Design Practice - 2
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Lecture - 25
Sensors and Actuators Design

Hello and welcome to this design practice 2, module 25 this module is on sensors and actuators some real case studies and this is reflecting some research which has been carried out in our laboratory related to two different topics one is Caselli where we have been able to use nano structures and we have selectedly nano structures in a manner so that they can be selectively to a certain gas and the response the change which is there on absorbents of this gas on the surface of the nano structure is merely a resistive change and this resistance would vary as a function of the concentration etc.

So, in the second module we are showing a slightly different aspect of calorimetric sensing where we are talking about paper flows or paper microfluidic flows through which there is the yes/no answer given on the presence or absence of an analyte in a particular sample through a change in color.

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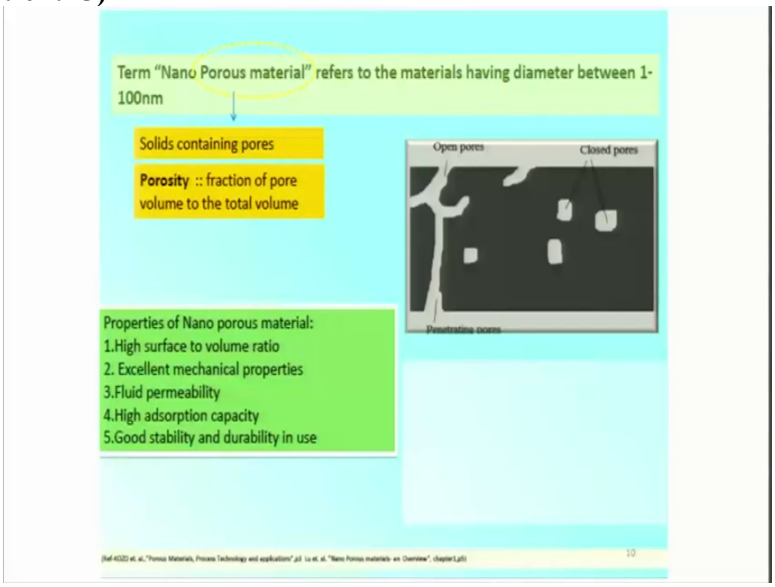

So, let us look at these modules so in module 1 we are talking about nano porous material and what a nano porous material really means that it refers to materials having diameters between you know pores with diameters between 1 and 100 nanometers. So, otherwise the other portions are solid as can be seen in the black region here and these small closed you know structures here

are closed pores whereas this right here is an open pore and as the name defines the open pore basically is a connection of such small pores across the bulk of the volume.

So, that there is one continuity of a bigger pore which is there and individual pores are the small nano pores which are there not particular volume. So, you know depending on the porosity level the number of pores per unit volume can be calculated by looking at the fraction of the pore volume to the total volume. So, if there is certain total volume of material and you know volume of pores which are there in the material porosity is basically a ratio between the two.

And you know we have the properties there are some very distinct properties of the nano porous materials one is of course the high surface to volume ratio an excellent mechanical properties you know fluid permeability. So, whenever we talk about gases and gas sensing question of gas like diffusivity comes into the material so a highly nano porous material with open structures or closed structures of pores would actually lead to permeablizing different gases through these.

And so the proper desorption absorption kinetics can happen which makes the sensor rather very quick. There is high absorption capacity and good stability and durability in use of these nano materials.

