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

Welcome to this lecture, this is the last lecture of this course and also of this module. This lecture is in fact continuation of lecture 20 in which we were discussing thin film characterization techniques.

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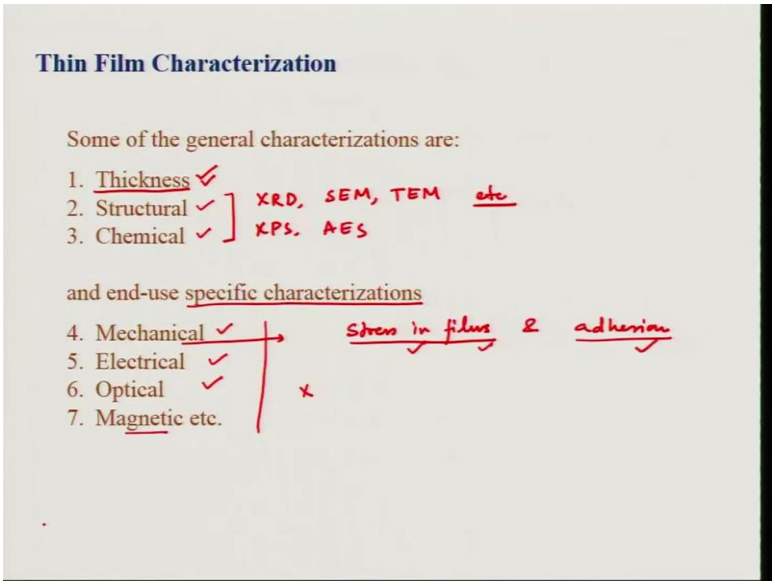

So, let us just recap briefly we discussed that the properties we are going to deal with in this characterization lecture would be mainly thickness and stress in the film and the adhesion of the film because all other properties are either end use related or these techniques are general techniques which are used in material characterization in material science or mechanical engineering in general.

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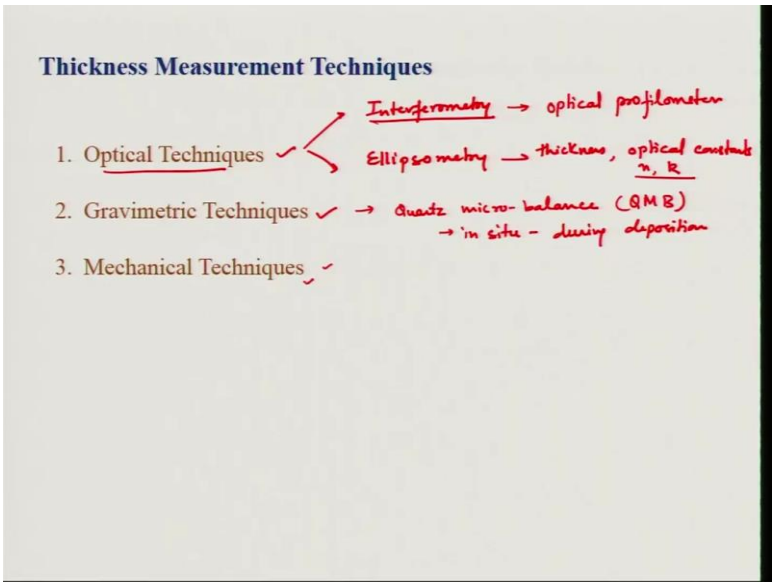

So thickness, stress and adhesion; for thickness measurements we said that there are various techniques, you can use optical techniques using either interferometry or ellipsometry you can use gravimetric technique such as quartz, micro balance QMB and this you can also do in situ; in situ means while or during deposition. So, we discussed the science behind this technique and how this technique is used for measuring not only the thickness, but also the deposition rate and in this lecture we will discuss mechanical techniques of thickness measurements. Interferometry techniques are usually called optical profilometer, ellipsometry techniques are usually used when you need not only thickness but also optical constants such as n and k. If you need to measure these properties as well, you can use ellipsometric techniques and these are mainly used for the electric films where you need to know the (Refer Time: 02:47) constants which are related to these optical constants.

