

Design Practice - 2
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Lecture - 15
Introduction to Device Fabrications

Hello and welcome to this Design Practice 2 module 15. I would like to today go into some more details about sensing or how sensors are designed or they are carried out and the first important significant thing that I would like to mention are about the methods of immobilization. I think I have in great details explained to you how a sensor surface is modified by putting a recognition element or a layer which would be associating itself with the analyte of interest that you are diagnosing or sensing.

And therefore a very important premise here is how do you immobilize the recognition element on the sensor surface.

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Methods of Immobilization

- Immobilization is needed between the recognition element and the transducer. The protocol is really complex in case of biological entities.
- 1. Adsorption on to a surface.
- 2. Microencapsulation: Trapping between membranes on the surface of detector.
- 3. Entrapment: Trapping with a gel, paste or polymer onto an electrode.
- 4. Covalent attachment: Covalent chemical bonds formulated.
- 5. Cross-linking: A bi-functional agent is used to bond chemically the transducer and the recognition element.

And particularly when we are talking about a transducer surface which is able to monitor chemical, biochemical, biological, or gaseous entities obviously the protocol becomes very complex. And some ways and means are established for you know this immobilization of the recognition element. One of them is the absorption to a surface. Then we have microencapsulation which actually is trapping between members on the surface of the detector.

So you have membranes. So you have two membranes. One membrane on the transducer surface and the other after the membrane has been sprinkled with the recognition element so it kind of entraps and these membranes hook themselves up to each other, adhere to each other or to the surface, transduction surface. So this is called microencapsulation. Then there is an entrapment process through which immobilization can be carried out.

In the entrapment process you can typically use gel which is you know a semi paste like semi you know semi fluidic kind of a state which would be able to give you know diffusion or which will be able to assist the diffusion process of the analyte of interest and the gel typically is a network. So you have a series of pores or sieves. It is highly porous and it also able to do some filtration as the analyte migrates towards the transduction surface or transducer surface.

You can also entrap with paste or polymers on to an electrode, okay. So that is how you can do entrapment. You can do the covalent attachment of the particular recognition element for example you can have chemical bonds formulated between the recognition element and the transducer surface which will give you some essence of how to immobilize. You can do cross linking.

Cross linking means that you have another molecule which will put in place by means of a covalent chemistry okay or you know because it is a bifunctional kind of a bi-linker kind of a molecule. So on one side it will link to the transducer. On another side it will have enough activity to link to the recognition element. So it kind of holds as a linker molecule, the recognition element close to the transducer surface.

And so therefore there are variety of these immobilization methods which one would use for the purpose of you know designing the uh how to outlay the designing the process of outlaying the recognition element on to the transducer. Then there are several other aspects of such sensors which are of engineering consequence.

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Performance Factors

- Selectivity: Ability to distinguish between different substrates.
- Sensitivity range: Usually submillimolar range but in some cases can go down to femtomolar range.
- Accuracy: Better than $\pm 5\%$.
- Nature of solution: The sensor needs to be designed to conditions such as pH, Temperature and Ionic strength.
- Response Time: Long (30s or more) for biosensors.
- Recovery Time: Time elapsed before the sensor is ready to analyze next sample. (not more than a few minutes.)
- Working Lifetime: Stability of the selective material. Short for Biological materials.

One of them is about performance factors. How a sensor performs with respect to how miniscule a sample it can probably take or are there any cross reactivities which may happen, which may create a false signal. So the performance factors if you look at are number 1 is selectivity which is the ability to distinguish between different substrates which are there in the analyte of interest. So substrate is basically an analyte or a type of an analyte or a type of molecule which we are detecting. So there are multiple such molecules which are there.

We also have something called the sensitivity range which is usually the range you know which at which detection can still happen uninterruptedly so therefore we are talking about the least amount or the least concentration of the material of the analyte in the particular solution. So usually submillimolar range is associated but you know it can go all the way up to femtomolar range as well when we talk about such sensitivity range.

