Non - Metallic Materials
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Module - 10
Measurement of the mechanical electrical, thermal, magnetic and optical properties of non - metallic materials
Lecture - 53
Measurement of optical properties

Welcome to my course Non-Metallic Materials and today we are in module number 10, Measurement of the mechanical, electrical, thermal, magnetic and optical properties of non - metallic materials and this is lecture number 53 where I will be describing the Measurement of optical properties.

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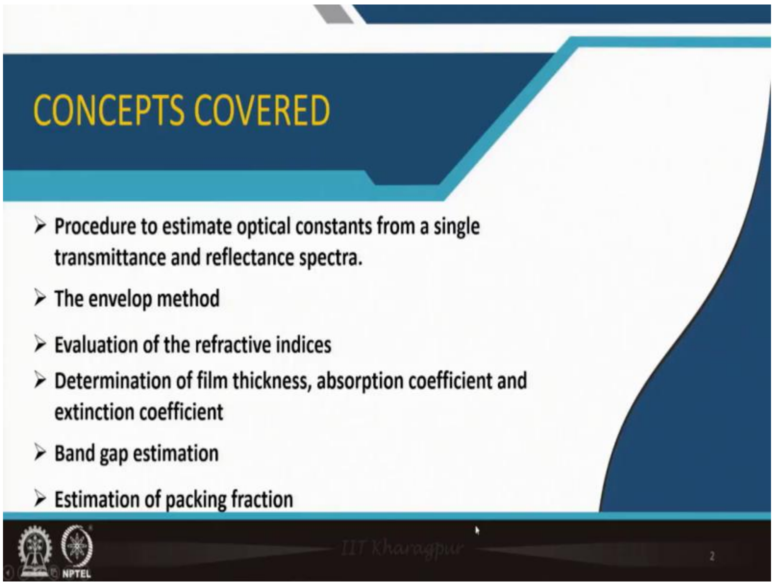

You know in my earlier lectures we talked about the basic optical properties of nonmetallic materials. In a subsequent lecture I also introduced the UV visible spectrophotometer and the principle of UV visible spectroscopy. Now, utilizing the data that you get out of this spectrometer, you can basically estimate a variety of optical constant from a single set of data, single set of transmittance and reflectance spectra..

And the process that I will be describing today is known as envelope method.

And this is a bit empirical in nature, but you will find it useful to determine the refractive index of a transparent thin film as a function of the values of lambda in the visible range. Then we can determine the film thickness, absorption coefficient and also extinction coefficient out of this single set data.

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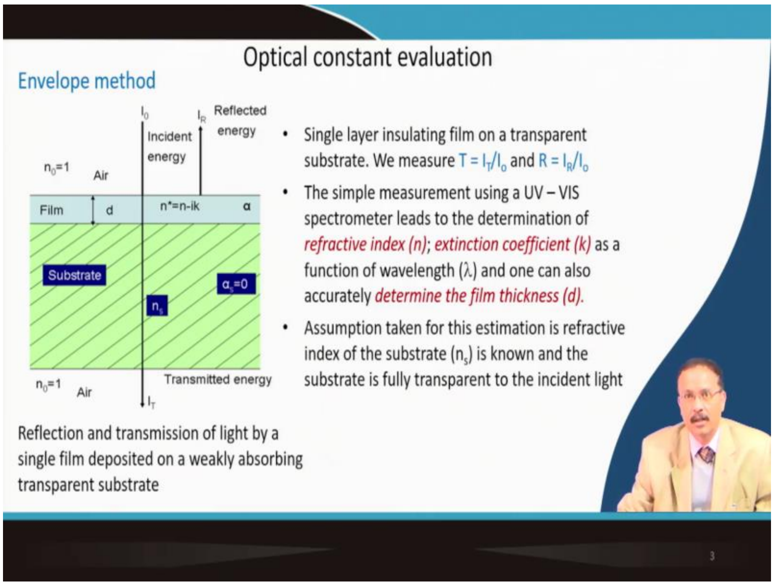

This data is also useful to estimate the band gap of the transparent dielectric thin film and finally, some idea about the microstructure in terms of the porosity inside the film can also be estimated by this powerful tool. Now, if you consider a substrate which is transparent in nature.

And on top of it a single layer of insulating film is deposited by the techniques that I have already introduced. You can use pulse laser deposition, you can use chemical vapor deposition, sol gel to make this kind of dielectric film coated on a transparent substrate..

And eventually using a spectrometer you measure the transmittance and the reflectance, the spectra you record in the spectrometer. And by a single set of data of this measurement whatever you have done you can estimate the refractive index, extinction coefficient as a function of lambda.

You can also estimate very precisely the film thickness. And in order to do this you will have to assume that refractive index of the substrate is known, the substrate that you are using you know the refractive index and it variation with optical range lambda and also the substrate is fully transparent to the incident light.

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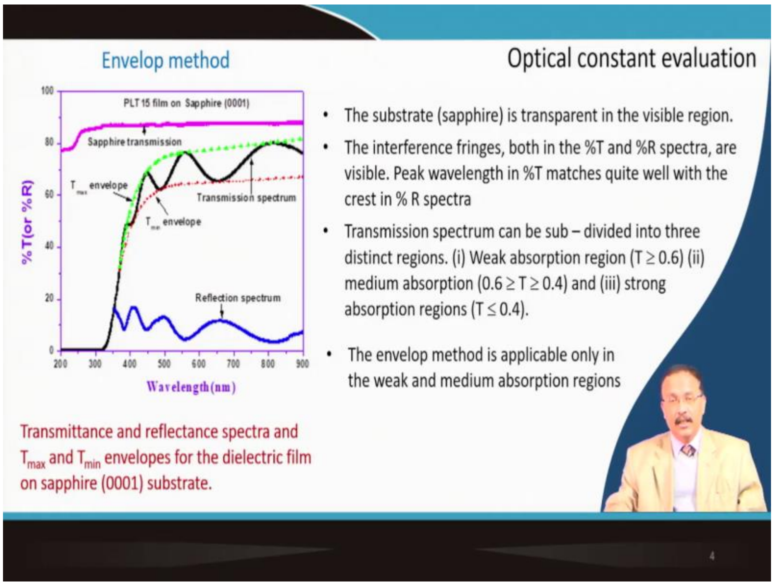

