Fundamentals of Materials Processing-2
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Module – 02
Thin Film Deposition
Lecture - 20A
Thin Film Characterization

Welcome to lecture 20 and in this lecture we are going to discuss various techniques which we can use to characterize thin films.

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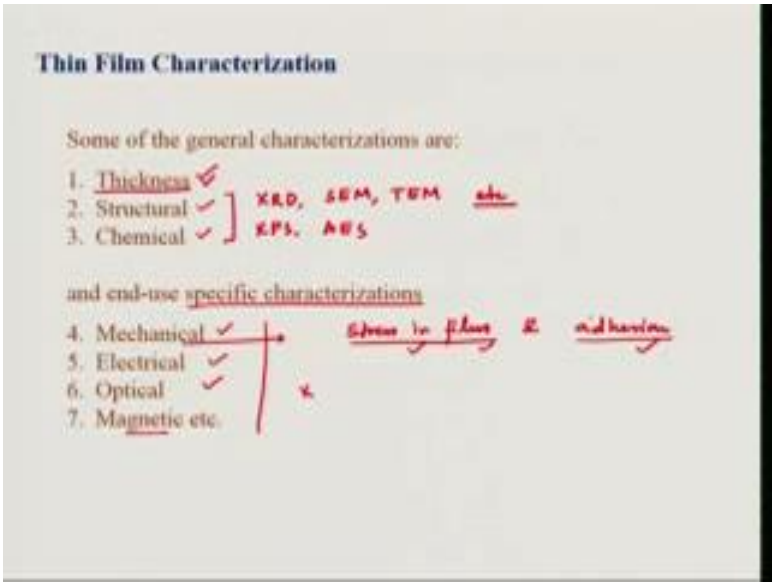

Now, for thin film characterization we are looking for that there are many properties of the thin films that we would like to know after we have deposited the thin films. The most important property we would want to know is the thickness of the film of course, we are depositing thin films, but we want to know what is the thickness and can be controlled.

Then we also want to know the structural properties which are morphology, the grain size, the material if it is polycrystalline what is the orientation? What kind of material is present phases, all those kind of things and also chemical properties? What kinds of elements are present? What is the stoichiometry of the film, if we have the two different elements in the right proportions or not? So, these are some of the general

characterizations. But then you also have you might want to use some specific characterization techniques, which will be for the specific application that you want to use for this thin film for. If there any mechanical applications are required like hardness, you would want to do mechanical testing stress, strain, if it is a thin film which is used for any electronic devices or electrical devices.

Then you would want to know the electrical properties, also if it is for optical applications then you would want to know the optical properties of the thin film. If it is for any magnetic applications then you would want to know the magnetic properties hence there will be many more depending on your application. In this lecture we will not discuss these because these are very specific to the type of film and the end use application and it can be tailored. We will also not discuss these two structural and chemical characterizations, because there are various techniques which are similar to what we you use for bulk materials like XRD, SEM, TEM and etcetera.

So, there are many techniques between this you can also do chemical analysis of the thin film as well like XPS, auger electron, microscopy or spectroscopy, AES. So, we are not going to discuss these techniques because these are similar to what you use for any material characterization. We are mainly interested in thickness because you usually in material science do not measure thickness unless you are depositing a thin film or a coating. So, we will discuss thickness measurement and in mechanical properties will discuss stress in films and also adhesion. We know stress and strain in the bulk of the material, but sometimes as we had discussed in the how the thin films grow there are sometimes stresses in the thin film we call it the mismatch. So, we will discuss the stresses in the thin film and also an important parameter how well it is attached the thin film is attached or adhere is it adhering to your substrate, this is an important property for thin films right thin films should not start to peel off. So, we discussed thickness of the thin film, stresses in the film and their adhesion to the substrate. So, these three characterizations will discuss in this lecture.