There can be a performance factor based on accuracy, how accurate the sensor can be to pick up you know analytes of trace concentrations. The accuracy better than plus minus 5% is always considered to be you know reasonable accuracy for a sensor to qualify. The nature of solution is a very critical aspect when we design sensors. You know sensors need to be designed for the chemical reactivity, the condition such as pH for example the amount of aberration that will happen.

So the sensor needs to take care of that issue so that the transducer does not dissolve away into the solution. It also takes care of issues like what could be the temperature range at which the performance or the functionality does not change of the transducer or for example what is going to be the ionic strength which is going to measure.

Performance factors will also include a very important aspect which is about the response time typically for gas sensing applications or biosensors, the smaller the better but you know the for the power sensing particularly this level is probably slightly high. With the modern technology you can say about 30 seconds or more you know of sensing time is considered to be quite reasonable.

There is also a recovery aspect that once these sensor surface has sensed something whether it can come back to the baseline signal so that it can start sensing another analyte or another solution or another sample and for that you need to find out how much time is elapsed before and what are the protocols that are to be carried out if any for a surface to again recondition itself which again measurements can start taking place. So time elapsed before the sensor is ready to analyze the next sample. That should not be typically more than a few minutes.

pH sensing in this particular matter is you know a pretty commonly used you know electrochemical process where we dip the electrodes in water and try to make the pH come to 7 again before the next sensing round can happen and so it has to be allowed sufficient time for whatever hydrogen ions have crept inside the glass bulb, the chalcogenide based glass bulb so that it can diffuse back and it can neutralize to the 7 pH okay.

So some time needs because of that mechanically driven diffusion process to happen completely before the next you know before the sensor is ready for the next run. So then we also talk about working lifetime of a sensor so stability of the for example the selective material is it for example in particularly biomaterials like enzymes or antibodies which are used by some sensors. The shelf life maybe as low as three months or two months.

So before that it is better to be used and after that one should discard because otherwise it will start giving false signals. So therefore the working lifetime also forms a very critical function when we talk about such performance factors related to the sensors.

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Reasons for miniaturization

- In general, the use of micro and nano-scale detection technologies is justified by,
 - (i) reducing the sensor element to the scale of the target species and hence providing a higher sensitivity à single entity/molecule
 - (ii) reduced reagent volumes and associated costs,
 - (iii) reduced time to result due to small volumes resulting in higher effective concentrations.
 - (iv) amenability of portability and miniaturization of the entire system
 - (v) point-of-care diagnostic,
 - (vi) Multi-agent detection capability
 - (vii) Potential for use *in vitro* as well as *in vivo*

Let us now also look at one of the reasons for why we must focus on making these sensors small and miniaturized. We know that the rapidity and the sensitivity both these factors are really dependent on the level of the miniaturization okay that must occur. At the same time the robustness factor may go down because of the small nature physical nature of the sensors when we talk about miniaturization.

And generally there are many reasons for doing miniaturization when we talk about sensing in the chemicals or biochemical processes and one of them is reducing the sensor element to the scale of the target species. So we must understand something that when we are detecting a small cell let us say for example a biological cell which is about 20 microns or a bacterial cell which is about less than 10 microns.

And we are doing it with a surface which is probably a few hundred millimeters then obviously there are issues related to how this surface would interact with such cell and there are going to be its own inertial delays because the surface is extensive in terms of the sizes as compared to the

target of interest as opposed to if you want to develop a surface which would be exactly similar to the dimension of the particular species.

The inertial response may not be that big of an issue in the signal that the sensor provides you okay. So therefore you can provide a higher sensitivity and that is out rightly the case when we talk about miniaturization. Obviously, when we talk about chemical biochemical processes reagents are very expensive, diagnostics particularly clinical diagnostics becomes very expensive because of the size of the reagent.

And if we are talking about miniaturizing the sensing element it automatically means that the reagent volumes also get miniaturized and so the associated costs of doing diagnosis by doing you know designing sensors would also come down because of such reduced region volumes. There is also reduced time to result due to small volumes because you can have effectively higher concentrations, very small volumes, small changes being sensed by a sensitive instrumentation setup.