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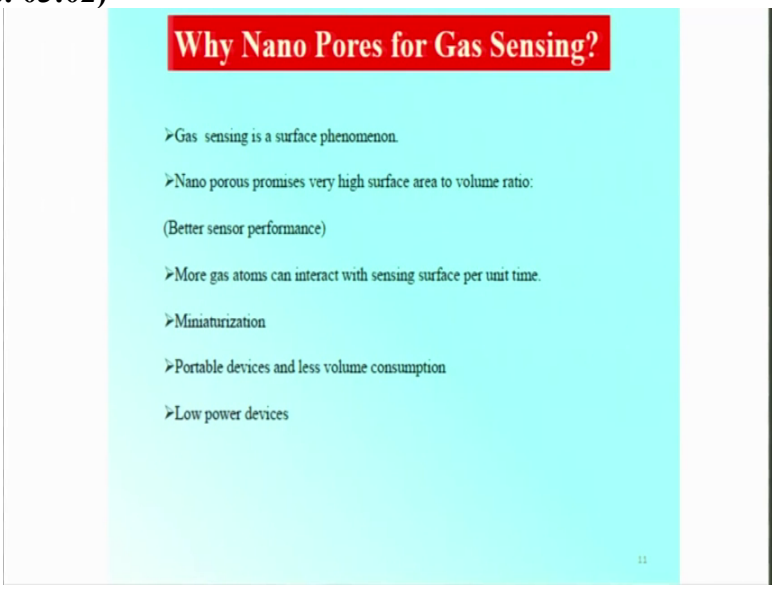

So, this is how the nano porous materials are particularly you would like to also delve into why nanopores for gas sensing and if we can look at you know gas sensing in totality it is a surface phenomena and because of the porosity and the high surface to volume ratio the quick diffusivity

of gases would enable it to adsorb dissolve quickly across the whole bulk of the structure because of the highly porous structure.

And so obviously the answer to the question of what better or how better the sensor performance would be would lie on what is the level of porosity of such materials. So, typically we are allowing for a case where more gas atoms can interact with the sensing surface per unit time and these are some things which can be miniaturized to very rapidly because we are talking about very small you know nano level porous materials on films which are probably several 100 to 200 nanometers thick.

So, we can really make a very small sensing element do a wonderful job in terms of change in resistivity which could give you an idea of you know how much gas is present in the atmosphere bringing a miniaturized in nature and very small volumes you can couple them to portable devices and it would lead to less volume consumption of the analyte which is in air in this case the analyte probably is a fixed gas like hydrogen for example we just present otherwise in not so much abundance.

And you know because of the small size their relative exposure to the smaller nature or smaller presence of hydrogen would be more in comparison to what would have been in case of a bulk film a bigger a larger film and so quick changes and resistance could be; because of the size comparison between the element and the smaller trace of the gas which is in question. And so because of these small volumes and small sizes obviously these are low-power devices the requirements are very small.

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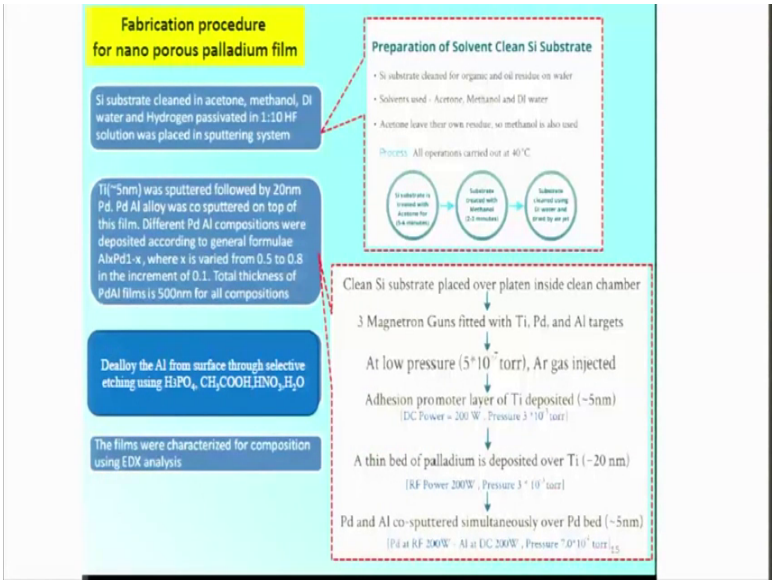

And we can see what are the strategies? Which are being utilized for doing fabrication? So, in the first year strategy we have developed a highly nano porous palladium film and this paper was also published some time back in censor letters where we showed a very substantially high repeatability as well as very low sensing and you know baselining time for the particular sensing element.

