

Mathematical Aspects of Biomedical Electronic System Design
Lecture 14
Oxidation and Thickness Characterization

Hi, welcome to this class. Now, what are the process for growing the silicon dioxide, we have taught, we have seen that, there can be two ways of depositing, or growing silicon dioxide onto silicon wafer. The first way is thermal oxidation and the second way is to go for CBD techniques.

We are talking about thermal oxidation, thermal oxidation there are further two processes, one is called dry oxidation and next is called wet oxidation. Again, why we are learning the silicon dioxide? Is because, we want to see a chip, that can be integrated onto biomedical systems of which we will do mathematical modelling and we will try to feed the data, if the model is good the data that experimental data that we get will fit well.

So, with that purpose we are looking at the steps that are required to fabricate a device. Now, having said that like I said we have dry oxidation, we have wet oxidation. So, what is dry oxidation? What is wet oxidation? You see if you have a silicon wafer and if you heat the silicon wafer and pass oxygen, then the oxygen would react with silicon to form silicon dioxide, very simple $\text{Si} + \text{O}_2 \rightarrow \text{SiO}_2$, at a high temperature.

Second is wet oxidation $\text{Si} + \text{H}_2\text{O} \rightarrow \text{SiO}_2 + 2\text{H}_2$, if you have to balance the equation then $\text{Si} + 2\text{H}_2\text{O} \rightarrow \text{SiO}_2 + 2\text{H}_2$. So, this hydrogen is a gas will come out and you will grow the silicon dioxide, both has its pros and cons.

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Dry and Wet Oxidation

Dry oxidation: $\text{Si(s)} + \text{O}_2\text{(g)} \rightarrow \text{SiO}_2\text{(s)}$; **Wet oxidation:** $\text{Si(s)} + 2\text{H}_2\text{O(g)} \rightarrow \text{SiO}_2\text{(s)} + 2\text{H}_2\text{(g)}$

- Both typically 900 – 1200°C, wet oxidation is about 10× faster than dry oxidation.
- Dry oxide: thin 0.05 – 0.5µm, excellent insulator, for gate oxides; for very thin gate oxides, may add nitrogen to form oxynitrides.
- Wet oxide: thick <2.5 µm, good insulator, for field oxides or masking. Quality suffers due to the diffusion of the hydrogen gas out of the film, which creates paths that electrons can follow.

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So, what I am telling here you can see that, dry oxidation you have $\text{Si} + \text{O}_2$ reacting at high temperature giving you SiO_2 , where wet oxidation you have silicon, which is solid silicon. So, bracket is s, water vapor is gaseous, so it is g, and then you get the $\text{SiO}_2 + 2\text{H}_2$, is it not. So, this is how it is done.

Now, if I see the temperature, you can see that both 900°C and 1200°C in between this range you can, you can deposit, or you can grow the oxide. And wet oxidation is about 10 times faster compared to dry oxidation.

So, dry oxidation another limitation is that, you can have a thin layer of oxide about 0.05 to 0.5 micron, however it is an excellent insulator and for the gate oxide, for, for students who understand the MOSFET, a MOSFET is a transistor and you have let us say n^+ , n^+ you have a gate layer, you gate oxide layer. This thin layer of gate oxide that you talk about, it is, it is grown using dry oxidation process, it is grown using dioxide process.

And then on that you have a gate, you here you also you have oxide, so it is protected with oxide, is oxide here and then you have you have to open the contact to get your source and to your drain and this is your gate.

So, for source and drain you open the silicon dioxide from window and silicon dioxide, this window is open using photo lithography process that we will see in some time, this is your grate, this is your source, this your drain, gate, source, drain this is your thin layer of SiO_2 and this thin

layer of SiO₂ should be an excellent insulator and we use dry oxidation for the same. So, that is for gate oxide, this is what it means.

And then you, you have to have wet oxide, where wet oxide you can grow a thick layer of oxide, however it is a good insulator for field oxide, or masking, but it is not used for the gate oxide. So, wet oxide cannot, we do not use it for gate oxide, but it can be used for field oxides, or masking oxide.

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- Room temperature Si in air creates native oxide: very thin ~1-2nm, poor insulator, but can impede surface processing of Si.