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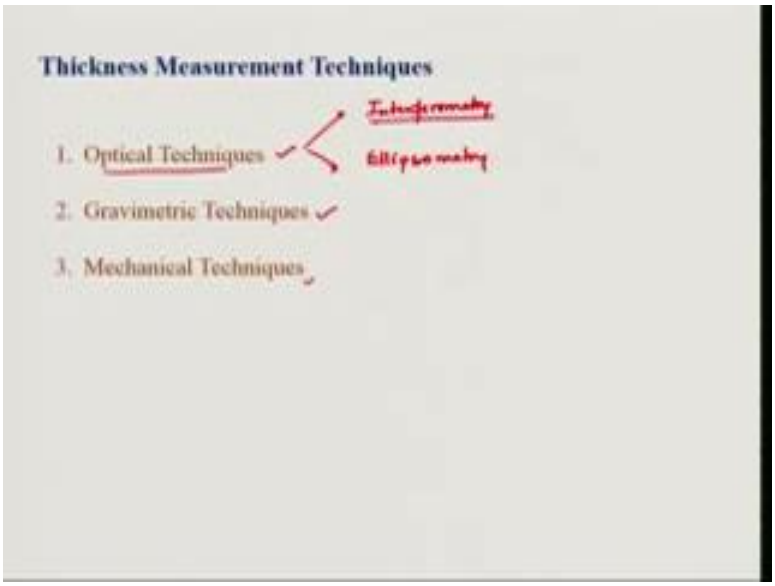

Now for thickness measurements, there are various ways to measure the thickness, there are optical techniques, there are gravimetric techniques and they are mechanical techniques. In optical techniques also we can have interferometry or the second one is called ellipsometry; we will discuss these two techniques and the techniques within these two methods of optical techniques; we will discuss gravimetric technique and mechanical techniques, how to measure that the thickness of the thin film and which technique to use for what kind of material and in what situations. So, first we will discuss optical techniques by interferometry.

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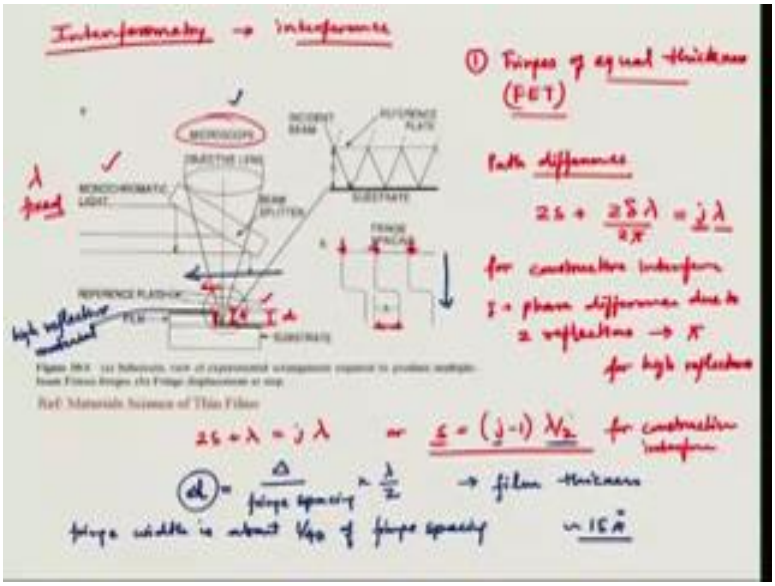

Now so this is interferometry, now we know that when from a surface the light is reflected and in the reflected light if between two rays there is a path difference, then we will have interference parameters right we will have either. So, this is based on interference and based on the interference if it is constructive or destructive will get the fringes and this constructive and destructive interference will depend on the path difference in the path of the two rays which will give as to interference parameter. So, this is a basic framework in which this techniques work.

For this method which is called fringe fringes of equal thickness or FET, now this is the schematic showing how you would do this measurement; first of all you should have a step on your film. So, you take your thin film you make a scratch, you remove the film from certain part such that you have a step height which is the thickness of the thin film. This is d thickness of the thin film and then you have a reflective coating on top, you want the light to be reflected from this so that you can use this light rays to form the interference better. Now there is also a reference plate and also in this method you use a monochromatic light. So, your lambda or wave length of the light is fixed lambda is fixed. Now how you do it that you have this you through an objective lens or a beam splitter, you shine the light, so this beam splitters reflects the light onto a substrate and then you scan this onto a surface.

So, you scanned this onto your surface and the lights reflected from the surfaces are collected and observed by a microscope through an objective lens. So, this is the principle of on which this method works. Now this is a reference plate which is slightly tilted, so that you can get multiple reflections from any given point, so that you can have. So, this is your reference plate and you have substrate, if you have multiple reflections they will give you a more reflected light to form interference patterns, higher intensity. Now suppose this distance is S. So, the path difference for 2 rays would be 2S plus 2 delta lambda over 2 pi and should be equal to multiple integer of wavelength right for constructive interference.