And so any small signal which is represented statistically of the you know sensing modality would indicate that there is analyte present or absent. Okay, so this is a big you know advantage of miniaturization that you can actually work at those scales in much reduced time. There is also amenability of portability and miniaturization of the entire system, obviously smaller size the better it is to carry off.

There are concepts where sensors are taken to the bedside or even to the fields for doing detection be it agricultural sensing or be it patient based medical sensing or even sensing of farm animals or farm products. If you can make the sensor small it is always going to give you more you know portability, more usefulness in terms of taking it to the actual spot and doing measurements quickly and rapidly.

This can be used more as a screening data and based on it you can see whether you can take counter measures then and there rather than waiting sensing data to come from a sophisticated lab. So this is also known as point of care diagnosis, okay. So it is a very big area of work and

right now there is billions of dollars of research spent in designing and developing point of care medical biomedical sensors.

There is also you know a scope of multi-agent detection when we talk about miniaturizations, smaller the size the better it is you can have in a smaller space or volume. Many sensing elements which can sense each of these species which are there in an analyte. So if you wanted to do multi-analyte scanning and sensing the best idea is to again start miniaturizing it and then obviously it has potential for use in vitro as well as in vivo capacity and so these are some of the reasons why we must miniaturize sensors.

When we talk about sizing them down, when we talk about small you know overall volume within which the sensors would be occupied, a major issue comes up and which indicates how do you handle such fabrication or what are the kind of agents or what are the kind of techniques which are available through which you can carry out those fabrications. So I am going to now go across certain small modalities where we can talk about little about micromachining or little about photo driven process through which machining can be carried out.

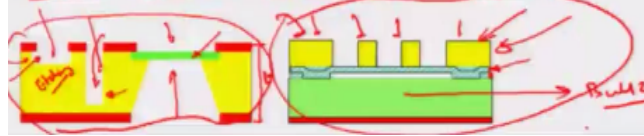
And you can see that how small sizes can be embedded or MEMS can be made with respect to all these processes and typically once you have an idea of how MEMS can be done and MEMS designing, development sensorial design through MEMS route etc. becomes easier and for a designer uh who is into the area of sensors design one must know these basic modalities when we talk about sensor technologies. So let us talk a little bit about device fabrication.

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Introduction to Device fabrication

NEMS/ MEMS silicon fabrication

- Formation of structures that could be used to form sensors and actuators.
- Processing of electrical or non electrical signals.
- Conventional and new semiconductor manufacturing techniques are used.
- Etching, Deposition, Photolithography, Oxidation, Epitaxy etc.
- Deep RIE, Thick plating etc
- Bulk and surface micromachining



MEMS and NEMS processes typically emerged from silicon and associated silicon processes because microelectronic industries had silicon as the base material. The carbon electronics which has grown probably much later did not quite come into picture when microelectronic processing was developed ab initio and therefore MEMS has all sort of focused on to silicon based structures but later towards the 80s changed gears and went into the polymeric structure.

So I am going to from my perspective just give you some idea on fabrication on the silicon and some idea of fabrication of the polymer structures. So MEMS and NEMS are typically fabricated by formation of structures that could be used to form sensors and actuators at that scale, micro and nano scales. Also when we talk about MEMS and NEMS we mean that it is about the processing of electrical or nonelectrical signals.

Nonelectrical for example mass based or in thermal means you know which is not driven to any electron flow as such. So then when we again talk about MEMS NEMS silicon fabrication we generally use conventional and new semiconductor manufacturing techniques some of them maybe etching, deposition, photolithography, oxidation, epitaxy and I am going to do these, some of these techniques with you guys.

So you understand about how these techniques are being done and then there are some which are actually MEMS specific techniques for example this deep reactive ion etching can be a MEMS

specific technique which is used for developing high aspect ratio structures and silicon surface or thick plating which is related to again electroplating of metals particularly which can be several micros in thickness and it can be used for purpose of fabricating MEMS structures.

So when we talk about developing a miniaturized super structure for MEMS we mean by developing this structures which can be either bulk micromachined or surface micromachine and you know there is some fundamental understanding to what is a bulk micromachining activity or what is a surface micromachining activity. For example if you look at the schematic here to the left, we see that this is a silicon wafer okay we just thickness of the silicon wafer this t right here is the thickness of the wafer.