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So, room temperature oxide also create silicon, if you put silicon in air it also creates a native oxide and the thickness of the native oxide is about 1 to 2 nanometer, it is in poor insulator and whenever you start the process, you have to remove this oxide by dipping the wafer in HF, HF is hydrofluoric acid, hydrofluoric acid is the HN for silicon dioxide, that is why we dip the wafer in HF for about a few seconds to remove the native oxide before we start the process, even new silicon wafer would have a native oxide if kept in air. So, that is the reason of removing the native oxide, we do not use native oxide for, for any process.

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Dry and Wet Oxidation

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- Room temperature Si in air creates **native oxide:** very thin ~1-2nm, poor insulator, but can impede surface processing of Si.
- Volume expansion by 2.2× (=1/0.46), so SiO₂ film has compressive stress.

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So, this is the volume expansion, if you want to see how it works and it is $2.2x = \left(\frac{1}{0.46}\right)$. So, silicon dioxide if you see, it has a comprehensive stress and whenever you fabricate a device like piezo resistor, a piezo resistive cantilever to compensate this compressive stress you have to grow silicon nitride at the back side of the wafer, what I mean is if you have seen the cantilever, cantilever is a silicon wafer.

So, here the cantilever if you deposit silicon dioxide, it will have a stress compressive stress and to compensate the stress on the back side of the wafer you grow a silicon nitride, or deposit a silicon nitrate. So, that is what I was telling about the compressive stress is created by because of the silicon dioxide material.

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Thermal Oxidation Methods of Silicon

Wet oxidation using H_2 and O_2 is more popular (cleaner) than using H_2O vapor.

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Now, if you want to grow silicon dioxide, again there are three different techniques, first one is dry oxidation, second one is wet oxidation, but there is third one which is called pyrogenic oxidation. So, let us see one by one, the first one that I am showing you on the schematic, if you see the schematic, then you have a oxygen, you have a nitrogen, you have this is a furnace and this is called a horizontal tube furnace, horizontal tube furnace, temperature is about 1100°C.

And when you load the wafer you can see here, it is shown like you have a slot on to each slot you are loading a wafer, and this is quartz. So, it is a very high melting point you can, you can load the wafer onto this and load the entire 25 wafers, you generally you get a set of 25 wafers, all way in all the way into the for horizontal tube furnace.

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Thermal Oxidation Methods of Silicon

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Now, how does it work, how does it work? You can see here, there is oxygen and nitrogen, oxygen, nitrogen. So, initially when you load the wafer when you load these entire wafers into the horizontal tube furnace, the oxygen valve is switched off and nitrogen valve is switched on, that means you are loading the wafer in the presence of nitrogen. So, there is no reaction, there is no formation of oxide onto silicon wafer.

Then once you have loaded the wafer in into the furnace and the temperature is also optimized 1100°C , I told you it is between 900 and 1100°C. So, suppose I want to grow oxide as 1100°C.

Now, also it depends on not only temperature, but also the time, longer the time thicker the oxide, longer the time thicker the oxide.

So, first initially I will start, or open the nitrogen valve, close the oxygen valve and then once the wafer is loaded and the temperature is reached, we will close nitrogen valve and open oxygen valve. So, now oxygen is flowing into this furnace and reacting with silicon. So, $\text{Si} + \text{O}_2 \rightarrow \text{SiO}_2$, dry oxidation.

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Thermal Oxidation Methods of Silicon

Wet oxidation using H_2 and O_2 is more popular (cleaner) than using H_2O vapor.

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Now, you see the second schematic, this is the first one, second one and third one. So, the second schematic what you see here is that you have a heating mantle, this is a heating mantle and that is

nothing but the heater, this is called a bubbler in which you have a water and when you heat the water, it becomes water vapor and there is a mixture of water vapor.

Now, what you do? Initially when you load the wafer you keep this valve off, this valve off, this valve open and nitrogen open, oxygen close, you can see oxygen is closed, valve that goes into the bubbler and that comes out of the bubbler is closed, valve that directly goes to the furnace is open and nitrogen valve is open. Then you load the furnace, already load the wafers into the furnace.

Next step is, when the wafer is loaded your temperature is reached, oxygen temperature is reached, then what you do, you now close the valve, this valve open the valve that goes in the bubbler, open the wall that goes out of the bubbler, close nitrogen valve, open oxygen valve. So, what will happen? Oxygen will go here, it will enter through here, it will carry out the water vapor and it will reach to the furnace.