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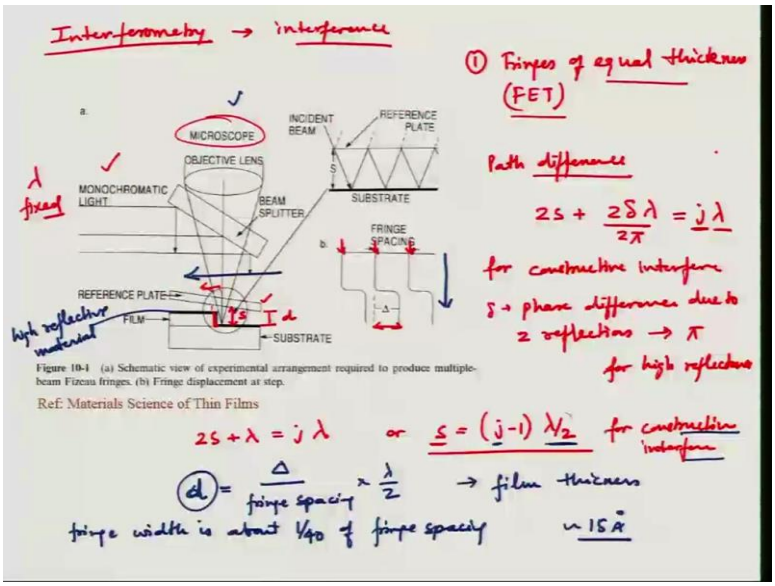

So, we discussed various interferometry techniques based on interference which were FET and FECO.

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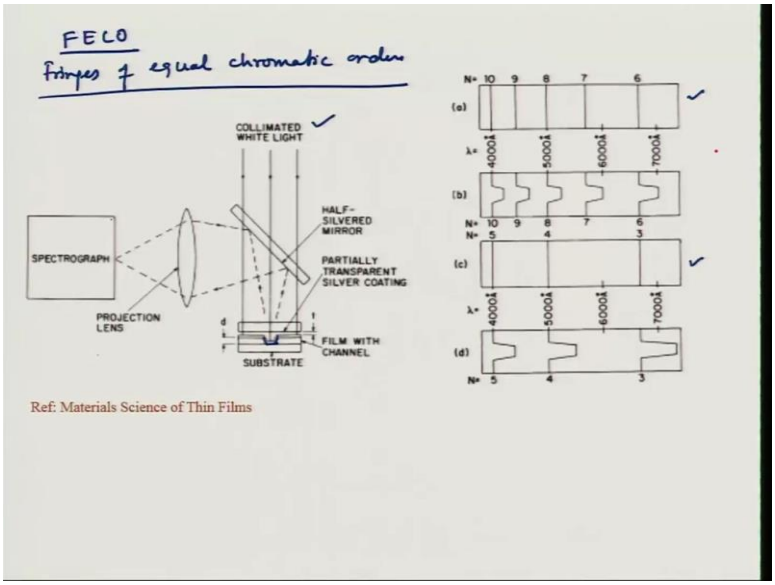

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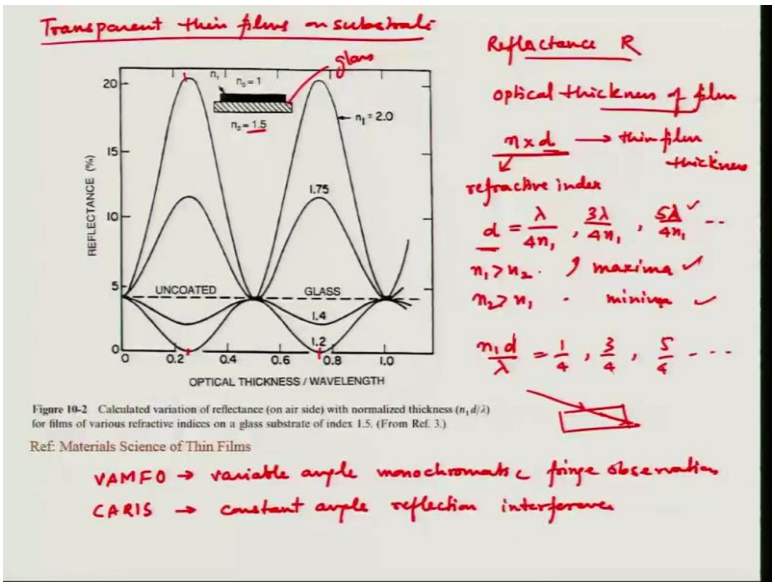

Figure 10-2 Calculated variation of reflectance (on air side) with normalized thickness ($n_1 d/\lambda$) for films of various refractive indices on a glass substrate of index 1.5. (From Ref. 3.)