So, if this configuration is satisfied then one can estimate a variety of optical constant using this envelop method. So, the typical spectra that usually you get is something similar to this whatever has been shown. So, the substrate is fully transparent in the optical wave range, which is basically from 400 to about 700 nanometer and the substrate is transparent in the visible region as you can see almost 90 percent transmittance one can achieve.

And you get the interference fringes both in case of the transmittance spectra or percent transmission as well as the reflected spectra. And one difference you can see that the peak of this percent T matches with the crest of the percent R and this transmission spectrum you can subdivide it into three distinct region.

First one is a weak absorption region where the transmittance is more than 60 percent then you have a medium transmittance region where the transmittance is in between 40 percent to 60 percent and then there is a strong absorption region, where the transmittance is typically less than 40 percent or lower.

So, the envelop method that I am going to describe this is applicable only in the weak and medium absorption region. So, in this case I am assuming that I have deposited a dielectric film on a transparent sapphire substrate and sapphire is single crystalline in nature and the orientation is along with its basal plane that is 0001, it is a hexagonal system.

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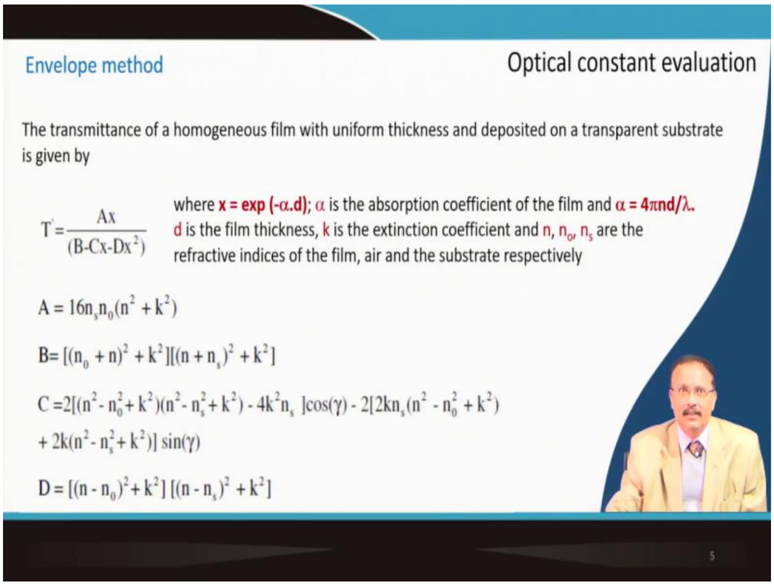

So, four indices index is required for this. So, if I assume that it is a homogeneous film and thickness is uniform. So, all this thin film deposition technique will lead you to a uniform film thickness and it is deposited on a single crystalline transparent sapphire substrate.

Then the transmittance that is given by this empirical relation and you will be noting that I will be using lot of empirical relations and assumption that will reduce this empirical relations into a analytical form. So, eventually you can do the necessary calculations repeatedly using a software like Excel or Origin or any other software of this type which can do the calculation part and you can extract the optical constant data. So, this will be illustrated in this lecture.

And if required I can give you the transmittance and reflectance as a function of lambda data which actually we get from a UV visible spectrophotometer. For you to independently do all this series of calculations and the results that I will be showing in this lecture, you can exactly repeat it to see how it works to have a fill how it works. Otherwise, just looking at this equations it may not be apparent that how things are actually being calculated.

So, it is required for you to do it yourself so that you can have a fill and this process is quite generic in nature. It is applicable for any transparent film and it is a powerful tool and I will show you at the end that people have taken advantage of this and they have come up with a commercial equipment, where all these equations are fed already fed and you just press a button and you get the data estimated or calculated for you in a very convenient way.

So, how is it done, that I will explain in this lecture. So, this transmittance that is given by this simple relation where x is defined as you know that film thickness is involved and the absorption coefficient is involved.

And the absorption coefficient is again related to the refractive index in with the film thickness as well as the wavelength in whatever you are considering. So, this A, that is given by this relation which uses the refractive index of substrate refractive index of air this medium I have considered is air.

So, n 0 is practically 1 and n and k are the refractive index of the sample and the extinction coefficient of the sample that we are studying. Similarly, you have an empirical relation for B and you have the empirical relation for C as well as D given by this equations and this is given in the reference I have cited one book and this equations are illustrated in that particular book chapter. Now, in order to do the so called envelope method,

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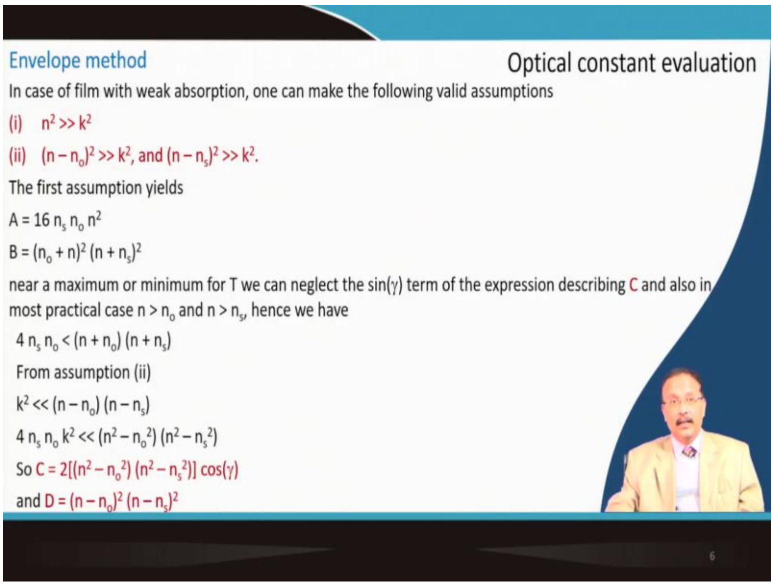