So, when this happens then you have $\text{Si} + \text{H}_2\text{O} \rightarrow \text{SiO}_2 + \text{H}_2$, but you have to balance this equation. So, if you have $2\text{H}_2\text{O}$, then this equation gets balanced, otherwise you cannot form SiO_2 . So, SiO_2 this is in gaseous form, this is solid, this is again solid and this is gas. So, this 2H_2 will, will come out of the furnace and there is a growth of SiO_2 onto silicon wafer.

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Thermal Oxidation Methods of Silicon

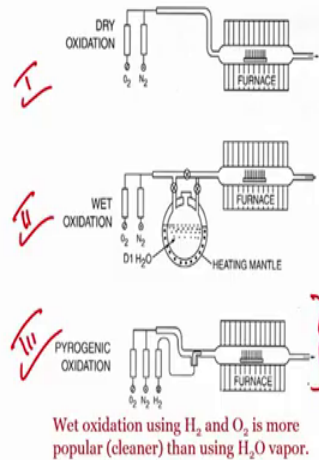
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Thermal Oxidation Methods of Silicon



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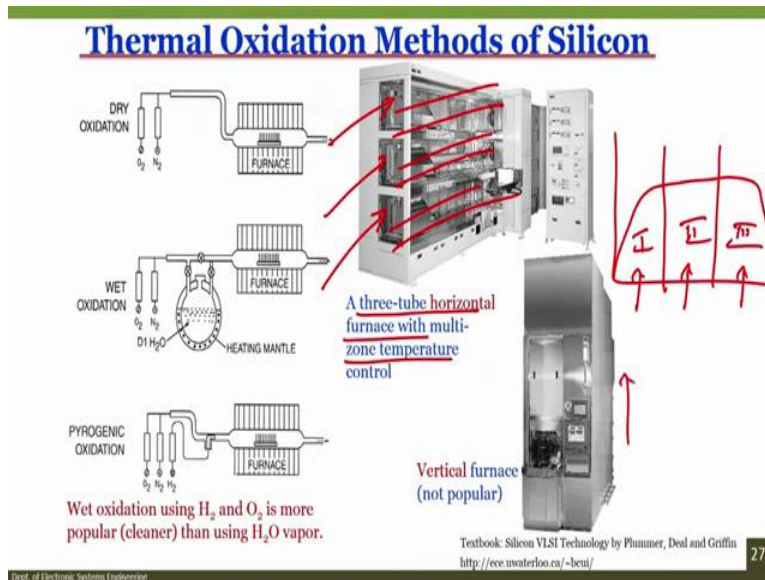
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The third step which is called pyrogenic oxidation, in this what we do if you see the third step third schematic you will see that, there is a connection for O_2 , connection for H_2 and connection for N_2 , nitrogen, oxygen, hydrogen.

Now, what you do in this case is, you initially, initially open the N_2 keep N_2 valve open, close oxygen valve and close hydrogen valve H_2 and O_2 valve is closed. Then N_2 is opened and in the presence of nitrogen the wafer is loaded, once the wafer is loaded into the furnace and it reaches its optimum temperature, then what you do, you open H_2 and O_2 and close N_2 . If I open H_2 and O_2 , then H_2 will be here, O_2 will come from here, react with H_2 to form H_2O , and will enter the furnace as a vapor and this vapor will form the SiO_2 .

So, in these all three cases, the wet oxidation using H_2 and O_2 is more popular and cleaner than H_2O vapor. So, this particular process is much more cleaner, however generally we use wet oxidation, because it is faster, and dry oxidation, because it is a very good insulator.

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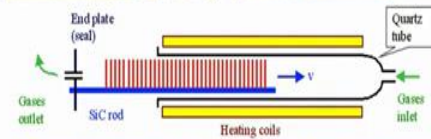
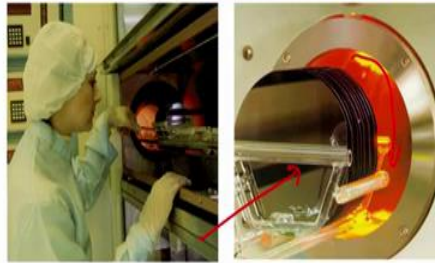


You can see that, this is how the horizontal tube furnace looks like, there is a multiple zone temperature control, that means that if you see here and if I draw the plot, then I will have first zone, then I have second zone and third zone, one zone, second and third, all three zones can be independently controlled at a different temperature, that is called a three-tube horizontal tube furnace with multiple zone control.