Suppose your film uniform you did not had any film, so this is the distance in absence of film. So, you will have this path difference and if these conditions are met, delta is phase difference, phase difference due to the reflections because light is being reflected at two surfaces: one is at the top surface of this and the other is the from this substrate surface in this case when there is no film. So, the light is being reflected. So, there will be you will

have two reflections and every reflection also changes the phase of the light because these lights are nothing, but electromagnetic waves and we are discussing the phase difference in the electric field or you can also discuss the phase difference between the magnetic fields. So, and this can be taken as pi for high reflectance; highly reflective surfaces will have a phase defense of pi 180 degrees. Now if we take this as pi then this equation becomes 2S plus lambda is equal to j lambda or S is equal to j minus 1 lambda by 2. So, this is how the separation between these two films is for constructive interference

Now, if you have a thin film then this like this, then this separation will not be as anymore if you scan your light on top of this in this direction, then this distance was S and then it will become less than S, S minus d right and then this S minus d then this condition changes, you will not get constructive interference or the fringe will shift. So, now, in this you are watching the fringes of high intensity, where that constructive interference occurs, but if you are S changes because of film thickness, then this condition the spacing between these two will also change and this change by delta. So, as you are scanning in this direction from right to left, you observe the change in the print spacing under microscope and from this delta you can calculate the film thickness d as delta over fringe spacing into lambda by 2. So, this will be your film thickness this is your film thickness.

So, now, you can observe the delta divided by fringe spacing, only if you have fringe width is small enough right if the fringe widths are very large and it is very difficult to measure this delta, the change in shift right. So, for highly reflective surfaces fringe width which is about 140 of fringe spacing. So, very small compared to the fringe spacing and about one and this fringe spacing would be lambda by 2 because j is my integer. Since j is an integer so you are spacing with for constructive interference will be lambda by 2. Now and this delta can be measured about one-fifth of fringe width. So, from this you can calculate what would be the detection limit, how much smaller thin films you can observe? It turns out you can observe film thickness about 15 angstrom by this method. So, this is the detection limit, but you have to make a step on to us by scratching or by some other methods. So, that there is a step between film and substrate, also you should coat it with the high reflective material.

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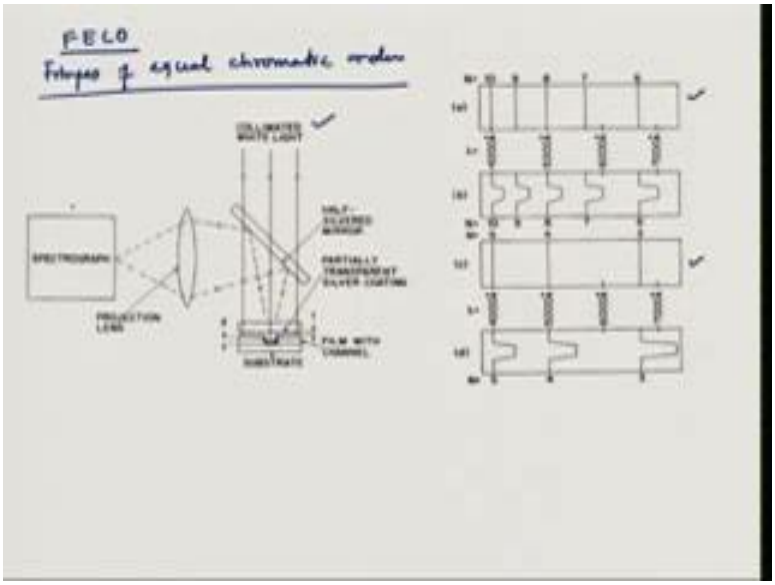

And it should be very thin. So, that it does not add to your film thickness. So, this is one method, the same interferon between methods can be adopted in a different configuration which is called FECO, which is called fringes of equal chromatic order, just term for this method; what you doing this method is slightly different you do not use monochromatic light, you use a collimated white light which has many wavelengths and you have found this and you do not need to scan any more, you just have this scratch again on your thin fill in the middle and again the rest of the things remain mostly the same, here you measure the intensity at different wavelengths.

So, these two patterns are without film and you see which order of the. So, these fringes are constructive interference for different wavelengths because different wavelengths would have different path difference and different condition depending on this equation, different condition for constructive interference and you observe all the wavelengths and from this because of and you see if there is no film then you will get the straight line, but if you have a film you start to see this dip because of this thin film and there is a mission between the film thickness which can be derived from the previous discussion.