We will have to make certain assumption ah. So, first assumption that I will make is the film is weakly absorbing. So, either it transmit the light radiation, part of it is getting reflected as you can see in the view graph that I have already shown and it is homogeneous in nature and quite transparent. So, scattering is marginal or almost nil.

So, light is not scattered and absorption is also considered to be quite low in the visible region. But, as you know if you change the wavelength down to a region where it is very close to its band gap then of course, absorption will take place and I have already talked about it when I was talking about the optical properties of material you know the electron transition takes place. It goes from valence band to a conduction band higher level crossing the band gap and this is applicable here also.

So, 1st we consider that this value of n this is much larger than the extinction coefficient and 2nd is we are considering air as a medium. So, n minus n square that is also much larger than the extinction coefficient square. It is a small number and you are squaring it. So, the value will be really very small.

And the same thing applies for the substrate and the film this difference in in in the refractive index that square is also much much larger than the extinction coefficient. So, if you apply this assumption then you are a, the A constant which I just described that is reduced to this and B is reduced to this..

So, near a maxima or minima the gamma term we can neglect and also in the most practical case the refractive index is larger than the air refractive index which is 1 and it is also larger than the substrate in question the sapphire we have used. So, we can have this assumption if you fulfill then this is valid, this expression is valid and if you take the assumption 2 then this equations are also valid. You can work it on and taking this simplified assumption which is practically possible.

You can derive the constant C in a much smaller less complicated term as well as the constant D whatever I described in my earlier slide. So, this value will be reduced to the red marked equation as compared to relatively larger equation that we had in the earlier slide.

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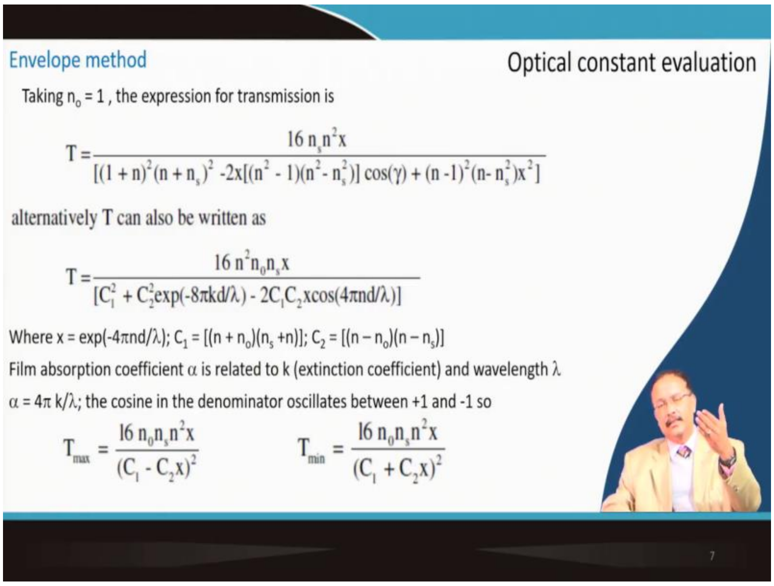

Now, we can safely put that air is from air the light is incident on the thin film. So, the value of n is equal to 1 and then we can have the transmittance expression the percent transmission by this big relation. And also by putting I mean just simplifying this you will eventually come up with this relation, where this x part is given by this relation C_1 and C_2 is dependent on the refractive index of air substrate as well as the thin film.

And film absorption coefficient is related to the extinction coefficient and wavelength by this relation.

So, the cosine part of this denominator that will oscillate between plus 1 and minus 1 giving the T max value, which already I showed in the in the view graph that the interference fringe the T max and T min you can have an analytical expression involving the refractive index the value of x and the constant C_1 and C_2 has been derived. So, you can also derive this relation based on this assumption and work it out.

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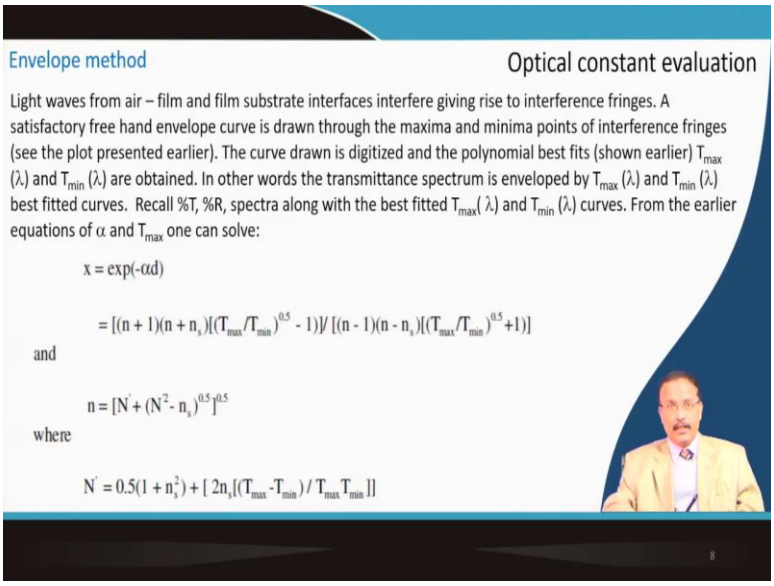

Now, what is happening? That light wave from air film and the film substrate. This interface they are interfering and giving rise to the interference fringe that you know that the optical path difference that will be either an even multiple or odd multiple of lambda. Depending on that you will get maxima and minima from the basic fundamentals of the interference pattern. So, then we will draw satisfactorily a line which covers through the maxima as well as the minima point.