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We discussed the reflectance, the technique based on reflectance which can be used to make the thickness of transparent thin films.

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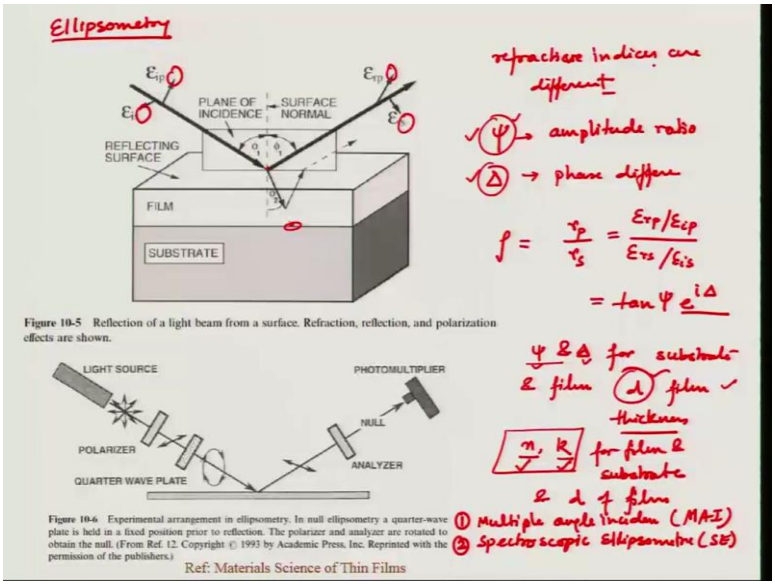

Figure 10-5 Reflection of a light beam from a surface. Refraction, reflection, and polarization effects are shown.

Figure 10-6 Experimental arrangement in ellipsometry. In null ellipsometry a quarter-wave plate is held in a fixed position prior to reflection. The polarizer and analyzer are rotated to obtain the null. (From Ref. 12. Copyright © 1993 by Academic Press, Inc. Reprinted with the permission of the publishers.)

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And we also discuss the ellipsometry technique in which you can calculate thickness and optical constant of the thin films.

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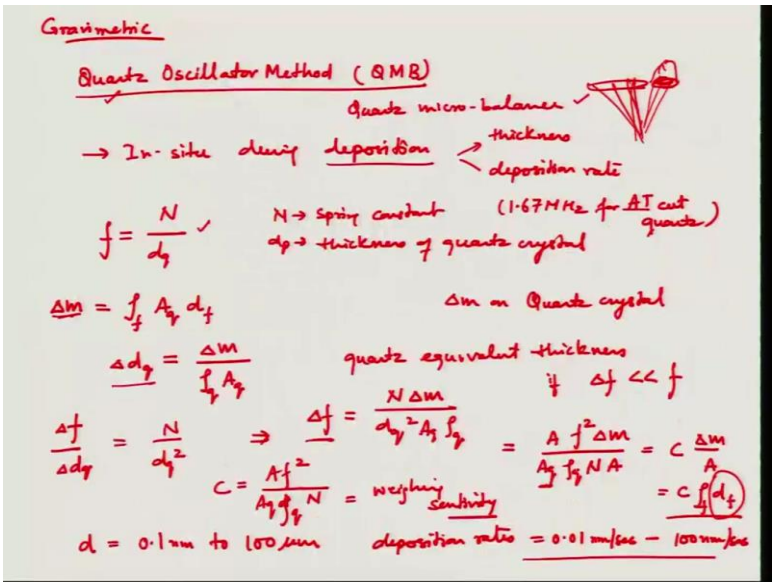

Then we discussed gravimetric technique which is based on change in weight or mass of a quartz crystal when the thin film deposit on it. So, quartz crystal is placed very close to the your substrate on which you are depositing your thin film and the mass which is being deposited on the substrate, the similar thickness is being deposited on the quartz crystal also and this change in mass of the quartz crystal changes is natural vibrational frequency.

