Indian Institute of Technology
Kanpur

NP-TEL
National Programme
on
Technology Enhance Learning

Course Title
Advanced Characterization Techniques

Lecture-30

by…
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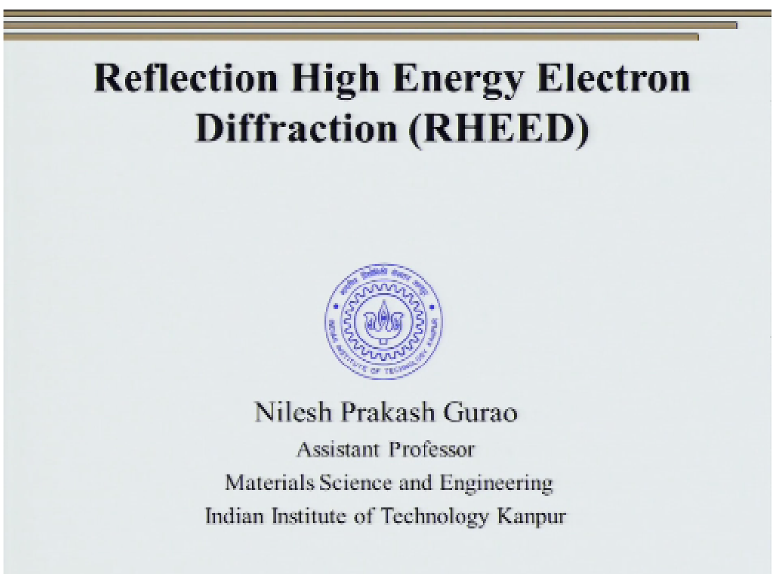

Hello everyone.

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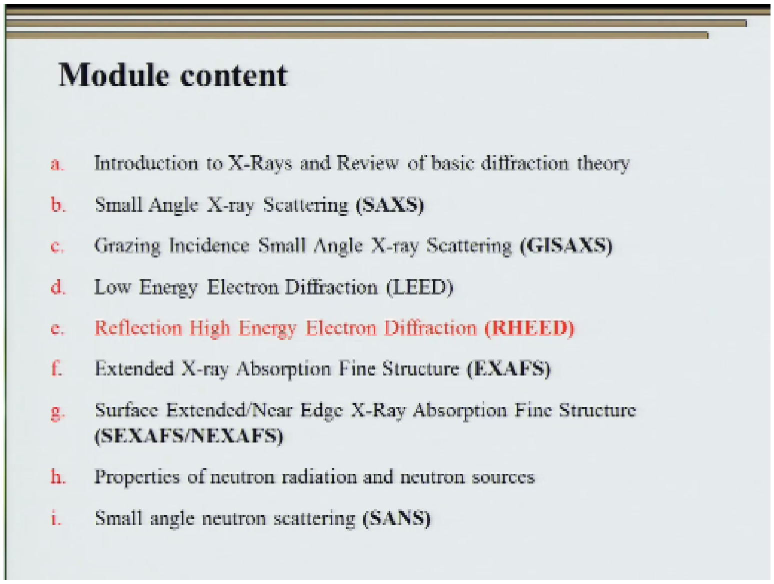

In today's class on advanced characterization techniques, we are going to touch upon the reflection high energy electron diffraction. In the last class we had studied, how does low energy electron diffraction works. I would like to mention that reflection high energy electron diffraction is assisted technique of low energy electron diffraction and it shares a lot of principles in common with low energy diffraction.

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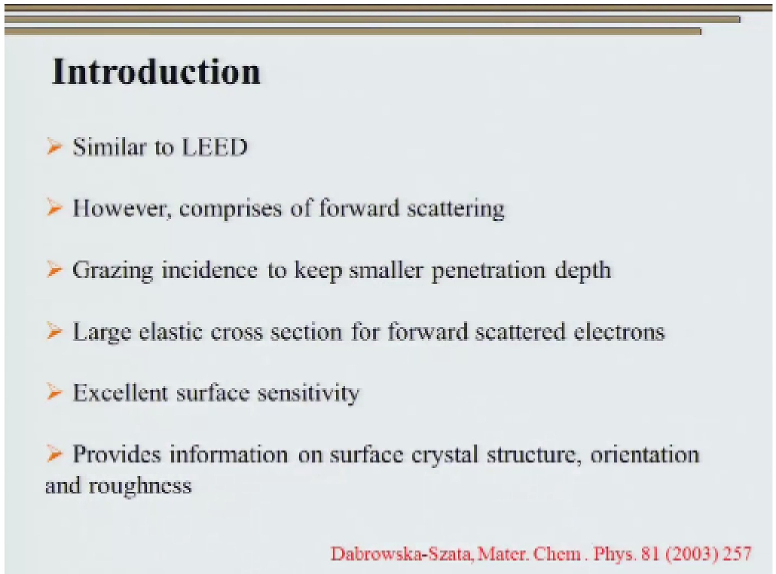