That already I have shown if you remember the first slide that it is a hand drawn hand drawn kind of envelope which is covering the maxima and minima, just look at the curve.

And this drawn curve is digitized and then you fit it with a polynomial. So, a polynomial fit can be made out of those point which is covering the maxima and covering the minima forming this envelope. So, the equation that I had derived this x was given by this and this in turn is also related to the refractive index.

So, this T max and T min is included in this relation, where the refractive index that is given by this simple relation N prime and it is related to substrate refractive index because n 0 I have put already 1 and this N prime that is defined by this relation. So, you can work it out the value of n how exactly it is varying with the wavelength.

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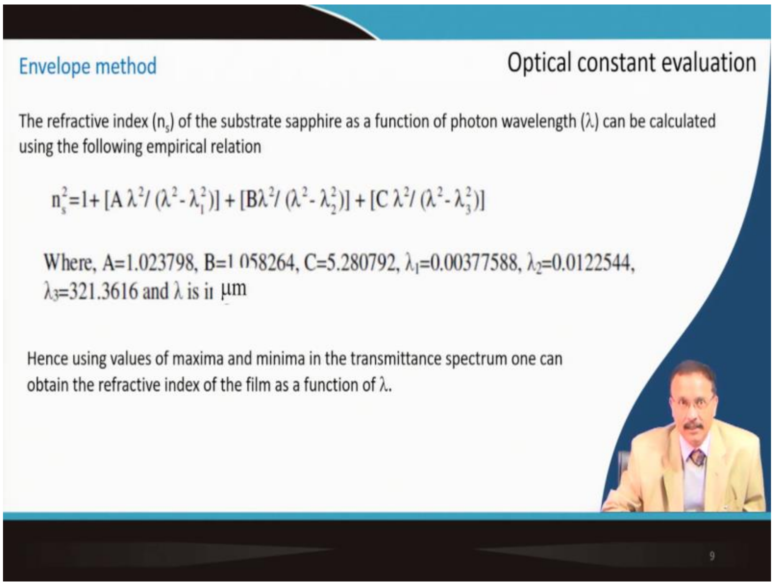

So, the refractive index of the substrate as a function of lambda that also is needed for this calculation and the empirical relation for the substrate which is sapphire is this. So, you can see the substrate refractive index that is a function of lambda and all these constants they are fixed for sapphire.

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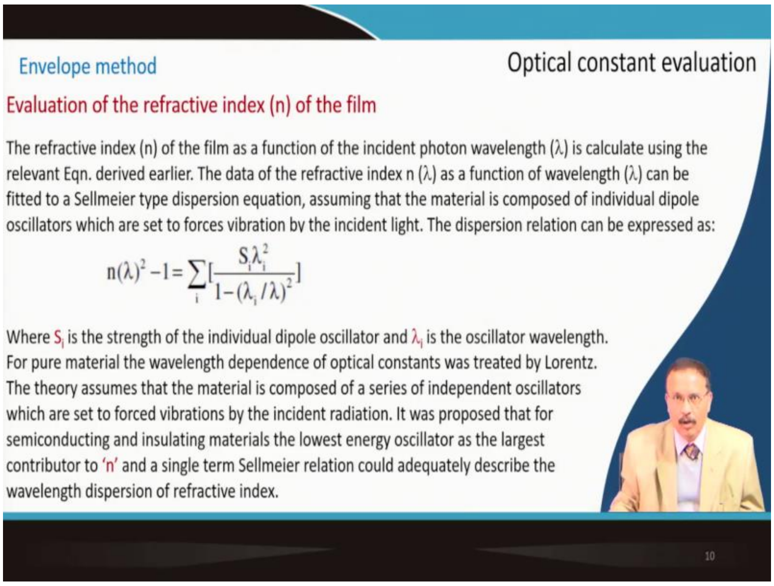

So, the relation of n s is also known as a function of various lambda in question. Now, you can estimate the variation of the refractive index as a function of the wavelength in a series of wavelength in the optical region, where the transmittance is either in the middle range or in a high transmission range. It is not applicable to the absorbing range.

So, you can calculate the value of n from this empirical relation as a function of lambda. So, the refractive index of the film as a function of the incident photon wavelength is calculated using the relevant relation that already I have shown. So, this data can be fitted to a dispersion relation, which is basically worked out by Sellmeier and he assumed that the material is composed of individual dipole because it is basically a dielectric material that we are considering and which can oscillate ah.

So, when the light waves come, you know that this is a electromagnetic wave. So, this oscillator is set as a forced vibration. So, the dispersion relation of this n refractive index as a function of lambda that is given by this equation, where you can see it is a summation.

So, if there are many individual dipole number of dipoles present with each of having a separate wavelength then this is just a summation of all the dipoles. Now, if you are considering a pure material the wavelength dependence of the optical constants that basically was treated by Lorentz.

And this according to the theory developed by him he assumes that the material is composed of a series of independent oscillators which are all forced to vibrate by the incident radiation. So, for semiconducting or insulating material the lowest energy oscillator is the largest contributor to n.