So, the way we fabricate is based on a step by step procedure where we are cleaning a substrate primarily made of silicon we clean with organic and oil this you know or clean off the organic and the oiled residue which is deposited on the substrate and we use it or clean it through solvents which are acetone, methanol and di mixtures and of course acetone alone would leave their own residue.

So, therefore a combination of methanol and di is used to clean the solvent of the surface and it is completely dried up so that there is no organics and then what we do is we actually use a process of deposition called sputtering which I have think I have discussed in some of the module earlier and so you know before the sputtering we try to also passivate the surface okay by hydrogen passivation techniques where we dip the surface in a 1 is to 10 HF solution ok.

And then we place the passivated surface on so there are lot of hydroxyl groups now on the surface okay of the silicon and we place this in a sputtering chamber. So, in this particular module what we are doing is using titanium as addition materials so once the passivated surface with let us say which groups from the surface are taken to a sputtering chamber. They are first

coated with a material which would act as an addition promoter in this particular case it is titanium.

There is a 5 nanometer titanium which is deposited through sputter coating process I think I have explained about it. So, sputter coating is some sort of a vaporization process where there is a metal target and there is a plasma which is generated. So, the plasma knocks off some of the metals and carries the metals towards the bias that the plasma system has and typically the substrates are on the ground plate where this bias voltage would drive then the plasma which is there along with the material which is there to the surface.

And this material is highly unreactive because it may be an iron argon plasma or a nitrogen plasma where there is not no possibility and the material that we are using is palladium or titanium where there is no possibility of generating a you know third species which has reacted or there are chemical bonds. So, it is taken back into the system and deposited 5 nano meter layer is followed by again another layer of 20 nano meters.

And this is pure palladium which is actually the sensing element we all know that palladium resistively changes because of the formation of palladium hydride at the grain boundaries and also a change in the state change from the Alpha to the beta state of the Palladium. And essentially after this 25 nanometers of film so with the hydrogen passivation the next step is a titanium layer 5 nanometers layer okay.

And then we are following this with another stack of a slightly thicker material 20 nanometer palladium layers. So, this is titanium this is you know palladium coated on the top of the titanium and then finally we deposit in alloy and these alloy is a palladium aluminum alloy which is co sputtered from two targets with one where we are using the magnetron gun and other which you know is with a DC bias.

So, basically both the palladium and the aluminum are knocked off together with the common plasma that is being created and this palladium aluminum composition is deposited according to the general formula al X P D 1 minus X where X is varied between 0.5 to 0.8 therefore there are cases where exactly aluminum is 50% and palladium is 50% there are cases again where alumina is higher 80% and palladium is 20% and you know we deposit this in an increment of about 0.1 or 10% increment and a total thickness of the palladium films that are being deposited in this manner is about 500 nanometers.

And why we are depositing it that because we want to make a chemistry through which we can selectively di-alloy the aluminum part and wherever the aluminum is deposited with the Palladium in over the palladium bed the aluminum comes off and in this place there are air pores which are generated because obviously air enters when aluminum is etched off. And so we are di allowing the aluminum from the surface through selective etching using a mixture of phosphoric acid, acetic acid, nitric acid and water.

And what happens is that there is a relatively high porosity level which is established by pulling out the aluminum in this manner from the surface okay.