So, this change in frequency delta f which is given here is measured by electronics of the system and then it can be related to the thickness of the film, given the density of the film you can calculate the thickness of the film and also the deposition rate and it is a very sensitive technique such that you can measure thicknesses down to 1 angstrom which is 0.1 nanometer all the way up to 100 micron. Also the deposition rates which are 0.01 nanometer per second or 0.1 angstrom per second can be measured.

So, suppose if you want to achieve a certain thickness then you want to close or stop your deposition after that thickness, you can made the thickness during deposition and stop the process or you can calculate the deposition rate and then predetermined how much time you want to give your deposition and this is an in situ technique and this is mainly used for either thermal evaporation or sputtering techniques or mainly for PVD techniques because CVD techniques usually imply higher temperatures for the

deposition and many times this is some surface chemistry is also involved; this is another in situ technique.

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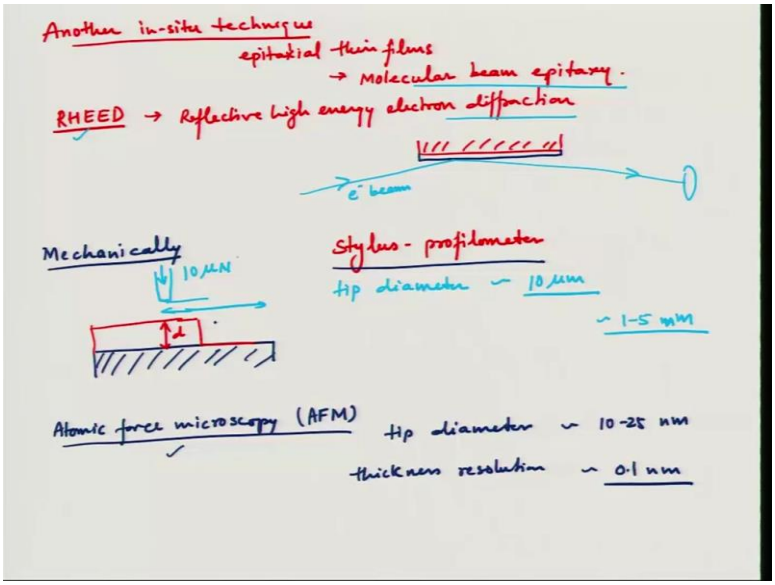

Let me which can be used in situ technique; which is used mainly for epitaxial thin films. So if you are depositing an epitaxial layer; layer by layer this technique can be used and this is mainly used for molecular beam epitaxy and this technique is called RHEED which is stands for Reflective High Energy Electron Diffraction. So, in this technique what you do is that on your substrate, you have this thin film is being deposited on top and this you have a high energy electron beam which is coming on to us your substrate and then there is a detector.

So and this you can see the formation of monolayer; one single layer coverage and it is very sensitive. So, if the monolayer coverage is not complete there will be a difference at different places in the step height which would be one atomic layer thick and then you will see the oscillation in the diffraction pattern on the detector and once this monolayer is complete then you would see that those oscillations stop. So, based on this; this is electron beam, you can determine how many monolayers are being deposited and you can stop the process after your desired number of atomic layers or moleculer layers are being deposited and so in this way you can measure the thickness of the film and control the thickness of the film in situ, while you are depositing this film. Of course, this principle only works when you work with in high vacuum systems. So, and since

molecular beam epitaxy works in high vacuum then this technique is good for molecular beam epitaxy; for measuring thin film deposition.

Now, will turn our attention to how we can measure mechanically. Mechanical measurements of thickness again in this you would have a substrate, be the thin film, but again you need to make a step either by covering this area during deposition or by removing the film mechanically after deposition so that you have a step which you want to measure and this is a thickness of the film d. Now you use a stylus and this techniques call as stylus profilometer; in this technique you use a mechanical or a stylus which is usually diamond or very hard material like silicon carbide tip and you apply certain force to this usually around 10 micronewton and then you scan this tip on top of your thin film with this step and you monitor the change in position of this step. So, as it scans over the top of this as soon as gets there, this tip goes down because of this load and then it will detect the change in position of the film which can be detected by a monitor and can give you the film thickness.