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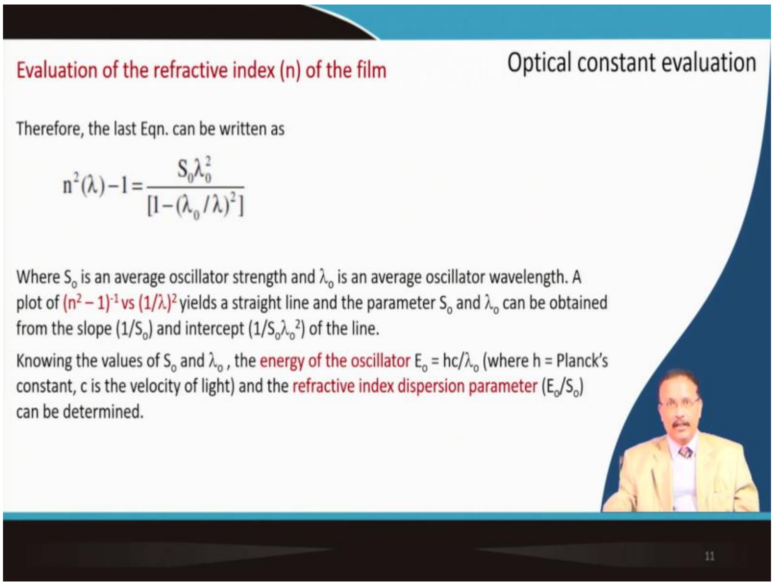

So, a single term of Sellmeier relation is possible and that could describe the wavelength dispersion of the refractive index. So, if you consider only one oscillator then this relation is valid. So, here S 0 is the average oscillator strength and lambda 0 is average oscillator wavelength. So, if you plot this n square minus 1 inverse versus 1 by lambda square that should give a straight line and from this straight line this is E 0 and lambda 0 of that oscillator you can obtain.

So, the slope is 1 by S 0 and the intercept is 1 by S 0 into lambda square from the straight line. So, if you know the value of this S 0 and lambda 0, the energy of the oscillator you can calculate. You know that energy is hc by lambda and c is the velocity of the light and refractive index dispersion parameter, so, variation of n versus lambda that is given by E 0 by S 0 that also can be determined from your n versus lambda plot following the Sellmeier types of dispersion.

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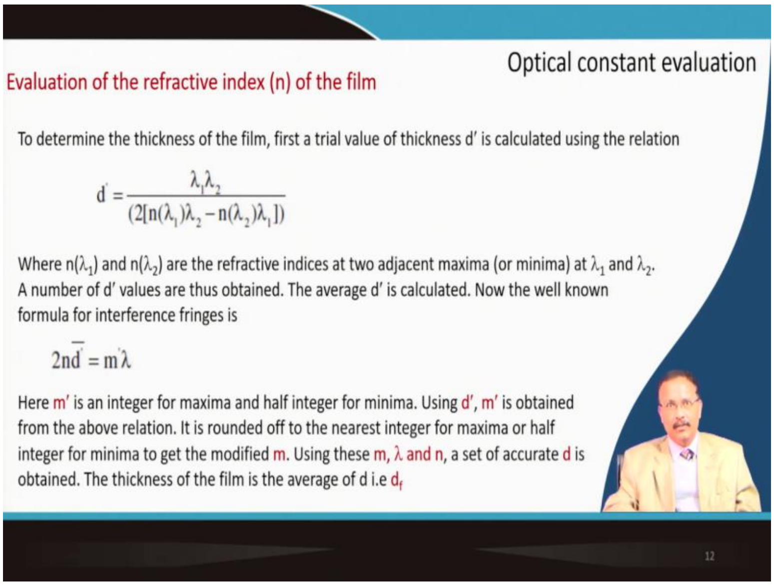

Next we can also determine the thickness of the thin film if you know the refractive index of the film. So, the relation is something similar to this, where you can know the 2 refractive index of either adjacent maxima corresponding n or adjacent minima the corresponding n.

So, you can get basically a number of d prime value and average of this d prime is first calculated. Now, you know that the interference fringe is actually related by this relation. So, it is refractive index that is the average of this d whatever you have calculated that is basically a multiple of lambda ah. So, this m prime is an integer for maxima and a half integer for minima. So, we will use this d prime and m prime we will obtain from this relation.

So, the value of m prime is rounded to the nearest integer for maxima or half integer for minima and we will get a modified m value. So, this modified m, lambda and n, using this a set of accurate d value is obtained and then we will average that d value to get the precise thickness of the film that you are considering.

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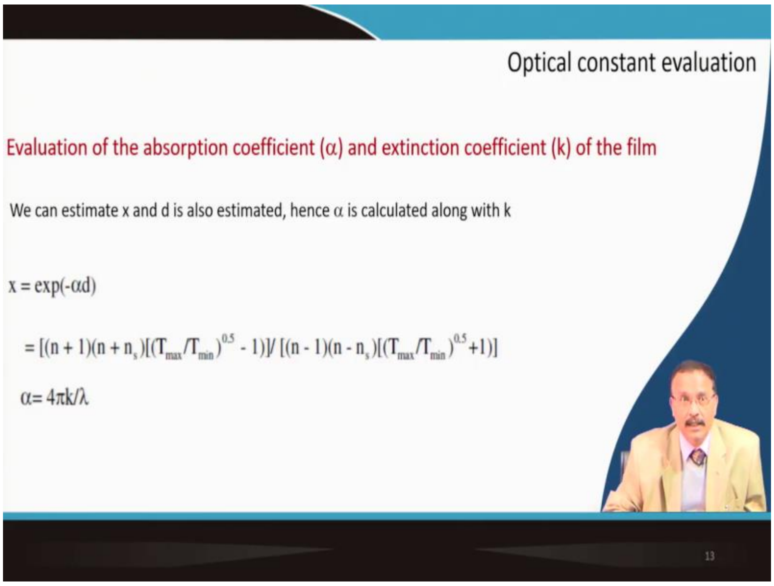

So, absorption coefficient and extinction coefficient also can be evaluated or estimated by this relation. So, these two relations are given. So, that will help you to get the value of alpha and knowing the lambda you can also get the value of extinction coefficient from this two simple relation.