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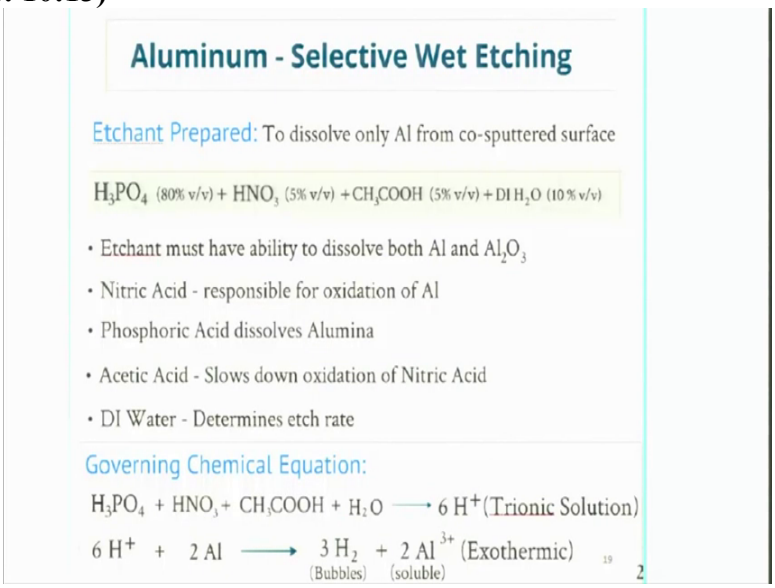

And this constitution of the particular etchant solution to only dissolve the aluminum is 80 % by volume of phosphoric acid 5% nitric acid 5% again by volume of acetic acid and about 10% of DI water so this makes the this makes it 100% ensure that we are removing on the aluminum and an aluminum oxide of course. So, there is a reaction between h3po4 and aluminum, aluminum oxide where aluminum phosphate is formulated okay.

And you know this aluminum phosphate as you can see here is solubilized so it comes back into the solution. So, wherever there is a presence of aluminum or aluminium oxide there is a formulation of the +3 state because of electron transfer chemistry which is generated through the etchant okay. So, etchant must have a ability to dissolve both and so nitric acid is responsible for oxidation of aluminum.

There is some oxide which is already present as I told you al2O3 because the passivation of the surface. But there is some oxide which is also you know being made through reaction with nitric acid and phosphoric acid alumina kind of reacts with each other acetic acid slows down the oxidation of the nitric acid. So, if this step is slowed down and not much aluminum oxide is formed phosphoric acid will actually not be able to be solved so much alumina.

So, we can have a good control by varying these percentage volumes of the various blocking agents as well as the enabling agents so that at a certain rate etching can happen. And we want a slow oxidation process to be enabled so that there is porosity distribution within the whole volume of the co-alloy material which has been deposited.

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So, we have now investigated the structures through scanning electron micrography and you can find out this is pre etched structure at magnification 50000 and this is a post XH structure and you can see a lot of pores suddenly appear where the aluminum was placed so it is highly, highly a nano porous structure. Each of these polls the scale being about 300 nanometers indicates this pores may be about close to 20 to 30 nanometers in size well distributed within the material.

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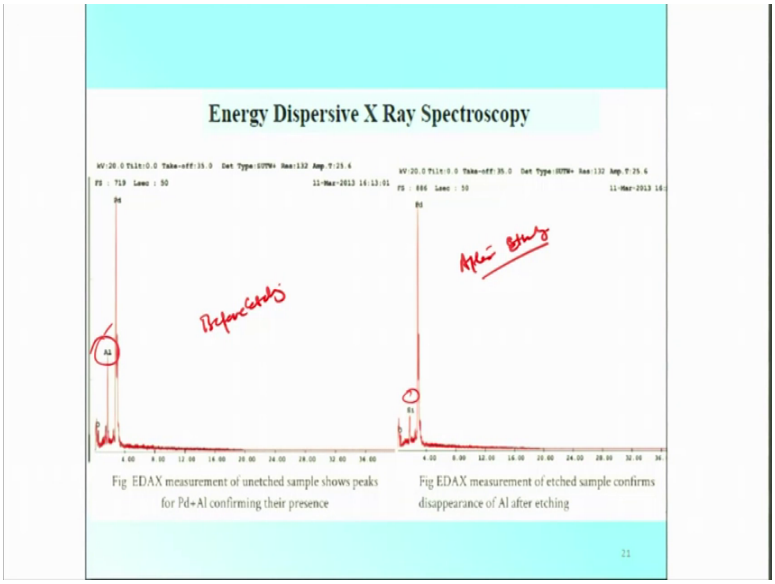