We are not going to do go into the exact discussion of that, but you can use a white light and you can you do not have to scan you can just use scan using a spectrograph at different wavelengths intensity, at different wavelengths of that light. So, this is called FECO or fringes of equal chromatic order. For both FET and FECO we need to use

highly reflective surfaces and opaque surfaces, this will not work for transparent surfaces, which will allow the light to or transparent thin films which will allow light to pass through. So, these methods are good for opaque thin films.

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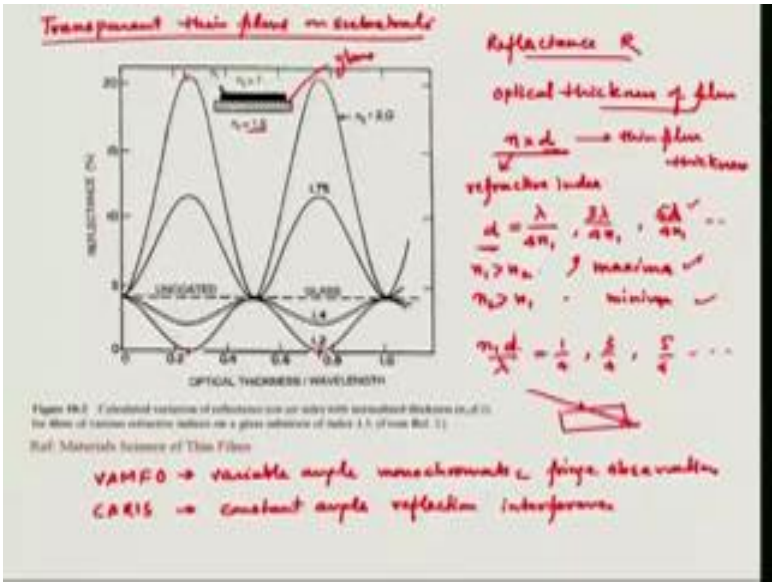

The next method which is applicable for transparent thin films on substrate is based on reflectance R. So, there is no interference pattern in this, you just measure the reflected light intensity and as a function of optical thickness, optical thickness of film and optical thickness is parameter which is n into d. So, d is film thickness and n is refractive index of the thin film material and this is thin film thickness. So, multiplication of the 2 is called optical thickness and in to d. So, if you have a material with under certain conditions, it will give you high reflectance and these conditions are when d. So, this is my configuration there n 0 is here, n 2 I have taken as 1.5 which is my glass substrate and n 1is my thin film of n 1 refractive index and d thickness.

So the d thickness will depend we will get the maxima of the reflectance for certain thickness of the film, which is described by this lambda by 4 n 1, 3 lambda by 4 n 1, 5 lambda by 4 n 1. So, if my d is any of these if it satisfies this condition, then I will get this maximum reflectance and this is for if n 1 is greater than n 2 you will get maxima at these, if n 2 is greater than n 1 then you get minima at these and if you just change this to n 1 d over lambda then you will get 1 by 4, 3 by 4, 5 by 4 like this. So, you will get either maxima or minima at these, now if you see in this figure this is 0.25 you get a maxima or

minima for different values of n 1, either larger than n 2 or smaller than n 1, so 0.25, 0.75 and so and so forth.

So, this is you measure the reflectance, and how do you measure reflectance? You just measure the intensity of the light which is being reflected compared to how much you are is incident on your sample. So, this is the basic principle and again you have to use a setup where you observe using a microscope, how these and you change the optical thickness and you see at what point this maxima occurring and then from this expression you can calculate; if you know n 1 what would be the film thickness? In this method you do not need to have any scratch or any step on your height in your film. So, you do not need to distract your film in this method. Again this can be done in two way because lambda is involved right, so either you can use a method called VAMFO which is variable angle monochromatic fringe observation.

Now in this method your lambda is fixed, you change the angle of the light which is falling onto this. While when you change the angle you are changing the optical thickness because that d part how much distance the light travels in your film is changing right. So, because if you are using this angle so the distance it travels inside the film is going to change. So, from this you can observe different peaks by changing the angle and then you can calculate what is the film thickness. There is another method in which you do not have to change the angle, you use multiple wavelengths. So, you can use constant angle reflection interference. So, you have white light of various wavelengths and then from that reflection pattern you observe the interference and then you see which of these are giving you high intensity and how much of the light is being reflected based on that and you can calculate film thickness from that using some models. So, this is for transplant thin films where you do not need to have any step height.