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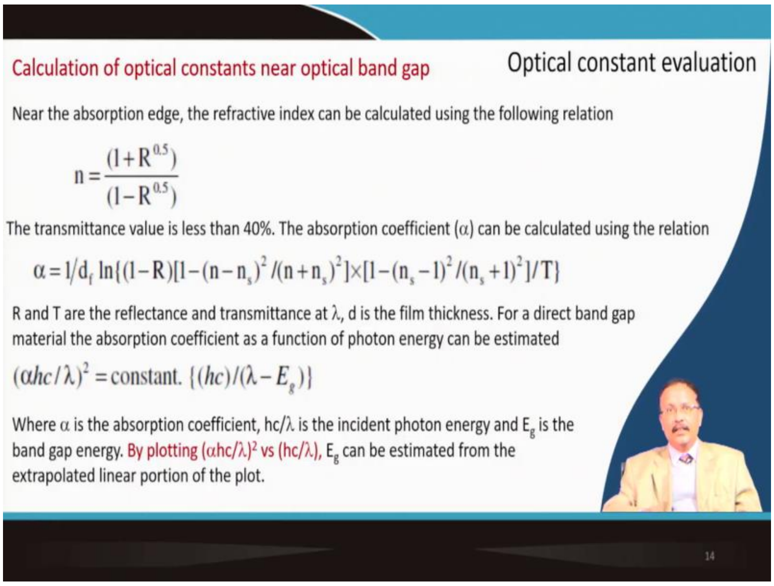

Now, when the absorption takes place, so, near the absorption edge the refractive index is estimated by this relation. Actually, this relation I also described in one of my earlier lectures. So, here the transmittance value is less than 40 percent and the value of alpha can be calculated by this relation.

So, here R and T they are the reflectance and transmittance at each lambda, d is the film thickness and you know that if it is a direct band gap material the absorption coefficient as a function of photon energy you can estimate by this relation. And you know alpha, h is known, c is known lambda is varying ah.

So, you can calculate the band gap by plotting this alpha hc by lambda whole square versus hc by lambda. So, E g the band gap of the film can also be estimated from the extrapolated linear portion of the plot.

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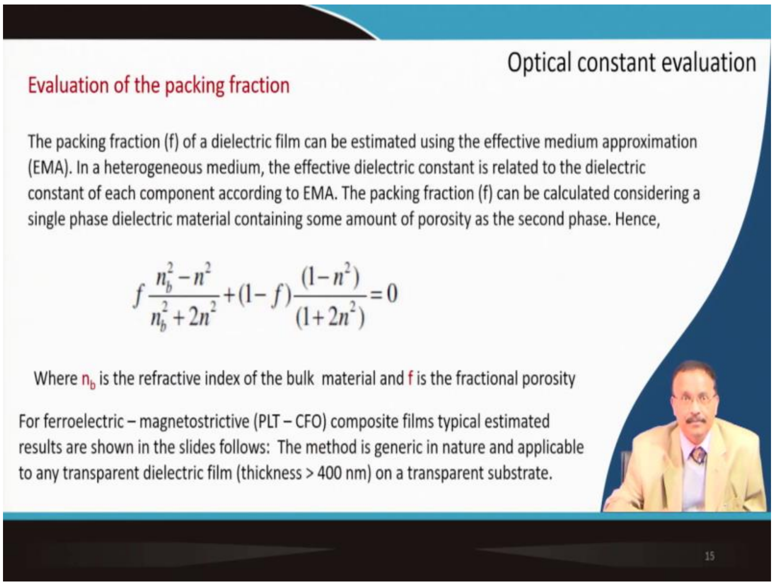

Finally, the packing fraction that means, that how much porosity is there inside the film that also can be estimated by this simple relation and here we have assumed a effective media approximation. So, in a heterogeneous medium the effective dielectric constant is related to the dielectric constant of each component according to this EMA and the packing fraction can be estimated. So, here you know the bulk refractive index of the film material, n already you have calculated.

So, you can have you can calculate the fraction of f from this relation. So, this basics we have used to calculate this optical constant for a complicated film that was deposited and it is a ferroelectric and magnetostrictive kind of composite film for a particular application.

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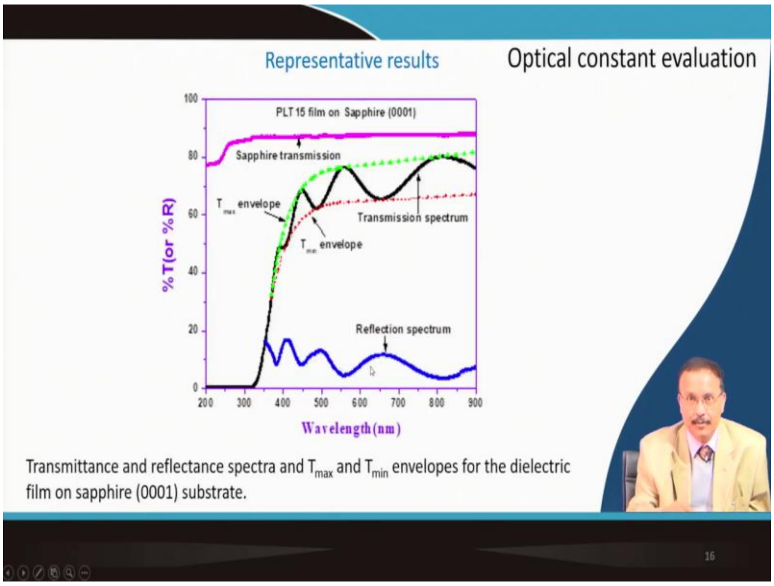