And you know this is also shown up in this energy dispersive x-ray spectroscopy where in the previous prior etching or prior to etching step there is a huge presence of aluminum which goes away and there is a presence of only palladium which comes out you know aluminum is gone completely because of the etching action after the etching. So, this is before etching and thus XRD shown later on is after etching.

So, we are kind of sure that chemically aluminum is removed and there is porosity through the images so it is the aluminum really which is getting removed. So, that is conclusive out of these two experiments.

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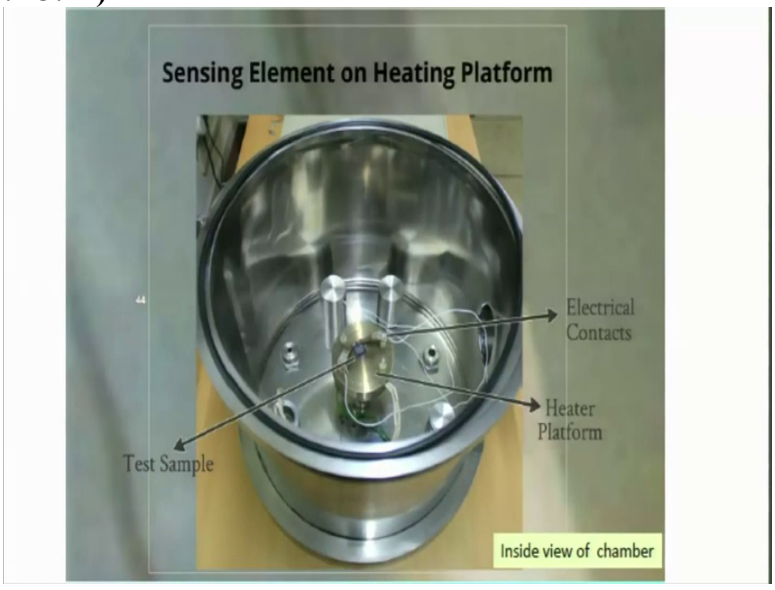

Now we want to test these small platforms which we have developed these small films for their various sensitive sensibilities related to the presence of hydrogen. Hydrogen otherwise the

reducing gas so it will enable some hydride States and try to change the resistance of the material and we can see how what is the difference if it is a continuous material as of to the poorest material that we have developed.

So, for that we have a platform here which does gas sensing inside a vacuum chamber which has a heating stage remember all these elements are highly sensitive at a higher temperature that is the biggest problem with gas sensing that particularly semiconductor metal oxides or even metals they cannot sense so well as room temperature as they can sensor slightly elevated 190 degrees for example 180 degrees temperature.

In the present case it will be about 180-85 degrees at which sensibility will be the highest in this particular case. So, there is also going to be a air inlet outlet where there is a evacuation step followed by and you know a carrier gas which would be otherwise an inert gas in which we can selectively engage or inject through very precise mass flow controllers ppm levels of hydrogen and then as the concentration in the chamber varies and we allow some diffusion time to happen.

We exactly observe the response behavior of this element based on such changes okay. So, there are certain you know so typically what we do is we make a set of inter digitated electrodes okay on a chip scale. So, the electrodes mean you know they are like capacitative kind of structures I am just drawing this for an easy understanding of people okay. So, this is one set the other set that can be there is going to be inside this particular module.