So as I had already mentioned, reflection high energy electron diffraction is very similar to low energy electron diffraction. However, unlike low energy electron diffraction, which essentially comprised of back reflection. If you remember, we had a source coming over here for X-rays and the sample position over here and we had this entire grid along with our screen positions of where over here.

So essentially it comprised of back reflection. Unlike low energy electron diffraction, we are now going to talk about reflection, high energy electron diffraction wherein you talk more about or rather we talk only about forward scattered electrons. As I had already mentioned if at all the electrons are going to scatter in the forward direction, we have to ensure that the geometry of the electron beam that we are using is essentially gracing incidence geometry.

Now the geometry that we are going to talk in reflection high energy electron diffraction is very similar or rather same as that of what we used in gracing incidence small angle X-ray scattering. By now, I hope you must have developed an understanding about how we play with the geometry of say X-rays or electrons or for that matter say even neutrons which we are going to talk about in a couple of lectures from now on to get relevant information or to achieve relevant diffraction condition.

Talking about forward scattered electrons, the major advantage that it offers is that, it gives us a large elastic cross section, at the same time it gives us excellent surface sensitivity. That was precisely the reason why we had opted for gracing incidence small angle X-ray scattering over simple angle X-ray scattering. So I hope you understand the geometries. Now we are going to use the geometry of gracing incidence for our own advantage in reflection high energy electron diffraction.

Now as we had seen any technique that works with gracing incidence provides us important information about the surface crystal structure, the orientation, as well as the roughness of the surface as well as any adsorbate or any entity say thin film, or quantum dots which are made grown on the substrate.

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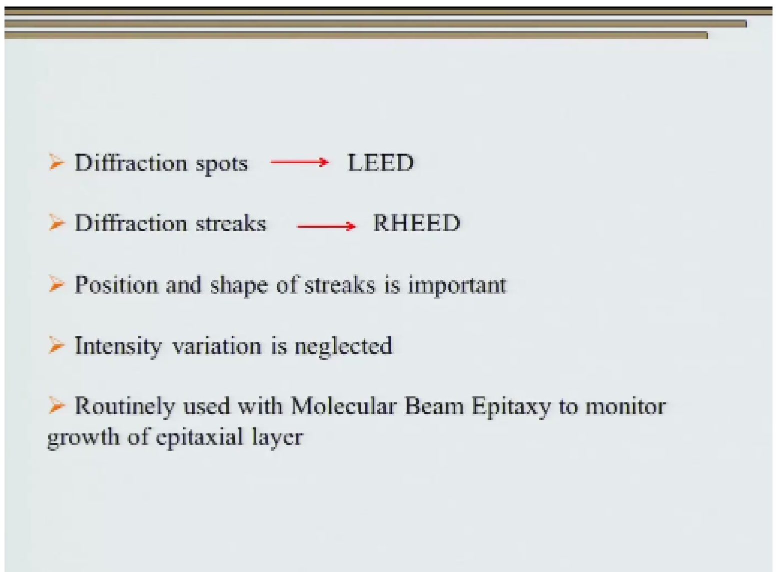

So we had seen earlier that you know the output of low energy electron diffraction was nice diffraction spots. We could actually envision the reciprocal lattice in low energy electron diffraction. Similarly in reflection high energy electron diffraction, we also see the reciprocal lattice. However I hope you appreciate that the diffraction condition is relaxed in a particular direction, and this precisely happens because the thickness of the film, the entity that we are talking about is very less.

Now once we go to the reciprocal space, the lesser distance in real space gets transformed into higher distance in the reciprocal space. And therefore, instead of getting diffraction spots, we end up getting diffraction streaks in reflection high energy electron diffraction. Now similar to low energy electron diffraction, there we had used the position of the spots. The position as well as the spacing of the spots to decipher the structure, we can use the position as well as the distribution of the streaks to find out the crystal structure of the entity under consideration.