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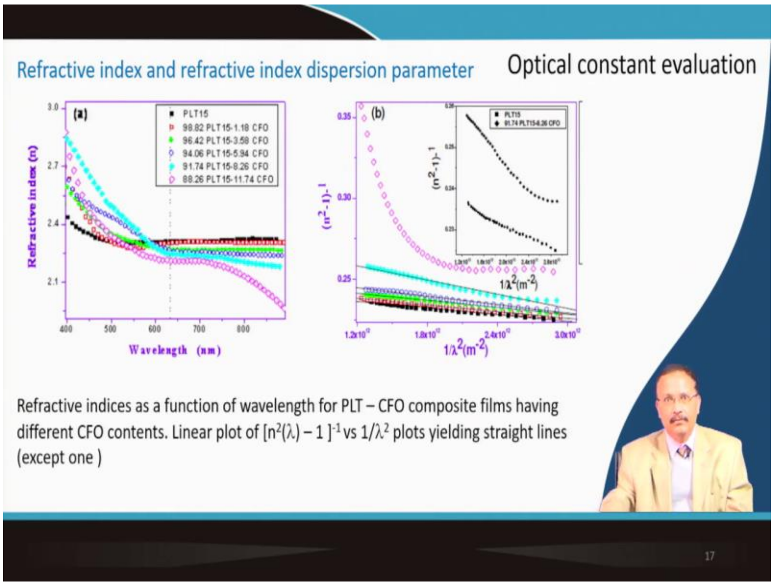

So, we got the transmittance pattern like this and then we made this envelope as already I have described. And then we estimated the refractive index as a function of wavelength. You can see for a variety of composition how the value of n is changing. So, then using the Sellmeier kind of dispersion relation we plot it.

And you can see for some of the composition we get in indeed a linear plot to estimate the refractive index dispersion and the other parameters which I talked about, but for certain other composition because it is a complicated dielectric film, it does not match. So, it is having its own reason, but that is beyond the scope of this lecture.

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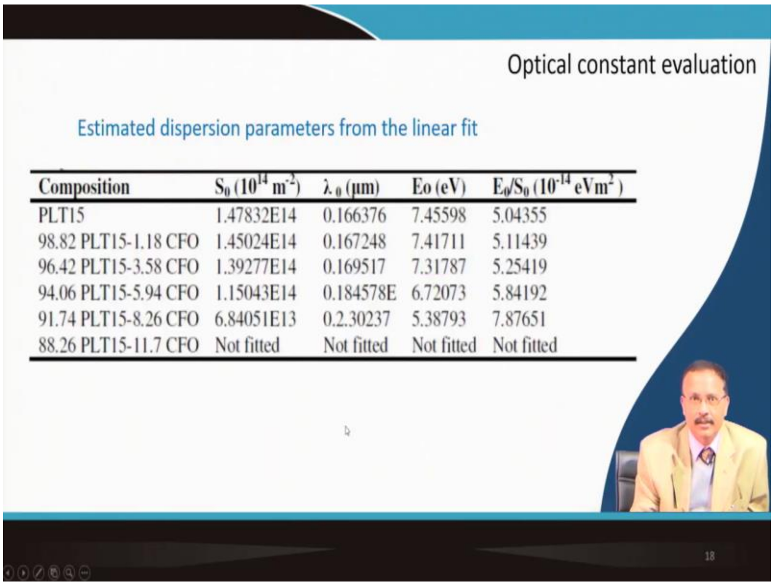

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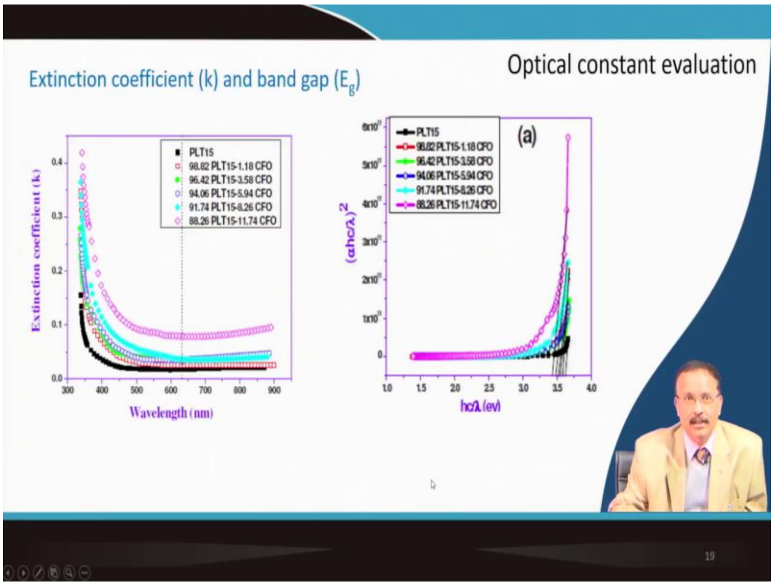

And we can estimate all this parameter S 0, lambda 0, E 0, E 0 by S 0, where it is fitted linearly, where it is not fitted you cannot do this. Then we calculated the extinction coefficient as a function of wavelength as well and in the absorption edge we also calculate the band gap. You can see the band gap of this composite film that also is insulator for a pure film, but as you keep on increasing the magnetostrictive component which is CFO then the band gap is progressively reduced.