So, tube one can be loaded here, second will be here and third will be here, three tube there are three tube, you can also have single tube alternative furnace. And multiple zone, multi zone temperature control I told you zone one, two, three, each zone within that same tube. So, in this tube also you have three zones, in this one also and then this one also, all three tubes have three zones to be controlled. Then you have a vertical tube furnace, which is shown here, the popular one is horizontal furnace rather than vertical tube furnace.

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Thermal Oxidation Equipment



- The tubular reactor made of quartz or glass, heated by resistance.
- Oxygen or water vapor flows through the reactor and past the silicon wafers, with a typical velocity of order 1cm/s.

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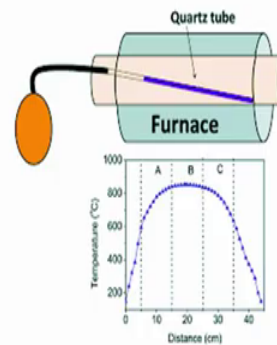
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Here you can very clearly see that, the quartz tube is used, silicon rod is used to push the plate inside and the tubular reactor is of glass, or quartz heated by resistant. And then you can see that, the operator is loading the wafer inside this horizontal tube furnace, the temperature is about 1100 °C, you can see it is all heated mantle and then you are loading this wafer, set of wafers, silicon wafers inside this horizontal tube furnace.

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Steps: Thermal Oxidation

1. Wafers pushed in with nitrogen gas flow
2. Oxidation process at 900°C to 1100°C (generally) with flow of oxygen (dry oxidation) or water vapour (wet oxidation) with nitrogen carrier gas
3. Wafers are taken out with nitrogen flow



<https://pubs.rsc.org/en/content/articlehtml/2019/cp/c9cp03305j>

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Then so the steps if you see, what are the steps, first step is wafer pushed in with nitrogen gas, I told you that one, second is oxidation process between 900 to 1100 °C with flow of either oxygen,

which is dry oxidation, or water vapor, which is wet oxidation and wafers are then taken out again in the presence of nitrogen.

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Steps: Thermal Oxidation

1. Wafers pushed in with nitrogen gas flow
2. Oxidation process at 900°C to 1100°C (generally) with flow of oxygen (dry oxidation) or water vapour (wet oxidation) with nitrogen carrier gas
3. Wafers are taken out with nitrogen flow

The diagram illustrates the thermal oxidation process. It shows a quartz tube furnace where a wafer is being processed. A graph plots Temperature (°C) against Distance (cm), showing a temperature profile with regions A, B, and C. A hand-drawn diagram shows a cross-section of a silicon wafer with SiO₂ on both sides and a 1µm thickness.

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Now, once you have the wafer, let us say if I have of silicon wafer and I say that I have grown 1 micron silicon dioxide, because the silicon wafer you kept it in this format, inside the horizontal tube, if oxygen is there oxygen will react here, oxygen reaction the back side of the wafer, that is why when you show the cross section of the silicon wafer, which is oxidized silicon wafer, you have to show that the oxide is on the both sides, silicon dioxide on front side silicon dioxide is on the back side, and the silicon is a substrate.


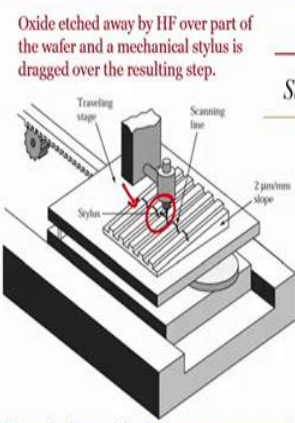
Now, I want to know whether the silicon dioxide suppose I say that it is 1 micrometer, how we know it is 1 micrometer, it can be 0.1, it can be 0.5, can be 1.5, how you know that it is 1 micrometer.

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Thickness Measurement

Surface Profilometry: Mechanical

Oxide etched away by HF over part of the wafer and a mechanical stylus is dragged over the resulting step.



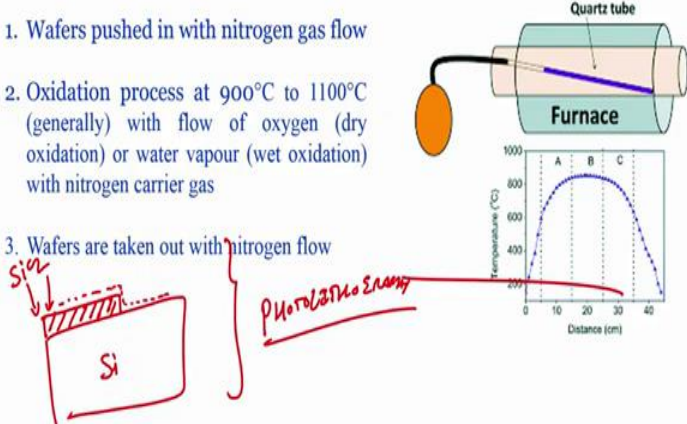
AFM can also be used for thickness measurement. (AFM: atomic force microscopy)