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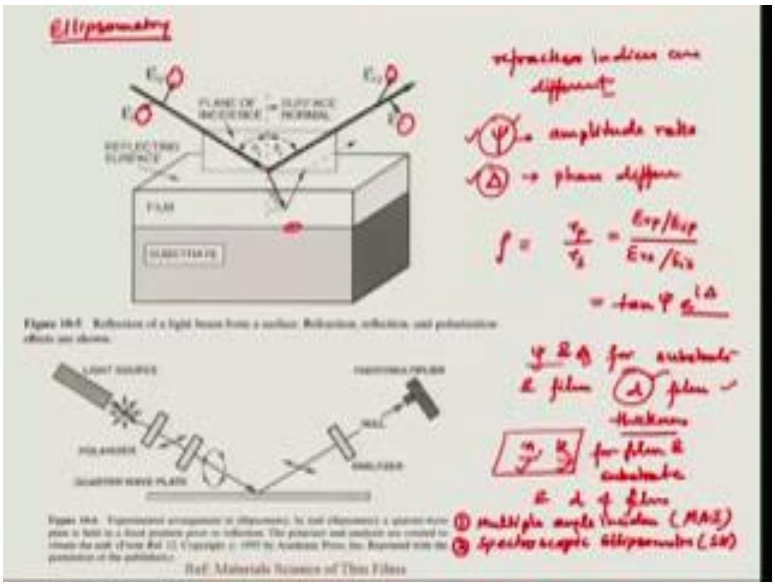

So these methods are based on interference pattern or interferometry. Next is ellipsometry; now if ellipsometry techniques are based on the principle then when a light a polarized light is reflected from the surface it changes its polarization and the phase. So, polarized or a circular polarized light or non polarized light will have both perpendicular and parallel components of the electric field, which are incident onto the surface and when it is reflected their ratio of perpendicular and in plane electric fields will change, and their phase will also change differently. So, once you have this film, you will get this reflectance from the film top surface and also you will get this from the bottom surface of the film and the surface properties the material properties of these two will are different their refractive index indices are different.

So based on the model that how much of different either parallel or perpendicular components of electric fields are reflected, we can have two components delta n psi. So, this sorry psi is the amplitude, amplitude ratio, amplitude ratio of reflected light and this is the phase difference. So, you can define the ratio row as reflected for perpendicular component read by the reflectance for the in plane component and this we can define as electric field i p. So, and this r s, E i s where these are E r electric field reflected for perpendicular component, electric fields for incident perpendicular component and this is for reflected in plane and incident in pain component and this you can define as 10 psi which is amplitude ratio into e to power i delta which is the phase difference.

Now you have various parameters involved in this, you have psi and delta for substrate and film and d which is film thickness; when you are doing this measurement you need to observe this psi and delta, you will observe psi and based on various parameters which are n and k, n is the refractive index and k is the extinction coefficient of that material which means how much of the light is being absorbed in that material, you will use some models to calculate the film thickness. So, you can calculate both n and k. So, because there are 5 parameters involved psi and delta for both film and substrate and also the film thickness their 5 components which all depend on n k for film and substrate and d of film. So, you need to use model based approach to create different models and which model fits the observed values of psi and delta. Again you can use two different approaches in this, one is multiple angle incidence or MAI, which is again your lambda remains fixed and you change the angle of incidence and you observe sigma and delta at those angles you measure sigma and delta and then you correlate by a computer model to n k and film thickness.

Or you can use what is called spectroscopic ellipsometry or SE, in which you do not change the angle of incidence, but you use multiple wavelengths and for different wavelengths you observe psi and delta and then you using our computer model laid back to the film thickness. In this method you only need to know the substrate values n and k film, you can by modeling you can also know the optical constants of the film. So, this is a method of not only measuring the film thickness, but optical constants also of the thin film simultaneously and this is based on a by measuring psi and delta and using computer models to fit the data which describes the film thickness and n and k values. So, this is about optical methods.