So this will give you a film thickness, the tip diameter for this is usually in the range of 10 micron. It will not give you very good lateral resolution, but will give you very good vertical resolution; it will give you vertical resolution of the order of around 1 to 5 nanometer. So, you can get the thicknesses with the resolution of 1 to 5 nanometer thickness, but since the tip diameter is very large; you will not be able to see this sharp as because the tip will not allow you to (Refer Time: 11:03) here, so there will be some loss of lateral resolution.

Another technique which is very similar and so this is stylus profilometer, another technique is called atomic force microscopy or AFM. In this technique for thickness measurement, you do essentially the same technique but here the tip is very very small; the tip diameter is order of 20 to 25 nanometer and your load is very small and it is attached to a piso crystal, so any small deviation in the position can be detected. So, in this the tip diameter is around 10 to 25 nanometer and also the thickness resolution is around 0.1 nanometer, so you can also measure very very thin films and the roughness of the film using atomic force microscopy and this is very very sensitive technique. So, you would usually use for very thin films, very smooth films. So, these are some other techniques which you can use, again you will have to have this step for both the technique you have to make this step either during deposition or after the deposition so

that you can measure the step height which is your film thickness. So, this is general overview of thickness measurement of the thin films.

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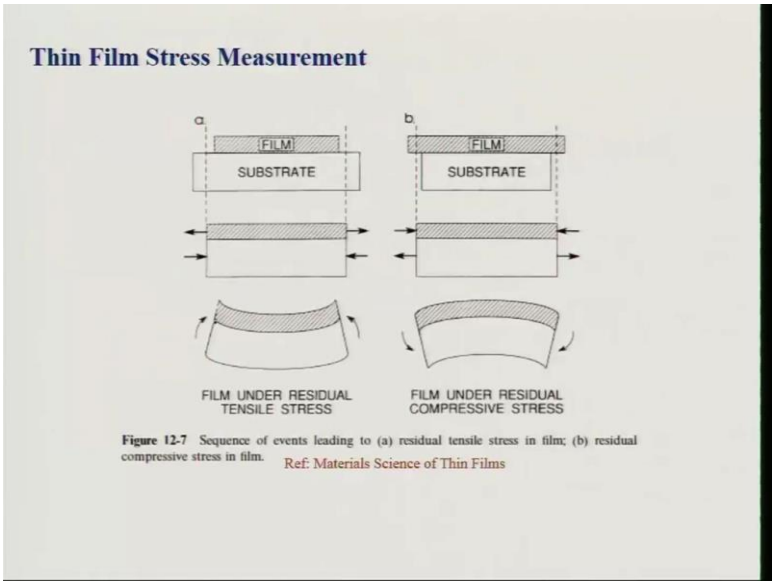

Now we will come to stress, we were discussing very often these thin films have stress and these stresses are due to two reasons. First they are different material, so they would have different lattice constants and different addition. So, when you try to match substrate and film, which have different constants; so often the film will be under stress. Now there is another reason that many times you deposit this films at a higher temperature, specifically in chemical vapor deposition techniques; you deposit these films at higher temperature where and the temperature expansion coefficient; coefficient of thermal expansion of film and substrate do not match.

So, as you cool down the lattice constants of the film and substrate which are different material will have different shrinkage and after deposition and this will produce another stress in the film. So, there many reason for the stresses in the film, lattice mismatch, different morphology, difference in coefficient of thermal expansion all of these will lead to stress.

Now, if the film is under stress it will lead to some kind of curvature in the substrate and the film. So, if my substrate well was under compressive stress and the film is under tensile stress then it will lead such that this bending moment on the two edges and it will lead to this curvature, warping of the substrate which where you cannot see optically by

naked eye because your substrate is usually very thick. So, you will not be able to see this, but we have techniques that can measure this bending and then relate it to the stresses in the film.