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Steps: Thermal Oxidation

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3. Wafers are taken out with nitrogen flow



Quartz tube

Furnace

Temperature (°C)

Distance (cm)

SiO₂

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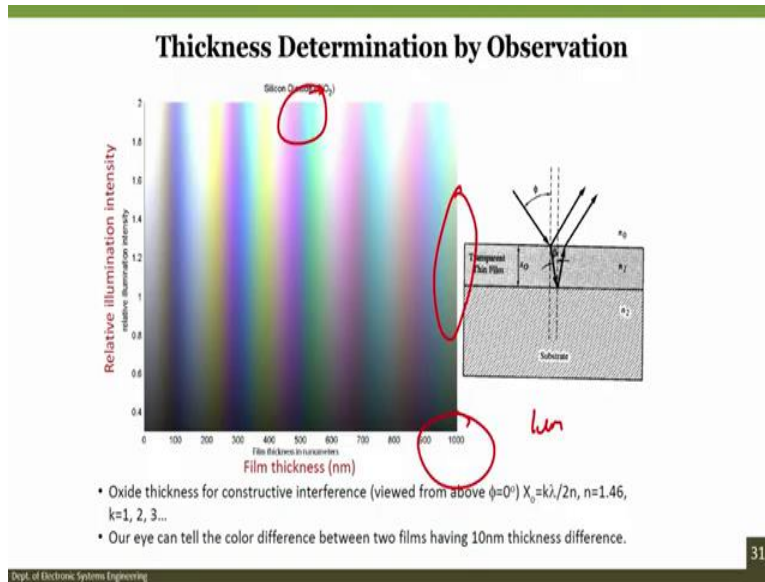
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So, there are two ways of measuring it and one crude way, the first one is the surface profilometer. So, as you can see here, there is a stylus, stylus is generally made up of diamond and it will move across the steps of the wafer, that means that I if I take this wafer and I have to perform a photolithography, that I will teach you in the next class as a part of the experiments.

So, you take silicon and then you have silicon dioxide and you create a step like this, silicon, silicon dioxide you create a step, this step is created using process called photolithography, photolithography. So, here when you load the stylus and you move the stylus in this way, what will happen that, when the stylus moves here, there is a corresponding change in the voltage.

So, you can see here that, when the stylus is moving, it is a traveling stage, stylus is moving in this direction, as there is stylus moves from here to here, there is a change in this step, because of this step change, you can easily see that what is the thickness of the step in the U I, U I, is a user interface. So, the way to create is of course lithography I told you.

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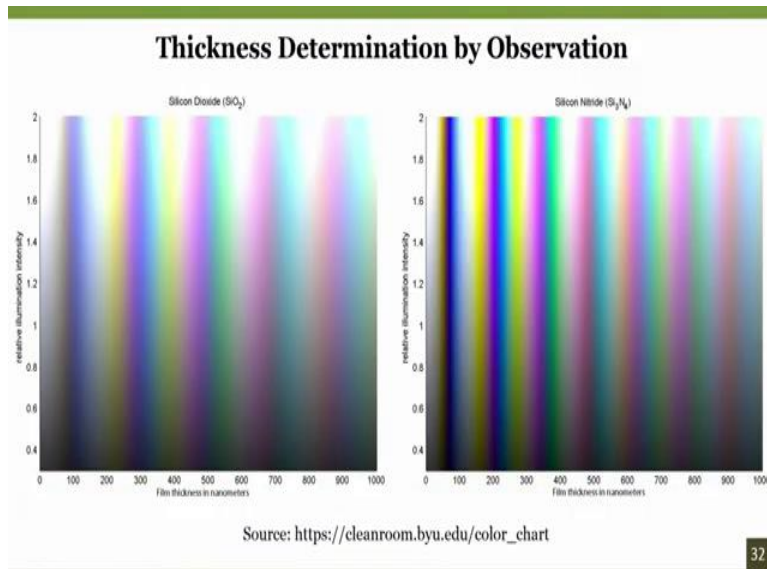
And then there is another method which is little bit crude method, in which you can just look at the color and because the silicon dioxide is transparent in nature. So, whenever you incident a light it will reflect back depending on the thickness of the oxide, you will have the construct interference can be given by

