacuum, in a sort of laminar flow like

condition. And this vapor has to be somehow bleeder out of the decicator, again into the exact output of the laboratory. So this an ampoules, so you have to very carefully crush the ampoule, and keep this ampoule inside the decicator, and then close the decicator. And turn the decicator on, so that whatever environment is being created here, would remain for sometimes and there would be treatment based on that environment. So you can switch on the vacuum from the pump, we should make the decicator stable and now the idea is that you can make you know this is become under vacuum as you can see. And whatever vapors are being generated by the HMDS now, or going to fill this whole chamber up here. And they are going to sit on that PDMS mould that we have created, ridge like film that we have created. And they are going to actually make the surface the glassy surface of the PDMS ridge, hydrophobic, so that you can use this for subsequent rarely another molding step, followed by this.

In the meantime, you start preparing the the PDMS makes again for the second round of replications, remember now you have to do the ridge which has been formulated in the last step two another channel now, so it is the negative of the negative that you want to formulate.

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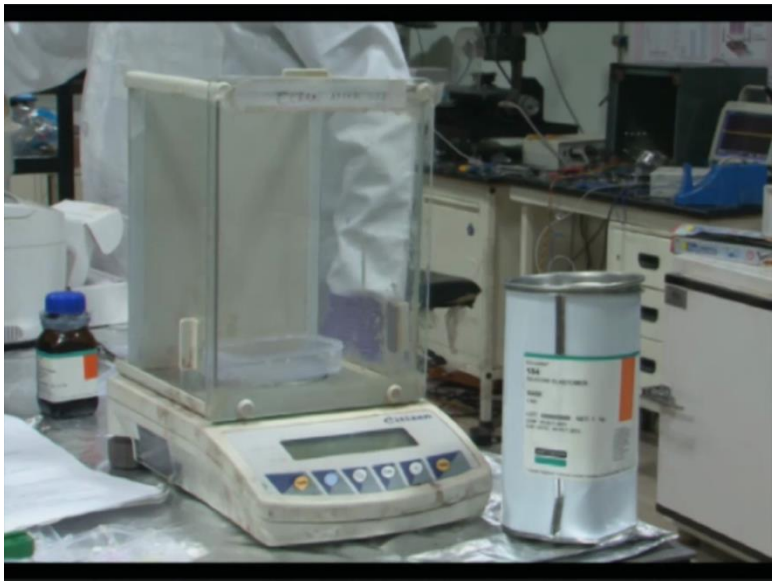

So here you now prepare the PDMS mix as in the next step, so you use that same process of measuring by weight. And you know there is a mould box again a sacrificial mould box being used. And you will be using this the commercial brand available for PDMS by you know mixing

two faces which is the result matrix along with the curing agent. And this would actually be in a ratio of ten is to one, so that by weight that you can have proper functionality by curing time etcetera.

Again the curing time is realized by making by heat treating the PDMS to a higher temperature. And so you can make a curing time shorter and shorter based on what is the amount of heat that is flowing into the system, heat is like a catalyze which would be cross bond the resin matrix and the curing agent, ok premixed resin matrix in the curing agent. So you basically measure and try to zero the weight and you can pour both the components ok. So you have now poured the PDMS ok, now you will pour the curing agent to exactly one ten to the weight. So you clear the system again, and put two point zero seven grams. While keeping the eye on the monitor giving the weight measurement of this particular weighing balance, so you can actually pour exactly about one tenth which is about two gram in the particular case over the twenty point seven grams of resin matrix which has been poured earlier.

A good idea sometimes is also to use a dropper in this particular case, so that you can have exactly the amount which is being used ok. And therefore now you have a premixed sample of resin and PDMS which would now be doing the mixing with in normal stirring process ok using a clean stirrer. So in laboratory is where stirrer are not easily available etcetera also, you can use the clean glass slide, you know to sort of mix both the PDMS as well as the curing agent. Only thing you have to ensure is the glass slide should be clean out of a packet. And you ensure that the proper mix ability is there, so while doing a mixing there is entrapment of all the bubbles ok within the PDMS, because this is the viscous fluid. And it has some inertial delays, so wherever there is a scope there is a tendency of the air bubbles to sort of get blocked, and it doesn't come out that easily which involves you know use the subsequent decication step, which I have already illustrated earlier.

So you have the mould box is treated and read ok the vapor has all been coated on the surface of this particular mould box as well as the PDMS membrane which had the ridge like feature. And now you pour the PDMS on the top of this particular ridge like mould box, this is a premixed resin curing agent matrix, pour it in this manner. It covers the entire pattern, and there is a little bit of PDMS remaining on the top which define the film thickness that would eventually need to

use on the top of this material. So you can see already lot of bubbles being entrapped on the surface of the the PDMS.

And the idea is that you have to have some desiccation or some other steps, so that this bubbles can be somehow gotten get rid of, so that you have a clear matrix, visibly clear matrix of PDMS in this particular zone. Always a sticking layer of PDMS to the bottom of the box, so it is a good idea probably to use whatever is left over and because this was by way to that you have to made in visioning certain height and dimensions of the final of the replica. And therefore, it is a worthwhile really to sort of scrap of all the PDMS which was sticking to the surface of the box, so that you could actually pour this on the top of the mould as it's shown in this particular illustration.

After the de casting step, and I didn't want to show you the intermediate step but the PDMS has gone through the process of thermal curing. So you have a completely rubberized matrix where somewhere down the line here in the bottom of this particular area, you have the mould box. So you have to now do the separation process in the retrieval of the replicated portion from the mould box and that basically do the same operation, you cleave this box on this side, using ordinary paper cutter. And try to first retrieve the PDMS, the rubberized PDMS from the mould box. You can see that you know this sort of it is come out quite easily actually. And you can actually trim of all the edges of this material, this material has to be cut and cleaved in the area which contains essentially the device part. Device part is quite visible, but although because you know now it is a PDMS over PDMS, that reduce the visibility little bit, because of the identical nature of the refractive indexes of both the materials. But you tentatively know where exactly you have place this device, use a pre cleaned glass slide to sort of try to use this axe jig to cut the PDMS tray.