At the same time I should mention that, the theory for reflection high energy electron diffraction, is not very well developed. Though I am going to show at the very end of this lecture, a couple of examples where we use, the theory and carry out simulations for reflection high energy electron diffraction on a routine basis, the theory is not as well developed as that for low energy electron diffraction, and therefore generally we do not account for the intensity variation that occurs in the spots obtained or rather streaks obtained in reflection high energy electron diffraction.

Another basic advantage that reflection high energy electron diffraction offers is that it can be coupled with any thin filmed growth technique for that matter which a technique like say molecular beam epitaxy, to monitor the growth of the epitaxial layer in C2. So wherein we can play with the proposition parallel parameters at the same time by seeing how the thin film is growing.

So this is one of the biggest advantage that reflection high energy electron diffraction offers in the processing as far as semi conductor industry is considered.

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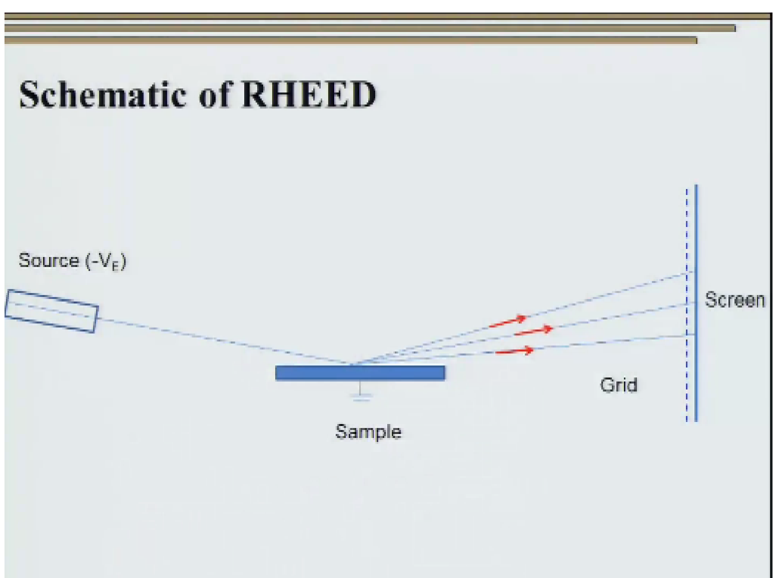

So let us look at the geometry of reflection high energy electron diffraction. As you can see over here, the source is shown over here and here is our sample. You can see that the electrons are incident at a very small angle. I would like to mention that this figure is a bit exaggerated and this angle what I am showing with the normal is very close to 90. Meaning this angle, the angle with the surface is very, very small.

So once the electrons which are incident on the sample get defracted out from the surface, we put a grid over here and give a negate to voltage, so that it repels all the in elastically scattered electrons, and only the elastically scattered electrons are able to travel and hit the screen and give rise to the diffraction pattern.

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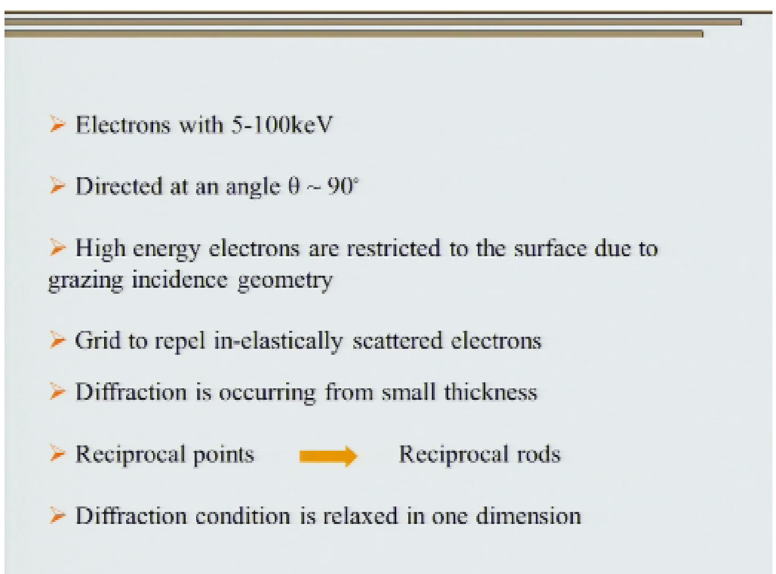

Another important thing that differentiates reflection high energy electron diffraction from low energy electron diffraction is the energy of the electrons. I hope you remember from our last class, that we had used the energy or electrons with energy of 20-500eV in case of low energy electron diffraction. However, when we go to reflection high energy electron diffraction, we use energies in the keV range.