$$x_0 = \frac{k\lambda}{2n}$$

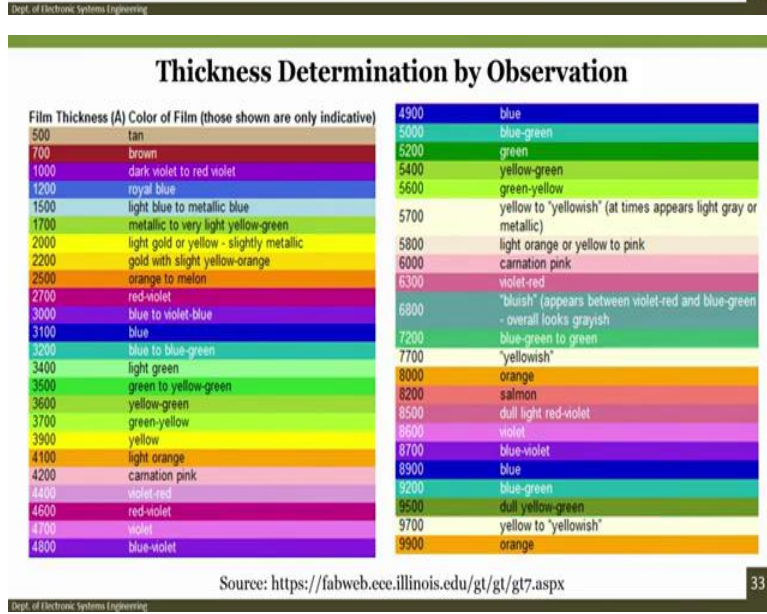
where n can be 1.46, k can be 1, 2, 3.

So, based on that, you based on the thickness you can differentiate, what is the thickness of silicon dioxide, sorry based on the color you can differentiate, from you can, here 1000 micron, nanometer means 1 micrometer, 1 micrometer silicon wafer should look something like this, compared to a wafer which is about 500 nanometer, it will have some pet, pet some visible light in this particular intensity, or color you will be able to see. So, depending on the thickness, the color of the silicon dioxide, on silicon would change.

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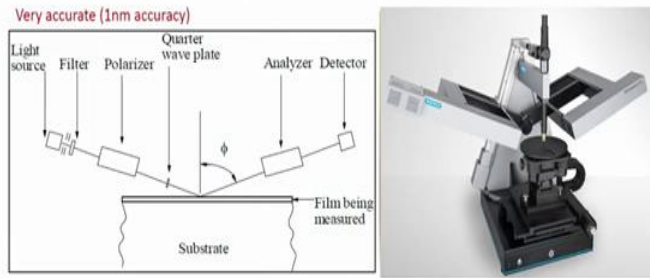


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Again, these are just understanding silicon dioxide versus silicon nitride, how the thickness, varying thickness will change the colour that we can observe. In reality like I said it is very crude method, and you have to be a super expert to understand, what is the thickness to be very precise. However, in reality the color based on the color this film thickness can be right from the 500 all the way to 9900, and you can see the change in the color from brown, dark violet, royal blue all the way to orange.

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Ellipsometer: Optical Thickness Measurement



- After quarter wave plate, the linear polarized light becomes circular polarized, which is incident on the oxide covered wafer.
- The polarization of the reflected light, which depends on the thickness and refractive index (usually known) of the oxide layer, is determined and used to calculate the oxide thickness.
- Multiple wavelengths/incident angles can be used to measure thickness/refractive index of each film in a multi-film stack.

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There is another technique, which is called an ellipsometer, and here the optical thickness measurement can be done for the film which are transparent in nature. Because the light has to pass through it and it can reflect back. So, the it is very accurate in particular about 1 nanometer accuracy is there.

And then there is a light source, there is a detector in between, there is a filter polarizer quarter wave, and the, the advantage of this is it is a non-destructive method to measure the thickness of the silicon dioxide.

So, next point after this one I want to take care is lithography and what is photolithography, we will see in the next lecture, you just go through these particular slides and see that how we can grow silicon dioxide, what are the technique to grow silicon dioxide. And what are the measurement characterization techniques to measure the thickness of silicon dioxide. So, after this let us see the lithography process in the next class, till then you take care, I will see you next class, bye.