Fabrication Techniques for Mems-Based Sensors: Clinical Perspective Prof. Hardik J Pandya Department of Electronic Systems Engineering Indian Institute of Science, Bangalore

Lecture – 51 PDMS Moulding

Welcome. This is the next module in the lab orientation part of the micro fabrication course. If you remember correctly, in our in one of our previous modules, we have looked at general how micro fluidics devices looks like, and how what is a peristaltic pump, what are the different types of pumps used peristaltic and syringe pumps we had discussed. Then like how to flow how to load samples in the pump, how to flow liquids. So, those things we had discussed.

So, that time we had I had shown you in the micro fluidic device channels, certain patterns and how liquid will be flowing through that. But then how are these patterns made? How are these micro channels made? So, micro fabrication processes will be used to make these channels. That is why the use of micro fluidic devices becomes relevant to the scope of this course.

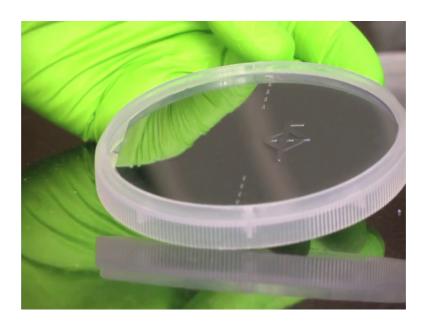
So, today in this module this will be an extended module, today is module we will run you through a very important portion or a part of a micro fluidic channel fabrication; which is PDMS moulding. So, what we will do is, we have as master or a mould, a master mould which we have fabricated with silicon, and on using that master mould, we will be pouring PDMS curing it and making channels out of it.

So, that whole process we will show it to you. What is PDMS? PDMS is poly di methyl syringe. So, it is it is a form of silicon that is very transparent ones it is cured. In it is native star normal room temperature form it is a thick viscous fluid. And once we do subject it to a certain set of heating processes, along with adding something called a curing agent that PDMS viscous liquid solidifies to for to a rubbery elastic, but solid transparent membrane; which is very good for making micro fluidic channels.

So, the channels will be engraved on this PDMS membrane, and that membrane we can stick on to an another substrate that might have a sensor like an electrode, or even stick it on to a glass slide, inside which we can flow the liquids.

So, this is a overall idea. So, today as I told we will be actually doing the silicon PDMS moulding using as silicon master. What is the master? Master is a mould which has a patterns on it, which will form the negative structures on the PDMS mould. So, let us see what is the pattern we are trying to make.

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So, I have with me here a silicon mould, can you see the pattern on it is surface? So, as I told, this is the silicon master we will be using to make the micro fluidic channels. As you can see the channel dimensions are embossed on the silicon wafer. How was this silicon master made? So, we took a silicon normal silicon wafer, p type silicon wafer; which usual silicon wafer dimensions are around 500 microns thickness, then we deposited oxide layer on top.

What after that what we did is, we did photolithography of the pattern on of this pattern. These are the micro fluidic channels, these will now they are coming as elevated structure, now they are coming as elevated structures, these will come as channels on the PDMS, because if negative of this will form on the PDMS. On top of this only we will pour PDMS as we told. So, silicon wafer we will take the silicon wafer is around 500 microns in thickness, then we will have an oxide layer on top. Then we will do photolithography, we will pattern the surface of this wafer using photolithography so that will get this pattern.

Then we will remove the photo resist after developing it. After developing it we will have the pattern formed on the surface of the wafer. Then what we will do? We have to do something called deep reactive ion etching.

So, wherever the pattern is not supposed to be there, we will etch away the silicon material so that only the pattern remains as an embossed structure on top. Using what we use the process we use is what? Deep reactive ion etching I think reactive ion etching you would have covered in the theory, or if not we will be covering.

So, the reactive ion etching is used and then it will etch out the pattern like this on the silicon wafer surface. Once that is done we will remove the photo resist from the rest of the wafer and then yes, we will have the silicon master mould with us. Once this mould is ready with us we are good to go to do PDMS moulding. Today we will be doing PDMS moulding basically of this channel on the PDMS membrane. So, for this we have made this setup for the experiment. So, what all things do we need? So, this is PDMS it will be highly viscous liquid. So, see silicone elastomer that is what it is generally called, this is called see silicone elastomer, that is what it is generally called.

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So, it is basically PDMS, it is effect it is a 1 kg bottle. We will use very less quantity of that around 50 ml. Then this is the curing agent. So, this is another chemical. So, this curing agent is what will make the PDMS become solid after sufficient time.