You can see that you know we trying to lay off by creating a hot surface over which we will put this PDMS mould. And then use a glass slide as a guiding jig, for the purpose of cutting of scraping of the excess PDMS. So we can actually now make impression so that we are sort of trimming of the edge, trying to separate out the material which is not useful from the mould material ok which is somewhere inside. And in the similar manner, you can do it on all the other side or all the other edges, but trimming them off. So this is essentially trimming of another edge,

so only that portion which will enclose or which would be containing the device will have to retrieve from the whole, stamp of the PDMS, which can be seen in this particular illustration.

Similar chunk of the material can be removed from the other side. Now you have a piece a central piece, where you have you know the two sort of the replica containing PDMS and the PDMS which has been replicated, the sitting one on the top of other. And then the hard process of sort of retrieving both comes to now remember, there is a glassiness of the surface, which allows the easy retrieval of both the surfaces. As you can see here, there is a retrieval of one surface, so now we want to retrieve both the surfaces and you can see the mass, if you are trying to slowly separate you know the two surfaces together. One being glassized gets very easily separated, and the irreversible seal which is formulated between the replicated and one which is the replica layer, the mould layer, those get you know you have to really remove it carefully. So that such a reversible seal may not be able to reversible seal, may not be reversible seal able to cause a damage of the replicated layer, while retrieval from the replica or the mould layer.

So you can see how carefully so all the edges of trimmed off and how carefully the retrieval process takes place. So you can now see both the layers sitting parallel to each other, and I would just like to now take off, so you can see that how this layer is coming off you know very carefully you have now separated both the layers ok. So now the layer which has been formulated right here, is the replica of the ridge which was on this particular side, which we are done earlier through the earlier processing steps, and it is actually a channel. And this channel can be easily used for micro fluidic applications, because you can actually bond this particular surface containing a channel to a glass piece. And before doing the bonding you can actually with a small syringe needle - laboratory syringe needle brick a small inlet-outlet holes on both the sides, so that there is a easy transparence of fluids, and you can that way connect the fluidic device to the external one.

And then you can typically do this drilling process by very slowly twisting the needle, so that the straight path of the channel can be created till it goes to the other sides. So you have now needles bricking holes on both sides of this device; fantastic thing about this is a rubbery material, so whatever small channel has been created across the thickness of the PDMS can be easily cut this channel , or cut this port in the thickness of the PDMS. Here PDMS is that it is a highly elastic material, so therefore if you put a small piping or a tubing inside this drilled hole, there is always

a tendency of sub seal to happen between the thickness of the PDMS and that pipe or channel the tube. And therefore, you can easily in a very leak proof manner handle fluids into this small structure which has been embedded on the bottom of this PDMS layer and closed subsequently by a glass wafer.

A similar drilling is done on the other side for ensuring that the devices is connected to you know an inflow and outflow port. So you now have actually a device with inlet-outlet ports. We want to irreversibly bond this wafer by exposing it to plasma – oxygen plasma. This is a Harrick plasma measure, which is a very commonly available in laboratory in order to make a RF, you know in a magnetically enhanced laboratory level plasmas. And you have a clean glass slide which you are placing with respect to this PDMS piece here, right here with the replication on the top ok the pattern on the top.

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And you exposing it to oxygen plasma, because you want to introduce the hydro fluidity of this surface, so that there is irreversible bonding between one side of the PDMS, replicated side of the PDMS and the lower glass plate. And these are some procedures associated without the plasma system is operated, you have going in a power on mode and then there is a vacuuming step, which is there. So this plasma chamber is now connected with a vacuum. And basically you can look through this holes here to see the plasma has been obtain, plasma is like a film. You

will be able to see on this particular you know through this viewing windows or this viewing gallery. And the moment the plasma is formulated, you wait for certain amount of exposure time in this case it can be very low amount of exposure time close to about thirty seconds or so. From this formulation of the plasma, and this you can measure using a stuff watch ok, the plasma typically formulated after switching on the magnetic circuit or the magnetic coil, and waiting for some close to few seconds, tens of seconds. So waiting for the few minutes till the plasma actually gets generated.

You will just about start to see the glow coming out of these holes shortly. So you can see the glow come ok, the plasma glow has come now. And you wait for about close to thirty seconds, after this glow is there. And that ensures the process is fully completed. This is how it actually looks like, the tube is all lighting up with the plasma as you can see. So as the plasma process is complete, we can retrieve, we can switch off the plasma and take out this samples, and then quickly overlay the pattern side of the PDMS to the exposed wafer surface. This is very important, because you know you should know what is the patterns of the PDMS, because you are trying to close that size for doing, side for doing micro fluidic deliveries.

So you have a micro fluidic device now, where you can do flow control on the surface of this PDMS. You can put the inlet-outlet ports on both sides of this drilled holes that we had done earlier. And so therefore you can have a very good flow channel, which may be only defined feature size that was there on the laser wafer or laser made PMMA substrate, which was only a few microns. So here we can do some characterization of this micro channel using fluorescent microscope, or in the optical field using the bright field option. So here, you can actually see how this channels would typically look like, this is how the microscope has been focused onto this channel. So you can actually get some good images, and this microscope as scale, so it can do some metrology as well. You can actually get a very good illustration of what are the various wedge of the device, which are obtain over this particular channels. So you can actually measure from between any two places ok on a scale which is definable by the magnification length, the magnification factor on the lengths, that you were using.