So, let me just do it in a little more precise manner so that there is understanding okay. So, this is one set and the other set is here so these are like inter digitated capacitor plates which are parallel okay. And you know if I put this layer of electrodes through some sensing metals and put some contact points across which we can sense the resistivity typically if there is nothing inter connecting there is no issue.

But if there is a layer of a sensing element which is interconnecting this will actually feel the necessary resistive behavior of the sensing element because that is the only inter connecting element between the different electrodes that is in question. So, these two posts which are the contact posts are used through these electrical contacts given here to sense the resistance of the resistive layer and the changes in resistance therein as we are introducing a different percentage of hydrogen gas.

So, on one hand we are heating up this particular chip you know this chip and on another hand we are using inter digitated electrodes to sense what is the resistance of this particular number.

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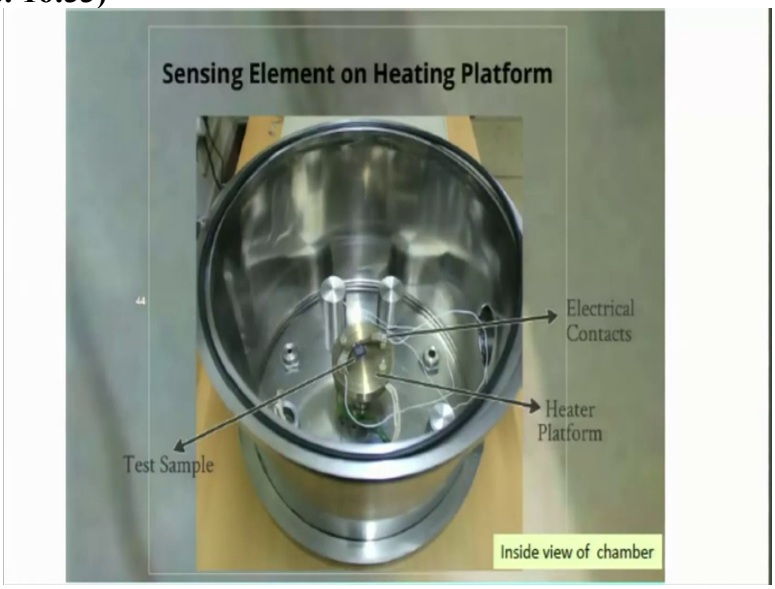

So, these are some results which I would like to discuss and these are published results so you could refer to the detailed paper as well later. But as you look at it that you know if I had different PPM's of hydrogen being sensed in and this is a plot between percentage change of resistance of the film and the time you can see that as the hydrogen is introduced in the chamber from 0 concentration to some different concentration here.

There is an increase in the resistance and then there is a plateauing effect meaning that hydrogen is no further diffusing into the element it is stabilized in terms of its environment and then it actually falls down as the hydrogen is switched off and the carrier gas comes and occupies the place of hydrogen okay. So, this way we have done various plots for different PPM's of hydrogen we have also tried to see the percentage sensitivity that is the percentage change in resistance per unit the original resistance.

And we found out that the percentage sensitivity is probably the highest at about 180 degrees Celsius. So, this is the operation temperature okay so the sensor performance shows about 80% change in the resistivity with respect to the initial resistance at 180 degree Celsius okay. And so therefore this is the operational temperature for the device to any device which will be taking this thin film you know element has to have the ability to go up to 180 degree Celsius for absorbing the film or absorbing the gas.

So, that there is a change in the sensor there is a you know 600 ppm pH2 concentration for which highest sensitivity is again recorded and a cycle time of 28 seconds it is recorded. These are plots which indicate what happens at different temperatures okay you can see that as we are making you know the MFC which injects hydrogen on off the resistance increases first plateaus and then goes down. And so all these different plots suggest that around 180 degrees which is this particular module here the change is maximum.

And the other issue that we can see is also that the recovery also because of the highly porous nature is more or less baselining to the same level as the hydrogen is switched off. Although there is some time cycle involved in the desorption process so that hydrides can convert back into newer pure palladium where the resistance would again be back to the baseline resistance value.