So, we have to mix PDMS and curing agent in 1 is to 10 ratio. So, one part of curing agent and 10 parts of PDMS. That is the usual mixing ratio. After ones we do that, once we mix it we have to stir it continuously for 10 minutes so that they get mixed properly.

In the stirring process lot of air bubbles will be formed in the mixture. But air bubbles should not be there in our membrane because we need it to be a very clear transparent membrane. Then what we will do? This mixture we will keep inside a desiccator unit and depressurize it so that all the air bubbles will start coming out. Once that process completes that will need around 20, 25 minutes. Once that process completes we will get a very clear liquid of the PDMS clearing agent curing agent mix. Once that mix is ready we will take that mix and pour it on a Petri dish that is containing our silicon master.

Once that is ready we will have a dish containing the PDMS and curing agent mix with the silicon master at the bottom; that dish then we will keep it at around 60 to 70 degree Celsius for 2 to 3 hours inside the oven. Then it will after that if you take it out we will see we will see that the PDMS has become cured, and it has become a solid layer, then whatever dimensions we need we can cut out that PDMS from it.

This is a overall process which we will be showing it to you today. So, what all things do we have? What all equipment do we have? We have our silicon master; which is in a wafer career, we have a 135 mm glass Petri dish, where we will keep our silicon master. We have aces or stainless steel tweezer to handle our wafer. We have a stirrer, this stirrer is used to thoroughly mix the PDMS and the curing agent.

50 ml tube where we can mix the PDMS and the curing agent before we pour it on the on to the Petri dish and we also have a 15 ml tube for increase we need it during the process. And obviously, we have the PDMS and the curing agent with us. So, these are the major things that we need for doing this experiment. At the end of the experiment you will be able to see a PDMS membrane clearly getting formed.

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So, we have taken 50 ml of PDMS here, 50 ml PDMS and 5 ml of curing agent. Now we will mix it in this Petri dish.

So, first let me add the PDMS. So, if you see closely observe you will see that it is a very viscous chemical. It is basically another form of silicon. So, if you can see the Petri dish, you will see that lot of air bubbles are formed in this.

So, we have to have a proper process to remove air bubbles otherwise, this air bubble will form artifacts in the final PDMS layer that will be formed using the silicon master. So, that is why we will be using a desiccator which I explained before. So, I am just waiting for the whole PDMS to get poured, because we have measured 50 ml in the so let us maximum from the tube go to the petri dish.

So, if you just I will just tilt the Petri dish, if you see it you will see lot of air bubbles in it. So, this air bubbles have to be removed at some point. Now we are adding the 5 ml curing agent to this mixture. So, we have added 5 ml curing agent. Now we have to mix it thoroughly as we have discussed. So, we use a glass stirrer, why are we using glass? Glass is a very inert material. So, it will not react with the chemical, that is why most of these equipment like Petri dish and all are made of glass.

This is a very general knowledge, but then many people do not know that that. So, that is why we are discussing that. So, we are using a glass stirrer as you can see. So, with a

glass stirrer we have to thoroughly stir the PDMS and curing agent. So, I am stirring it see, this is how we should stir it, we should stir it for almost 10 minutes. So, we have now thoroughly mixed the PDMS and curing agent for 10 minutes more than 10 minutes actually you can see the mix now it is full of bubbles, we cannot use it directly, actually this is the mix we need to make the mould, but then with these bubbles. This is the same layer that will be formed as a thick solid.

Once we cure it, but then lot of bubbles are there inside which is not desirable. We need to remove all the bubbles. So, we have thoroughly mixed the PDMS and curing agent for 10 minutes, and the mix is now ready. But then, as we can see very clearly that the mix has lot of air bubbles in it. Lot of air bubbles, which is not desirable because this is a mix that we have to pour on our silicon master. And once it becomes solid this air bubbles if they are there no they will they will form part of the layer. Then it will not be transparent and we will not be able to see what is beneath.

The channels will be formed at the bottom of this membrane effectively the membrane that will be formed. So, it is very important that this liquid mix fluid mix should be free of air bubbles. So, the next process is to remove air bubbles from this mixture. How do we do that? We do that using a vacuum desiccator and a vacuum pump.

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We have already seen in prior modules how the vacuum desiccator and the vacuum pump are used. Now we will see the actual practical application of it. What we will do now is we will keep this dish inside the desiccator.

So, I am opening the lid of the dessicator. Now we will keep this dish inside the desiccator, ok. Now we have kept the dish inside the desiccator, we are closing the lid. Now we will connect our vacuum pump to the inlet of the desiccator. And now we will switch on our vacuum pump. So, just now we saw the air bubbles see, air bubbles are accumulating at the top of the you have seen as a close up videos graph; where the air bubbles are accumulating at the top of the dish, all the air bubbles are coming out.