And we are seeing material being taken off subtractively from different regions of this particular thickness of the silicon wafer. So any such process which involves subtractive machining where material is taken away at the micron scale of the microscopic lens scale is better known as bulk micromachining. Similarly, if on the other hand if we develop features and structures on the surface rather than subtractively taking it by adding the material.

For example if we look at this particular cartoon here on the right side, we are talking about films which are being developed on the surface through multiple techniques like deposition etc. This for example is a P++ film okay. And then there are certain techniques which are again further used for bringing out structures and features on the surface by adding. The bulk here is not getting sacrificed or the bulk is not getting machined.

So these are the surface micromachining. So all additive processes are categorized as surface micromachining. All subtractive processes are categorized by bulk micromachining. If I looked at such structures and how they are formulated, this structure right here can be formulated through etching. There is a process called etching okay. Etching can be again a high aspect ratio etching which is done using usually gas plasmas.

This structure here is a high aspect ratio etching. This is a low aspect ratio etching but this can be done on a wet chemical stage with wet chemistry processes. This right here the green region is a

P++ silicon which is done by doping of the silicon structures. P++ meaning thereby that you have high amount of doping concentration of the group 3 material which will pump in more number of holes and you know this structure right here is basically the structure which is again etched out from the silicon okay.

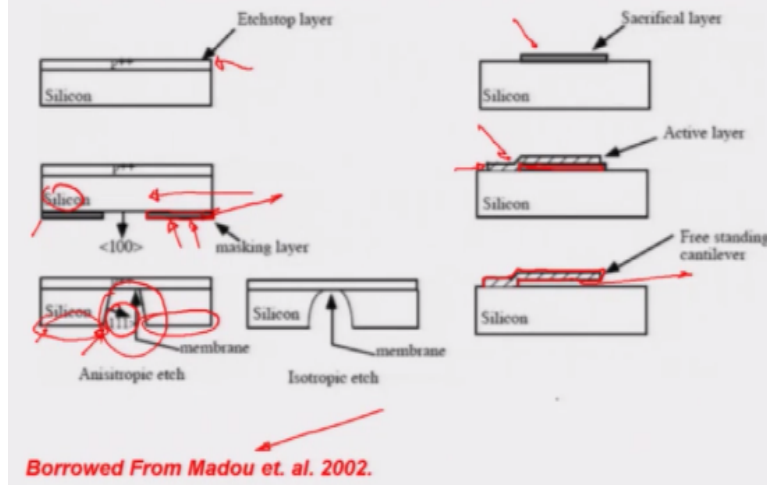
And this meets the green surface right about here and proceeds no further because whatever etching is being carried out stops wherever there is this P++ layer okay. So this is again a process which is called a selectivity driven process so that you can obtain a very thin film, a small film on the surface of this particular silicon wafer. Similarly, quite interesting in this particular case, probably there was some material which was here in this gap which has been later on sacrificially removed.

The material was earlier patterned on the surface and there was a deposition of a thin film which is this hatched area right here which took place and later on this material was removed through chemical etching so that you have this gap created which is like a small embedded chamber which can now be used to carry fluids etc. Okay so this again you know this deposited material is a sort of a polycrystalline silicon material which is higher in strength, mechanical strength aspect so the idea is when this layer is removed here it should not bend or warp okay.

The structure should be stable and this is through again surface micromachining that you can build up such structures. So that is what devices are typically fabricated by. And now we will start looking into some of these processes in some little details.

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Introduction to Micro-fabrication



Let us talk about this particular process here which is borrowed from a micro-fabrication, a very famous micro-fabrication text book. It talks about again using a Etchstop layer and a certain you know masked, certain masking process which would actually lead to the removal of this mask. The mask you know is the literal meaning of the English word mask meaning it kind of shields okay.