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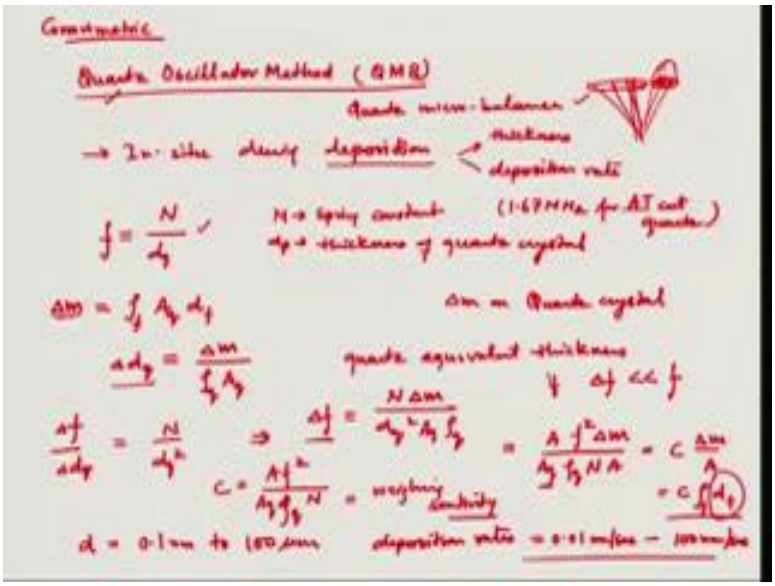

You can also use some gravimetric method gravimetric where you see the mass how much mass of the material is being deposited and correlate that to film thickness. Now this particular method is called quartz oscillator method or quartz micro balance QMB quartz micro balance. Now thin film the mass of the material which is being deposited in few angstrom a few nanometers thin film is very very very small, it is like difficult to weigh that mass right or the change in mass because of thin film on any substrate. So, this is a method which is sensitive to very small changes in mass and moreover it can be used in situ during deposition. So, this is a big advantage of this method over other thickness measurement methods this because you can monitor the thickness while you are depositing the thin film.

Not only the thickness, but you can monitor thickness or also you can monitor deposition rate of course, if you monitor the thickness and with time then you can also calculate the deposition rate. Now this method works on the principle of natural vibrational frequency of quartz. So, vibrational frequency of quartz can be given by N over d q. So, N is a spring constant and d q is the thickness of quartz crystal and this is a 1.67 megahertz A T cut quartz and which is generally used for quartz micro balance and this AT cut is a particular cut in the particular direction of quartz crystal. So, this quartz crystal will have a certain vibrational frequency natural vibrational frequency. Now you can measure this natural vibrational frequency using a piece of crystal, quartz is a piece of material right. So, it has the vibrations you can see so you can observe these changes in this frequency.

Now, suppose if delta m mass is deposited onto your thin film, then it will this delta m is rho of density of thin film, area of the quartz crystal and thickness of the film right. So, this is delta m on quartz crystal. Now how does this change the thickness of the quartz? You can use that this delta mass will change this quartz thickness by certain amount. So, this you can use that changing quartz thickness is delta m divided by rho of quartz and area of quartz. So, you are saying that how much will be the change in quartz thickness if the material being deposited was quartz. So, this is quartz equivalent thickness.

So how much change in the quartz crystal thickness if this material was, if delta m mass being deposited was of quartz, this is not quartz this is a thin film. So, we can use this and from this expression we can get what would you change in frequency with changing quartz crystal and this is N over d q square and this will be a negative sign because of thickness increases you are frequency will decrease, but we are just taking the change. So, with the positive sign there will be a negative sign also here. So, this will give me delta f change in frequency of the quartz crystal will be N delta m divided by d q square and this A q and rho q and if delta f is much much smaller than f the frequency, we can also convert this into A; this N over d q square we can write as N square over d q square divided by N. So, we can write this as A f square delta m divided by A q, rho q N into a. So, this is the area of the crystal now, this is some area of the crystal now we can correlate everything as C delta m over area.

This delta mass over area we can correlate back to C rho up the film thickness of the film. C is the parameter which is A f square divided by A q rho q and N this is weighing sensitivity. So, this is a parameter which can be defined by certain parameters knowing the natural frequency, the spring constants, the area and the density of the quartz and related to the area of the crystal which is exposed for thin film deposition during your deposition process. So, in your vacuum chamber during it sputtering or evaporation, you put this cross crystal with certain electronics right next to a substrate holder, very close to it. So, everything that is being deposited on your substrate is also being deposited onto your quartz crystal and using this change in frequency by monitoring by this by certain electronics, you can measure the thickness of the thin film while it is growing.

And this method is so sensitive that you can for film thickness you can measure 0.1 nanometers to 100 micron thickness also deposition rate, you can measure anywhere in between 0.01 nanometer per second to 100 nanometer per second and this is in c 2, you

do not need to do take out the material your thin film and do any measurement on your thin film. So, your film remains intact, this is a separate quartz crystal on which you are doing this measurement. We will stop with this lecture today remaining few parts will cover in the next lecture.

Thank you.