So, as and when the all the air bubbles come out, it will become a clear solution. So, it will take around 25 to 30 minutes to remove all the air bubbles from the mixture. So, we have taken 5 ml of the curing agent in this tube. Now we have to mix this first in another tube. So, as I told we will be adding PDMS and curing agent. In curing agent and PDMS in one is to 10 ratio, that is one part of curing agent and 10 parts of PDMS.

So, because we have added 5 ml of curing agent we have to add 50 ml of PDMS. So now, right now he is adding 50 ml of curing agent. So now, we have desiccated it for almost 15 minutes to remove the air bubbles. What we have done? We have kept the PDMS curing agent thoroughly mixed up it had lot of air bubbles in it, we kept it inside the desiccator and we are trying to suck out air and create vacuum inside the desiccator. In that process what happens? Whatever a bubble is embedded within the liquid it comes to the surface, because it is getting pulled by the environment.

So, all the air bubbles will first come to the surface they form a thick air bubble area; like when you open a what you call fizz drink, a carbonated drink bottle when you open you will see all the bubbles coming to the surface right. Like that these bubbles will come to the surface and again as and as and when we still continue taking air out of the desiccator these bubbles will start escaping from the surface. So, overtime all the bubbles will escape from the liquid from the bulk of the liquid and from the surface of the liquid and eventually in the liquid will become a very, very clear.

Now we will take out the liquid and see how clear it is. So, we have switched off the desiccator. Now we have, we cannot directly take out, because it will create a shock inside and that the Petri dish may vibrate or get thrown away inside, because there is no

air actually inside. So, we are slowly recreating we are depressurizing, we are removing the vacuum inside. So now, air is rushing back into the desiccator.

Once that is done we are removing the desiccator. Now we will see how clear the liquid has become. We are taking it out now see it is very, very clear now. It is a very, very clear liquid you cannot even see whether the liquid is there inside that is that clear. Now this clear liquid we have to pour on top of our mould. So, we have the mould here, we have put it in a Petri dish, now we have to pour our PDMS curing agent mix on top of the mould, there only there only there.

So, as and when we pour more air bubbles are getting formed. So we have to do one more level of desiccation to remove the air bubbles in the second round of pouring the PDMS onto the mould. So, we are pouring the PDMS onto the mould. This process also creates air bubbles. So, we now we have fully poured I cannot tilt this and show it because it will affect the surface profile of the PDMS. So now, we have poured the PDMS on the mould. It is evenly distributed, but there still it is formed few air bubbles. Now we have to do the desiccation process again with the PDMS on the silicon mould, and then remove this air bubbles also.

So, this one I am taking it, putting it again back inside the desiccator, I will put the desiccator back in the view for you. Now we have put the PDMS and the silicon mould. So, we have we have kept the Petri dish with the PDMS curing agent mix after removing air bubbles. We have put it on the mould now the mould PDMS everything is there. We have to again while in the process of pouring the PDMS on the mould some air bubbles got formed.

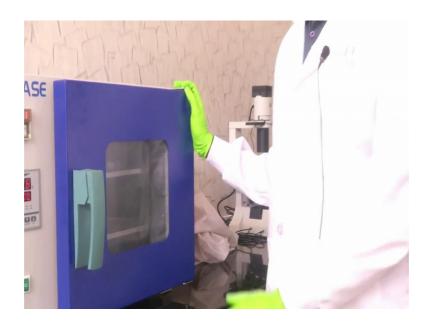
So now we are again closing it. So, we these air bubbles also we have to remove. Now this air bubbles also we have to remove now. So, what we will do? We will again desiccate it now this process is very clear to you, you have to desiccate it now. We have connected the pump, now we are taking out air from it. So, this will remove the air bubbles second time. Like last time, the last time there are millions of air bubbles actually inside. Now it is few 100's. So, this air bubbles will not take much time to clear.

So, maybe around 5 minutes it will take, and this air bubbles will get cleared and again will form we will get a very clear solution on top of the silicon mould. So now, we have performed the second round of desiccation for almost 10 minutes to remove the air

bubbles that were formed again when we poured PDMS on the silicon master. So now, we have desiccated it, most of the air bubbles around the device is gone. And we have got a clear layer of PDMS on top of our silicon master so, this is what we are having. So, we have this much thick layer of PDMS on top of the silicon master.