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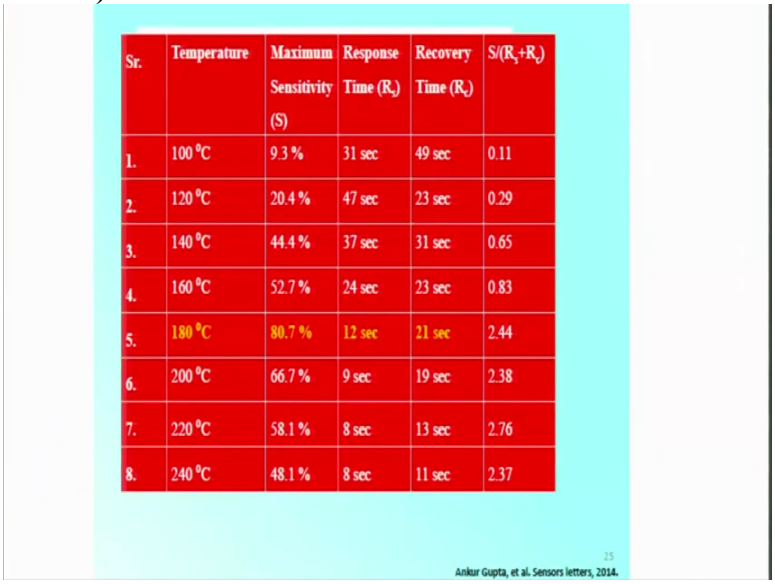

These are some plots which are being you know plotted with the expect aspect of maximum sensitivity recorded as well as you know the response okay and the recovery times. And recovery is basically meaning there by the time that is needed for the baselining effect to happen with the resistance come back to normal resistance and so as a percentage of what is the sensitivity per unit you know the recovery and response time we find out that the best example can be set up through this you know 180 degree Celsius where the timescales are relatively limited it gives a 12 second response time and a 21 second recovery time.

And the sensitivity per unit the total time that is needed for a single cycle is also reasonably high not high as what you can see in 220 but again you know in terms of operability 180 degree

Celsius is a much better temperature to attain than 220 degree Celsius. A lot more power will be needed in 220 degree Celsius temperature and maintaining it at that particular value okay. So, that is how we can and also we see that the percentage response okay is highest at this particular value.

So, 180 degree has 80.7% sensitivity so therefore from a measurement point of view the instrumentation would behave well corresponding to this particular temperature 180 degrees Celsius. So, this was a work which was done some time back in our group for a sensor and you can get an idea of how you design and how you develop sensors on a practical basis.

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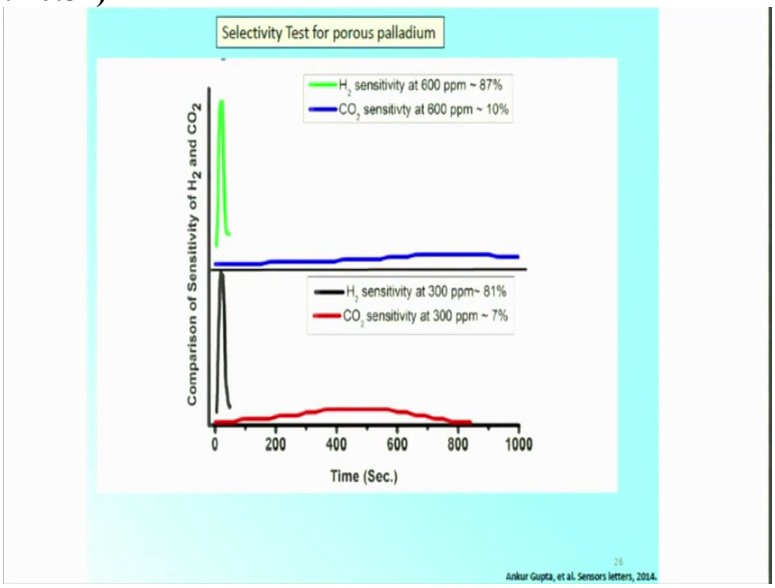