So we start with energies of the order of 5keV to a 100keV. I would just like to draw your attention with the fact and particularly at the energy levels that we are talking about. If you recollect, we can use these energies in the keV regime in a simple scanning electron microscope. At the same time, the higher spectrum is for a very low kV transmission electron microscope. So I hope you appreciate that the essential difference between reflection high energy electron diffraction and low energy electron diffraction is not just the geometry, but also the energy of electron beam that we are using.

As we had seen the incident electrons are directed at an angle which is almost normal to the perpendicular to the surface. That is the electrons are incident at gracing angle. These high energy electrons are restricted to the surface due to this very gracing incidence geometry. Now as I had already mentioned, these electrons which are getting defracted or scattered or can undergo elastic as well as inelastic scattering.

So we do not like the inelastic scattering because they contribute to background and therefore, you put a grid unlike similar to what we had in low energy electron diffraction, give it a negate to by us so that we get rid of all the in elastically scattered electrons and the diffraction or the elastically scattered electrons that are able to reach the screen, if they contribute to diffraction which is occurring from a very small thickness of the order of few atomic layers from the surface.

So because of this relaxed diffraction condition that I had mentioned earlier, the reciprocal points are replaced with reciprocal rods or rel rods. This essentially happens due to relaxation of diffraction condition in one particular direction.

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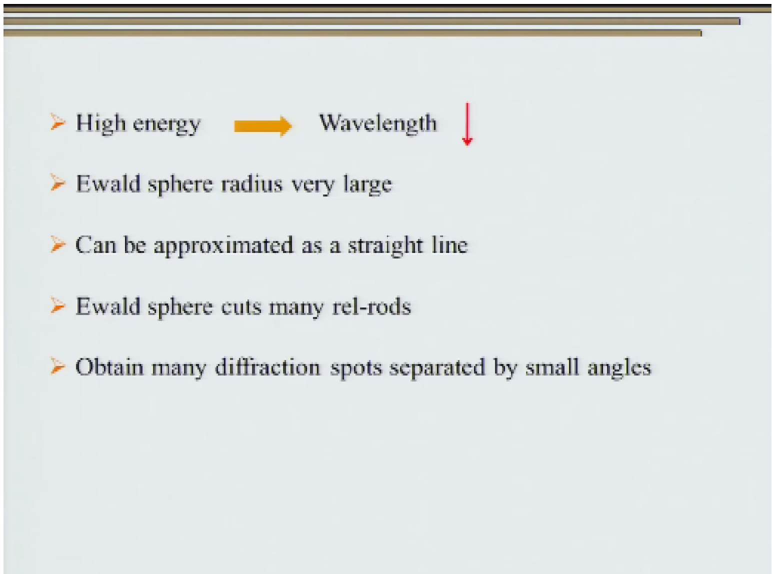

Now what are the consequences of the geometry we have already seen that you know everything is restricted only to a few atomic layers. However, another important difference that we mentioned was the tremendous difference in the energy levels. Now what does the energy level contribute to when we compare low energy electron diffraction and reflection high energy electron diffraction?

Well the high energy used in reflection high energy electron diffraction leads to a very small wavelength of the electrons. I hope you appreciate that electrons at such high energy can be easily considered as a wave. Now the basic importance or the basic consequence that it has is determining the ewald sphere radius. If you remember we had talked about this even in X-ray diffraction and during low energy electron diffraction that the radius of the ewald sphere is nothing but $1/\lambda$ or for that matter it is $2/\lambda$.

So you can imagine that if the wavelength is very small, the ewald sphere will be very, very large. Now you can also imagine that if the ewald sphere is very, very large, the circumferential part of the ewald sphere can be considered as good as a straight line and that is what essentially happens, say in a transmission electron microscope and similar things happen in reflection high energy electron diffraction.

Now what does this flat in nature or planar nature of the ewald sphere uses, well it leads to diffraction at multiple points. This is essentially because the diffraction condition is satisfied. We have more and more diffraction or reciprocal spots or for that matter in reflection high energy electron diffraction the rel-rods or reciprocal rods lie on your ewald sphere and this leads to many diffraction spots separated by small angles in case of reflection high energy electron diffraction.