Because it is so clear now you will not be able to even make out that there is a liquid on top of the master. But actually the PDMS curing agent mix is there on top of it ok. Now the next step is to cure it. What do you mean by curing it? We have to keep this at around 70 degree Celsius for a couple of hours 2 to 3 hours, that the PDMS will solidify it will undergo a chemical change where so, that the physical properties will change, and it will become a solid rubbery material which we can stick on to another surface like a silicon wafer or a glass slide. So, let us keep this inside the oven. So, the operation of the oven also we have shown you before.

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So, we have kept the set the temperature of the oven to 70 degrees Celsius. So, we will keep the glass plate in the oven. So, we have kept it inside, this is for the curing process. After keeping it inside, we are closing the door, now we have to leave this for around 2 hours. After that we will see we will take it out and see how the PDMS layer has been formed.

So, we have made we had we had prepared PDMS and the curing agent, air bubbles where there, we removed the air bubbles say 2 times. First when we prepared the we had

prepared the PDMS with the curing agent, first removed the air bubbles with the mixture, then we had poured PDMS onto the mould silicon mould, and removed bubbles from there also using a desiccator. Then we have kept in the oven for the curing process. So, that it is be inside the oven for the last 2 and half hours now.

Now, by now it would have cured properly. Now we will take out the Petri dish that contains the silicon mould and the PDMS on top and see how it has come. So, this is the Petri dish with a silicon mould and PDMS. So now, if you might not be able to see because the PDMS has formed a very clear surface on top of the mould.

It is almost like glass and you are not able to distinguish which is the glass bottom, Petri dish bottom surface and which is the PDMS surface. Now next step is to cut out the PDMS membrane with the pattern with the required size that we need. And remaining area also has a PDMS which we can cut it out also to see how PDMS has got formed.

So, first we will cut out from a normal area with which is not under the mould pattern. I will show you how PDMS after curing looks like, and then after that we will. So, for cutting out the PDMS from the glass Petri dish main things that we need are a very sharp knife so, that we can make fine features on the PDMS membrane and a metal scale. You need a metal scale because the cutter should not cut out the plastic if it is a plastic scale. A metal scale to take your measurements steel tweezers so that you can peel of the PDMS membrane from the glass slide.

And finally, we need to have aluminium foils to because once the mould is formed the mould gets stuck on to a glass plate. So, that you have you have to use your mould. To re-use your mould you have to cover your Petri dish with a aluminium foil or some ceiling material so that you can reuse it. Otherwise lot of dust will get settled on top of the mould and it becomes non re-usable. So, let us see how we can cut out. Now we will cut of PDMS from an outer area not from top of the mould to show you how after moulding the PDMS membrane looks like.

So, we are trying to peel off the whole membrane. So, we are taking it out from the edges. So, you can see the membrane getting peeled off. So, we are making a boundary around the Petri dish. So, that the whole membrane we can take out, see. So, we are coming covering the edges, the mould is in one part of the; so now, we will try to peel it of. So, you can see you can slowly see now the membrane. See it is very clearly visible it

is a very transparent membrane, and we are peeling it of now. See it is very transparent, but flexible also it is like glass, but flexible glass.

You have to be very careful around the mould, because the mould will gets stuck on to the bottom of the dish. So, till the mould we will take out, and then try to remove it see how we can take out the PDMS around the mould separately. So, we have removed PDMS from around the wafer. So, this is how the PDMS after curing looks like.

So, see this is how the PDMS that was formed around the silicon mould, see how flexible it is. So, preferably you should hold it, either with a gloves or tweezer please never holding with your bare hands. So, this is how the PDMS will look like, it is flexible and fully transparent. See the thickness this is how think it will be, and it will be flexible. Now we have now our core area we have to do, we have to peel of the PDMS on the surface of this silicon mould, you see the pattern here.

So, the pattern is here so, we need to take the PDMS below this pattern. So, that is what we are going to do, let us do the peeling of process. So, now we have to be very very careful while doing this. So, slowly we are taking it out, then somebody should parallely hold the dish, because it is glass it should not slip, and then slowly take it out. So now, we have cut across the PDMS membrane around the mould, and we are now taking it out as you can see. So, the pattern is there I think very clearly your pattern is visible here.

So, see the pattern has to be formed, the pattern that is there on the silicon master has formed on the membrane. Now we will see the membranes other side; to see that the pattern is formed, yeah see against my, yeah you can see the pattern here the pattern has very nicely got formed on the PDMS membrane, and it will be a channel. Here it will be arranged in the wafer, and on the wafer it will be a ridge here, and on the membrane it is a channel. This is how we make PDMS based micro fluidic channels.