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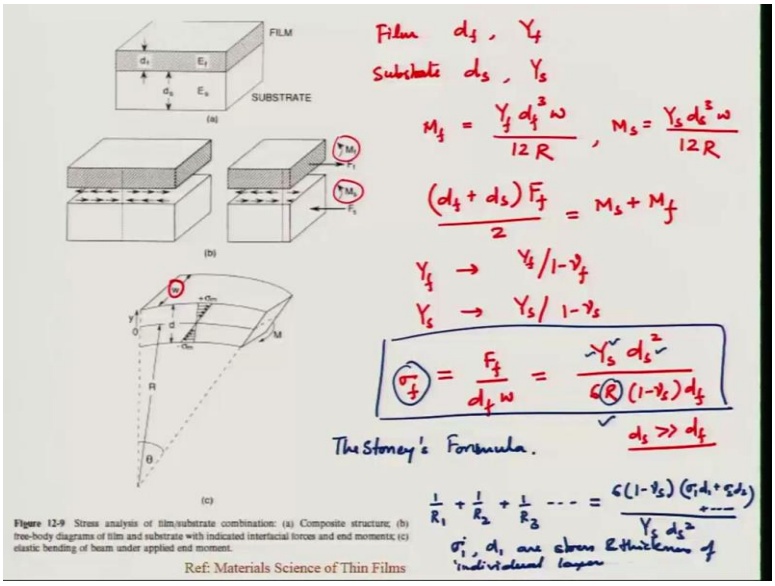

So here we show how this technique works; so we have films which have different film thickness and we relate it to the stresses in the film and with the Young's modulus. So, let us say material properties are such that the film thickness is df and Young's modulus is yf and so this is for film and substrate, the thickness of the substrate is ds and Young's modulus is ys. So, based on this the stresses and the null plane where there is no stress, we can determine which kind of stresses would be present based these bending moments for the film and the substrate. So, the bending moment for the film will be given by yf, df cube w; divided by 12 R and R is the radius of this curvature of the combined substrate and film bending. Similarly Ms can be given as ys, ds cube w divided by 12 r.

Now, this two bending moments together should be equal to the force, this equal and opposite force ff and fs. Since these, if you do a force balance these force are equal and opposite so we can take either of these two and the distance which will balance this bending moments will be the thickness of those films. So, we can say that df plus ds multiplied by film thickness or sorry force on the film or the substrates both are equal divided by 2 because we are taking bending moment due to both should be equal to the total bending moment in the system and we can also relate this since this is not a one

axial bending, because it is a plane right. So, we need to replace yf by yf divided by 1 minus nu f; nu f is the poisons ratio. Similarly ys we can replace by ys, divided by 1 minus nu s, using these we can calculate the stress in the film which is sigma f, the stress is nothing, but force divide per unit area.

So, if you now calculate force using these expressions we can calculate the stress in the film which will be nothing, but force divided by df into w; w is this width of the film and this will be ys, ds square divided by 6R; 1 minus nu s, df and this expression is valid for ds much larger than df. So where the substrate thickness is much larger than the film thickness, so this expression is called Stoney's formula and this can be used by measuring r and knowing these constants.

So, the thickness of a substrate is known, the thickness of your film is known; Young's modulus of your substrate is known and the poisons ratio of a substrate is known, then you can use this expression to calculate the stress in the thin film. So, this is you can do but important thing is that you need to know the radius of curvature. Suppose, if you have a multilayered structure, you have many layers. So, then if you want to know the total stress in the multilayer stress, you can combine all these radiuses as 1 over r 1 plus 1 over r 2 and due to each layer, the curvature due to each layer and then you can this equation will be given by sigma 1 d 1 plus sigma 2 d 2 and so on so forth divided by ys; ds square, where sigma 1 and d 1 are stress and thickness of individual layer.

So, you can not only (Refer Time: 21:12) the stress in one layer, but you can also calculate that what would be total radius and how you relate it to the stresses in different layers. So, this is about the science behind the technology, but in practice you would use an optical method to measure the bending; the radius.