So wherever this masking layer is present and I will talk a great more details about what really is the masking layer and how you will be able to remove the layer or make the layer come up in certain selected regions. So wherever the masking layer is present, whatever chemical we are going to etch or use to etch the particular material silicon here which is the white colored material here okay it does not touch the silicon if there is a masking layer.

So let us say in this region the chemical will not be able to get in touch with the silicon which is inside and wherever there is an exposure like in this region there is no masking material so it will start the etching process because the etchant has already contacted the particular surface through which there can be chemical removal or subtractive machining of the particular area. So you can carry out etching in many ways. You can carry out isotropic and anisotropic etching.

This is an example where an isotropic etching is carried and these etching happens because of a redox nature of the chemistry which is involved and ultimately it merges as an array of planes

okay which are corresponding to the 1 1 1 direction. Those are the slowest electron releasing planes because of which you know you can have this kind of a shape but whatever it is without getting into those details of chemical etching I can say that because the masking layer was present and later on you pull out the masking layer you see there is no masking layer here.

So you dissolve this layer in a suitable solvent. But till and until it was present the chemical did not affect the regions where the masking layer was shielding okay. So this is a very important essence. So you are covering something with a layer which is otherwise sacrificial layer and you are using a window created in that sacrificial layer to do selective etching. So supposing today if you were to etch on a very small region, let us say a 100 micron by 100 micron a pit is to be created.

So you can actually pattern and open up vias in the masking layer in a manner where exposure would be there to the parent material and the etching would sit on that and start etching okay. So it is very interesting way of carrying out machining at a very small scale all of which can be used into what we know as MEMS okay or what we know as microstructures which can then go into sensing and diagnostics. So this again is a structure what I described to you before.

There is a layer of material which is otherwise mechanically robust but there is a silicon, polysilicon layer deposited on the top of this sacrificial layer which you are removing and so out props this cantilever like orientation of this particular material. So this is how you do free standing structures okay through micro-fabrication technique.

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BioChip/ BioMEMS materials

- Silicon and microelectronic materials
- Glass, Quartz
- Polymers
 - Poly (dimethylsiloxane) (PDMS)
 - Poly (methyl methacrylate) (PMMA)
 - Teflon, etc.
- Biological Entities
 - Cells, Proteins, DNA
 - Frontier of BioMEMS !

So when we talk about the materials which are mostly used for micro-fabrication, mostly the materials which come in handy are silicon and other microelectronic materials. The chips or the microchips which are being formulated would have the necessary modalities of being able to be transparent on the top so you can have covers which are made using glass or quartz which will not absorb characteristic wavelengths okay.

And gives a free, gives a leeway for the detection process to happen of what is within the chip as opposed to its own component which it might otherwise give in the signal if it is a nontransparent or a slightly translucent kind of material. Obviously, people have realized that when we talk about clinical diagnostics and biofluid samples they are not very well to do with inorganic material like silicon because most of it would not behave in a proper manner as they do within the human body.

And so therefore polymers are another class of material which have been found to rhyme and rhythm with that aspect and therefore people have actually developed a series of polymers which are fabricable at the small scale or the microscale, some examples could be PDMS, poly dimethylsiloxane; PMMA, poly methyl methacrylate. These are the polymers which are used for MEMS fabrication okay, fabrication at the micron scale using resolutions.

And tools with resolutions which are high, particularly tools which should provide high resolution would be able to act on it and quickly produce an array of structures within the surfaces. I am going to get into the precise details a little bit later. Teflon for example is another such material and then there are also materials which are related to the entities themselves.

For example you can today develop devices with DNA printing or you know where sequences of molecules are heaped together or stacked together with processes like micro contact printing or dip pen lithography. So you can actually be able to print molecules and do some you know sensing diagnostics of other molecules based on the chemistry of the molecules that you have printed.

So you can make assays okay or assay technology in a very rapid manner to screen off a bunch of different molecules which may be desirable or undesirable in a certain you know fluidic sample. So I think I am close on this particular module now because I have talked enough about the basics of fabrication. In the next week's module we will start with some fundamental processes. We will also do some aspects of actuators and how to design some of the sensors and actuators. As of now thank you. Thank you very much for being with me on this.