The next task is to cut out the device with our required dimensions that we can do with a knife. And then we can cut it out with our whatever dimension we need because now this membrane is circular. Now the membrane is circular, but we need to make a rectangular shape out of this membrane. So, that is the next final task. And then once that is done, we can store this device, now this is also a device, this PDMS membrane is a device which can which we can stick on to an electrode or to a glass slide.

So, we have to cut it out to our required shape and store it in aluminium foil or any other storing material. So now, we are cutting the channels into a rectangular shape, we have already made a cut here to remove this piece and now we will be making another side of the device. The device is here and we are going to cut it out. So, another dimension we have made. Once the membrane is ready and it is successfully removed from the master. It is a relatively easier task to cut it out, but remember always be very careful with your silicon master is a very expensive piece of device that we have made.

Once you have removed peeled of your PDMS membrane, then it is ok, you can just cut it out whatever required dimension. So now, we have made all 4 directions with as metal scale we have cut. Now you will remove the extra material around that square rectangular portion. Yes, that is removed now we have peeled off the extra PDMS membrane and made a rectangular portion for our device. Do not make sure that you do not reach with your tweezer to your device area, keep it far away from it and you can see that device you can see the device also here, in this region against white background no against black background.

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No pure white so there, yeah. So, anyway you might have seen the device, yeah you can see the device here now yes. So, that is the device. So, make sure that you do not contaminate with air. So, we have to quickly packet of in aluminium foil and store it for use. So, we will put in a aluminium foil and then we will store for use. So, people; so students I think now you have got a very good idea of how using a silicon master micro fabricated silicon master, you can make a good nice thick PDMS membrane like this with your micro channel in it.

So, this is a very important process in micro fluidics fabrication, and it is very important that you know each and every single step and the importance and purpose of each of the step each of these steps in the whole process. So, once the membrane is ready as I have told, you can stick it onto a glass slide or a silicon wafer that my may or may not have sensors, they may have electrodes or they might be pure optical analysis based devices.

So, how do we bond PDMS membrane to glass? So, that is done through method called oxygen plasma bonding. So, we have fabricated the PDMS mould as you have seen. So, I you have understood the whole process as I just explained that how important each process is. So, once the membrane or the channels are ready in the PDMS, we can bond or stick is the colloquial work if you are working in a technical field. So, use you should use the technical terms. So, that is bonding you need to bond your PDMS membrane

with the substrate; that the substrate might have sensors which may be electrodes heaters or any other metal or material that may be deposited on your substrate.

Or it might be a very clean glass slide also and your device might work on optical measurement basis alone. So, you can bond your PDMS with glass or with say silicon wafer. So, here we have a glass slide with gold electrodes patterned on it using photolithography. Are you able to see clearly?

So, this is a glass slide with so, this is a glass slide where we have patterned gold electrodes using photolithography. So, what we will do? We have to first clean the glass slide properly. So, that there are no contaminants, then we have to coat it with gold ok. So, that you can use methods like sputtering which you have learnt in the theory, you sputter a fixed the thickness of gold on the glass slide, then you pattern it after doing PR coating, and then exposure using a mask aligner of about a general photolithography machine.

And then you form your pattern, then you will expose it, then once your pattern is formed, you will etch of the remaining gold using a gold etcher like KI. So, then finally, you will have your gold electrode. On this gold electrode, you can bond your micro fluidic channels. So, make sure that you do not touch the device. So, like this you can bond your micro fluidic channels with your electrodes aligning your channel with the electrodes. Ones that bonding is done your device is ready, especially a micro fluidic device will be ready.

How do you bond it? Bonding is done through a method called oxygen plasma. So, we are not going to the details of how that is done, but this is how bonding is done. Similarly, bonding can also be done to silicon wafer. So, next is we have another glass slide, this is a heater use that is patterned using nickel.

So, similar methods you first coat nickel. So, like deposit nickel and then you pattern it using photolithography and then again you can use micro fluidic channels on top of this nickle electrodes. So, this is not a electrode this is a heater, it is still an electrode, but it is the construction is different. Its purpose is to just heat to a particular temperature so that if you have biological experiments, you can maintain your samples at a particular temperature.

So, hope this you got an overall wholesome idea of how a micro fluidic device will be fabricated, how micro fluidic channels are made, how electrodes are patterned on glass or silicon wafers, then how do you bond it. So, hope you have got a good idea of the whole process. Like the way you have learned in theory. You have seen it in practice being fabricated in our lab series. Hope this was useful to you.

Thank you.