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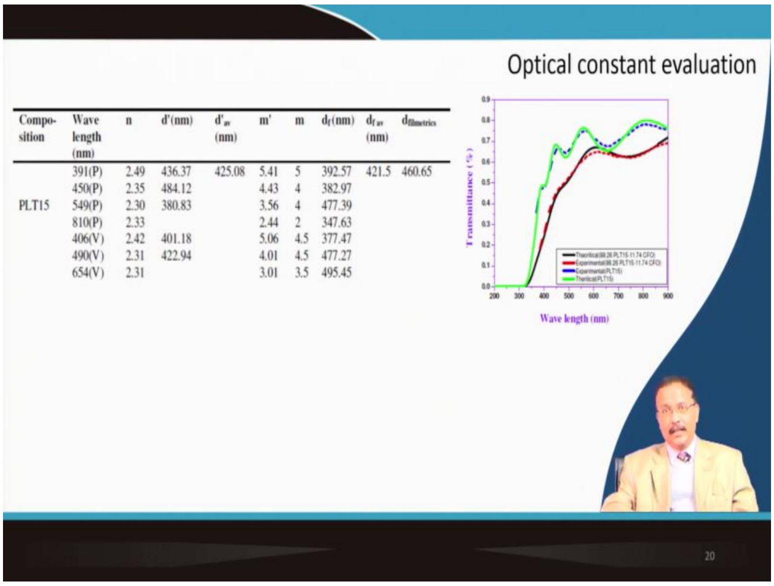

This is the example of the calculation of thickness which I was talking about. So, first you calculate the d prime, then you make it an average, then you have the m prime value and nearest integer or half integer you take and then you calculate the value of d r which is varying little bit. So, you average it out and this you get around 421.5.

And using a commercial equipment which also apply the same principle we got it is about 460. So, there is slight mismatch, but still it is a close. Now, all these parameters this n value, k value etcetera you can put in the transmittance equation and you can reconstruct the experimental plot.

So, as you can see the experimental and the reconstructed plot they are exactly identical for different composition of the thin film. So, that tells that our estimation by this envelope method to calculate the value of n, k etcetera to construct the transmittance and also the reflectance spectra that is quite accurate.

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And this phenomena is quite interesting. So, commercially people have made this kind of equipment where they first calculate sorry measured the reflectance and transmittance and then using all this equation whatever I described in the earlier I mean in the lecture.

So, they can calculate this and what you get is a through the software you just put the sample it measure the transmittance and reflectance spectra do the internal calculation to get you the value of n, value of k, E g extinction coefficient and accurate film thickness. So, this is already been commercialized. So, it is a good thing for you to do it yourself.

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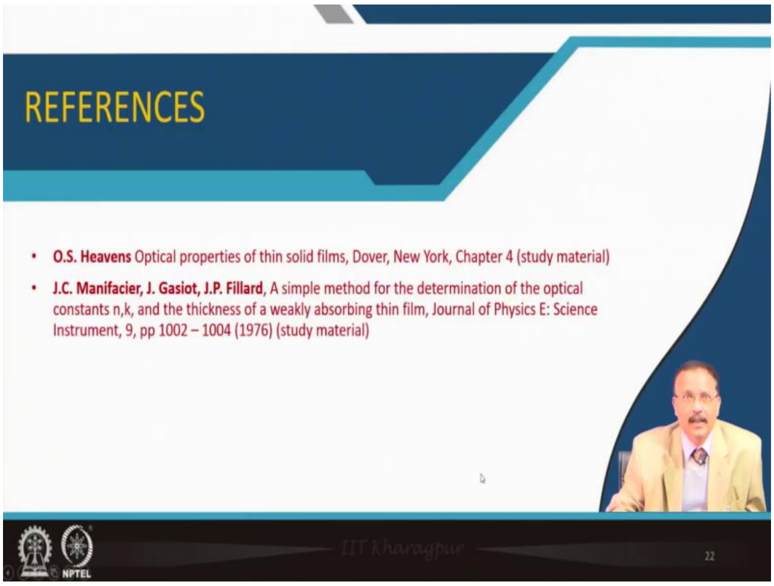

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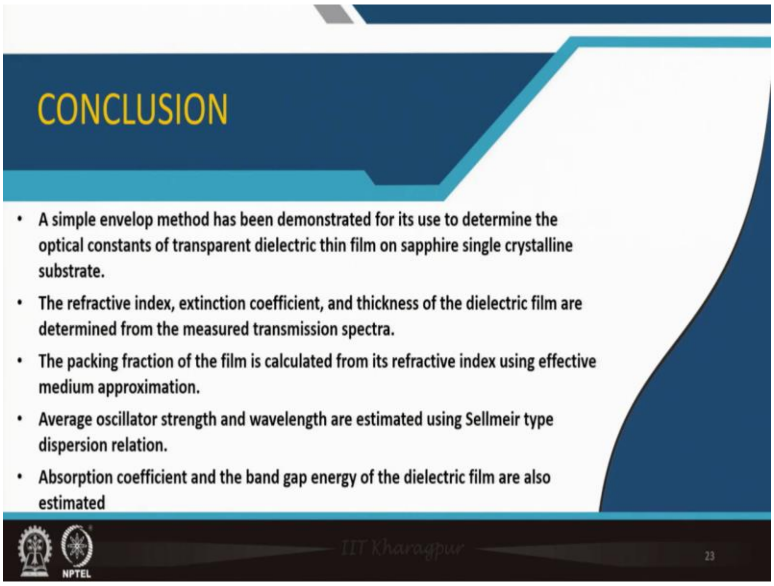

So, there are 2 good reference ah. One is a book by Heavens and the envelop method is described in this research paper. So, you can just use it as a reference. So, I have described a simple envelop method to and it is used for determining the optical constant. The refractive index, extinction coefficient, thickness of the dielectric film are determined from the measured transmission spectra. Packing fraction of the material the thin film can also be evaluated.

Average oscillator strength and wavelength are estimated using a Sellmeier type dispersion relation. And absorption coefficient in the band gap of the dielectric film are also estimated.

Thank you for your attention.