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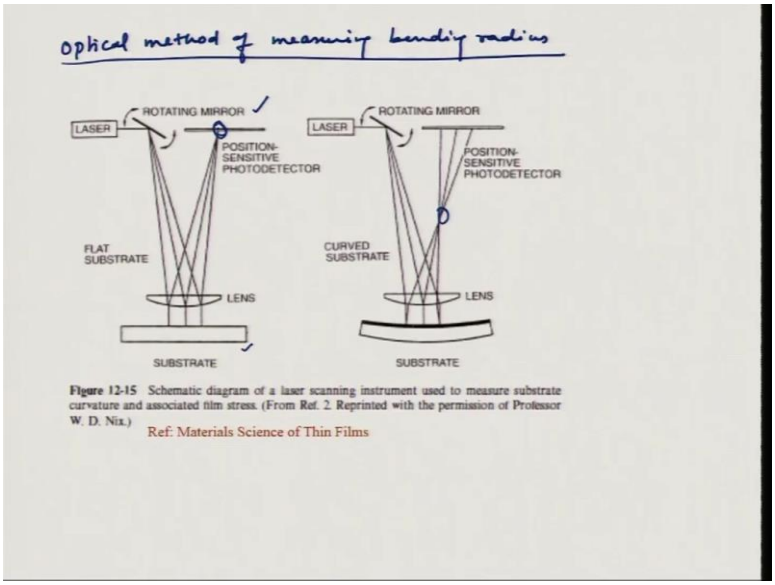

So, this is optical method of measuring bending radius and it is very difficult otherwise because you will not be able to see this bending by naked eye. So, what you use is that you use a laser and you have to first use the flat surface and you need to know the stresses of the substrate before you deposit your thin film. So, you use the substrate and calculate if there is any bending in the substrate. So, you focus a laser use using a lens and then you mark its position, if it is perfectly flat you will find one position; fixed position which will be same as this laser and by rotating this mirror, you can scan the surface of the substrate.

So, usually you would record this position of the laser by positioning your laser at two or three points or maybe four points at the edges of your substrate. So, you would record and if these position change with your deflection in rotating mirror which would be the indication of any banding in the surface. If the substrate is perfectly flat, optically flat then we will not see any change in the position.

Now, you take this substrate and deposit your thin film by PVD, CVD or any other method and then you repeat the measurement on the same substrate. Now because of the stresses in the film, you will have very small amount of bending because of the stresses. Now these stresses we cannot see, but this laser focus will be able to pick that small bending to the film stresses and you will see their change in position. Now knowing the prior positions and at the same points and the new positions at the same points, you can

calculate what is the bending radius of this and once you had the bending radius, you can go back to the Stoney's formula and use it to calculate the stresses in the film.

This is particularly important because sometimes, once you deposit the film and it has high amount of stress, overtime it will start to peel off; it will crack if the film is under too much of stress. As we have seen that for epitaxial films these stresses or strain in the film are relaxed by formation of islands which if you want to avoid we need to minimize the thin film stresses also as well, so this is a method of measuring thin film stress.

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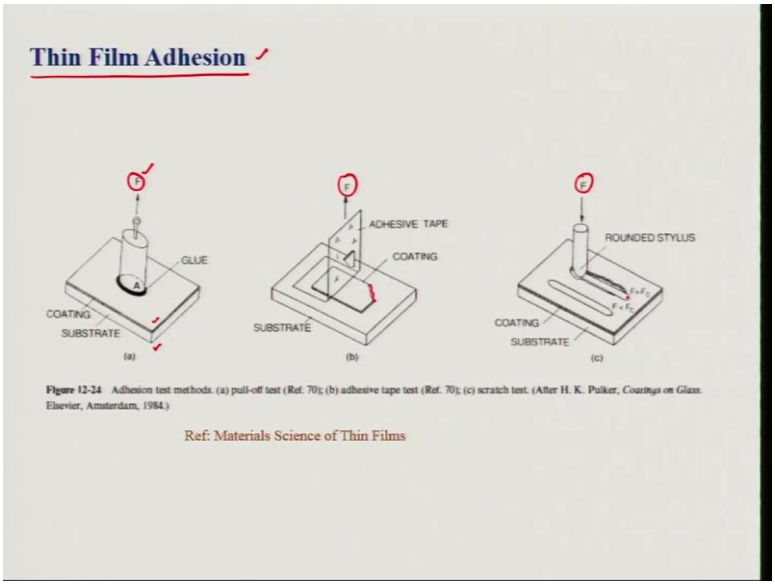

Now, last characterization that you want to discuss is thin film adhesion, how strongly our thin film is bonded to our substrate. Now there are no standard techniques for this, you have to go by your experience in this and try different techniques. If you can do of visual inspection, if film is not very good does not have very good adhesion, it will start to peel off like the paint peels off from metallic or very smooth surfaces. There are some quantitative measurements if you want to quantify the adhesion what you can do, you can on your substrate and the thin film you can put a glue and then attach something and then pull, so the force that you need to apply to peel off this coating. So, the glue is stuck to the coating so which is very strong adhesion, but if the adhesion between coating and substrate is not very good then the force that you need to apply to remove this coating or the thin film from a substrate will be quantified measurement of how good is the adhesion between the thin film and the substrate.