We have also investigated the sensor for the selectivity towards H2 okay. So, let us say there is an ambience created through two different gases H2 and CO2 and we see that if we induce you know both the gases at 600 ppm what is going to be the effect? So, we can see that the percentage change in the resistivity due to hydrogen is 87 % whereas that due to CO2 is only 10%. So, if there is an environment where you have equal ppm of H2 and CO2 it H2 is going to be the dominant you know cause provider for resistance to be varying and resistance to go up to that particular level okay.

And CO2 may not be that specifically being able to get identified so once you prepare a sensing film it is important to see that in mixed gases which set of gases cause the maximum response and who are the interfering gases which may be able to deliver a similar kind of response where there may be confusion okay and false positives. So, this screening off is needed when you develop such a sensor element.

Again another plot at 300 ppm you can see that 81% is the sensitivity of H2 and CO2 is only about 7% this is reflected through the you know the sensitivity plots as a function of time okay and so therefore in this particular time window it may be possible that you know if CO2 does not give many impact at all. Well what this 10% number and this 7% number is based on the whole you know span of the time window.

Whereas the time scales are completely different in case of CO2 sensitivity it takes quite a while for the film to change in the resistance whereas H2 is almost immediate effect so that also helps you determine what is more sensitive and what is less sensitive to a certain specific gas.

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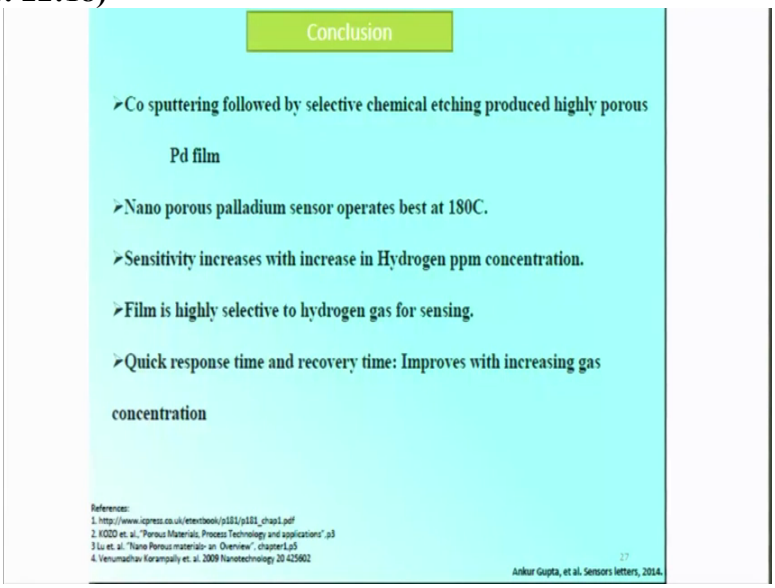

So, you know the in conclusion to this set of experiments what I would say is that you know the coast pattering followed by selective chemical etching is what produces highly porous palladium films and we have seen the performance operational performance of nanoporous palladium at to be highest at 180 degree Celsius. We also saw that sensitivity increases with increase in hydrogen ppm concentration and the film is highly selective to hydrogen gas for sensing.

There is a quick response and recovery time and in general you have designed a thin film resistance based sensor for sensing hydrogen. So, I am going to close on this particular module here in the next module maybe I will keep a small section in the interest of time I had to do this on the micro fluidics oriented Diagnostics okay maybe some 7,8 slides initially. Before we start a completely new topic which is about rapid prototyping and you know how you can handle CNC systems etcetera. So, with this I would like to close this particular module, thank you very much.