Another technique that you can use and it is very often perform just to test how good the thin film adhesion is called scotch tape test or adhesive tape test, what you do is you take a scotch tape and you apply on top of a thin film and peel it off, peel the scotch tape of and the force you need to peel the film off and if the coating comes off with the adhesive tape, then you will know that how good their adhesion between thin film and the substrates.

Another technique that you can use is by wear, you can have a rounded stylus and you can make a scratch on your thin film, the higher the adhesion the more force you would required to make that scratch, to remove that film and using that force as a measurement, as a quantified measurement of adhesion you can also measure how good is the adhesion. Now we need very good adhesion between the thin film and substrate we do not want it peeling off or crumpling during application.

There are several ways of enhancing this adhesion; one of them is you make the surface rough, you have inter mixing of the layers at the interface of thin film and substrate if you enhance the inter locking if items or the interfacial layers then you will get more adhesion. So, this can be promoted by either chemical bonding or ion bombardment during physical vapor deposition. So, there is a inter mixing of the layers at the interface, but sometimes this inter mixing will lead to not very sharp interface. So, for the application where you need very sharp or atomic layer sharpness at the interface, these techniques cannot be used then you have to rely on the surface energies and other parameters to design your thin film and substrate adhesion.

So, with this we conclude our thin film characterization part. Of course, there are many other characterizations you would need to use XRD, the scanning electron microscopy TEM or various other techniques to know much more about your thin films and also their functionality, if we are using it for optical applications or electronic applications you need to do those characterizations as well.

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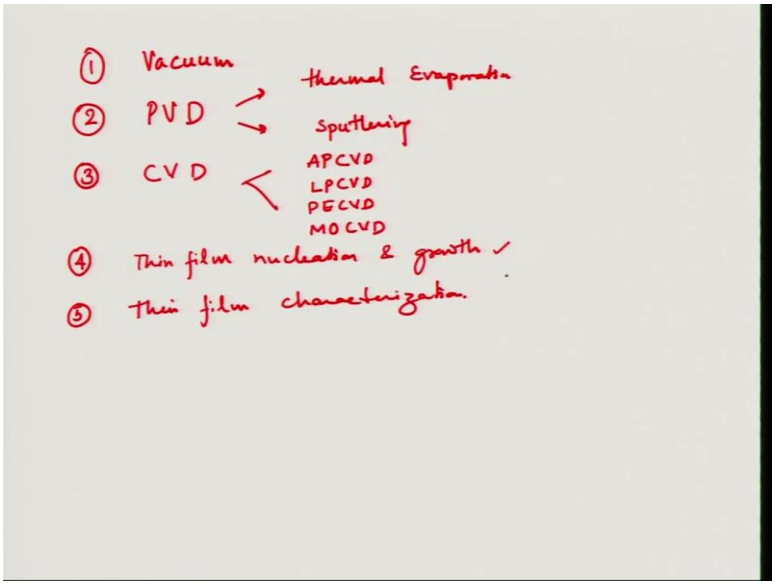

With this we conclude the course on the module on thin film deposition, in brief I will Point out the major thing that we have discussed in this course. First we discussed the use of vacuum, why is it important for thin film deposition, second we discussed physical vapor deposition techniques. In this we discussed thermal evaporation and sputtering; these two broad range of techniques. Then we discussed chemical vapor deposition in which we discussed many techniques, atmospheric pressure CVD, LPCVD, PECVD, MOCVD and many other techniques. We also discussed thin film nucleation and growth, different models and different parameters which determine how your thin film will nucleate on the surface and grow what would be the morphology and how do you control these and also we discussed some thin film characterization techniques.

So, I think at the end of this module it is a good point to go back and revise all the lectures. So, that you can understand why we were doing and what kind of techniques we can use for our film and what kind of morphology we can expect and then what kind of thin film thickness is another characterization you would need to perform on your thin films.

Thank you very much.