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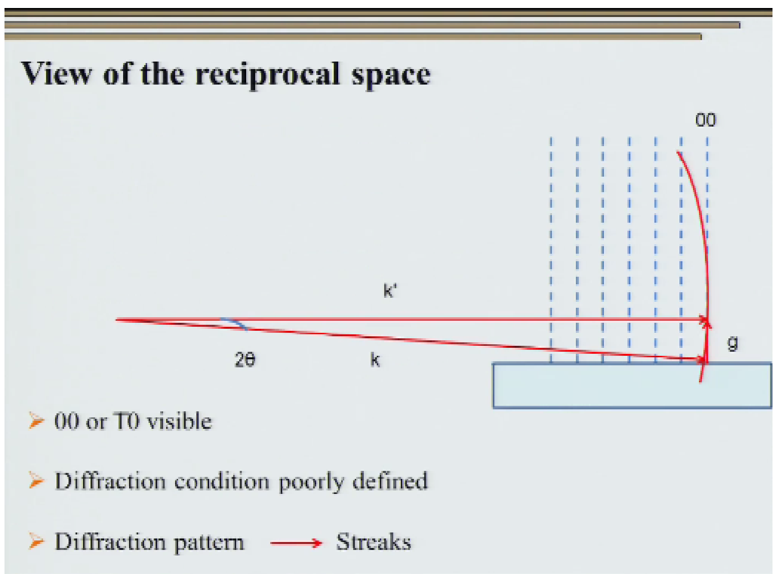

So the image showing the actual geometry of the process is shown over here. I hope you remember that this is what corresponds to our planar reciprocal space where you have the 00, bar 10, bar 20 or here and 10, 20, 30 and so on. So here as you can see the ewald sphere which is shown over here having wavelength of $2\varphi/\lambda$, we see that the Bragg's condition is satisfied not at a particular point but at multiple number of rel-rods.

Not only that, we get refraction condition not only for this 00 but also for the 1 bar 0 spot. We do not get either of them, but most of the time we do get diffraction from this 00 or 1 bar 0. Now as you can imagine from such a figure that the diffraction condition is very, very poorly defined and therefore it is very difficult, if not impossible to do any quantitative estimate of diffraction from such a diffraction condition.

Having said that these diffraction pattern obtained from reflection high energy electron diffraction comprises of streaks and not reciprocal spots.

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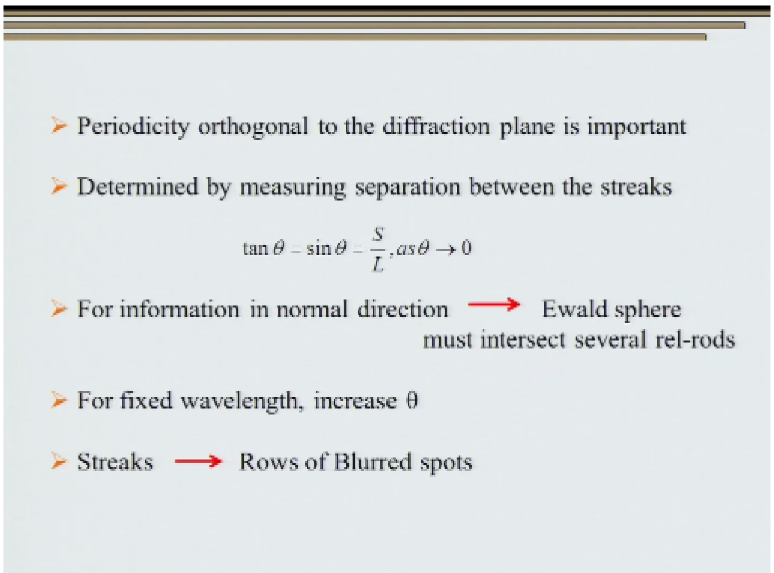

Now what information we get from this diffraction pattern that we are getting in reflection high energy electron diffraction? Well, we get the periodicity of the orthogonal to the diffraction plane using this, the diffraction pattern that we get in reflection high energy electron diffraction. Now this information is essentially obtained by measuring the separation between the streaks and it can be easily for very small angles you can always determine it using the similar formula like your $\tan\theta = \sin\theta = S$, where S represents essentially your distance between the two streaks, and L represents the distance between your sample and the screen.

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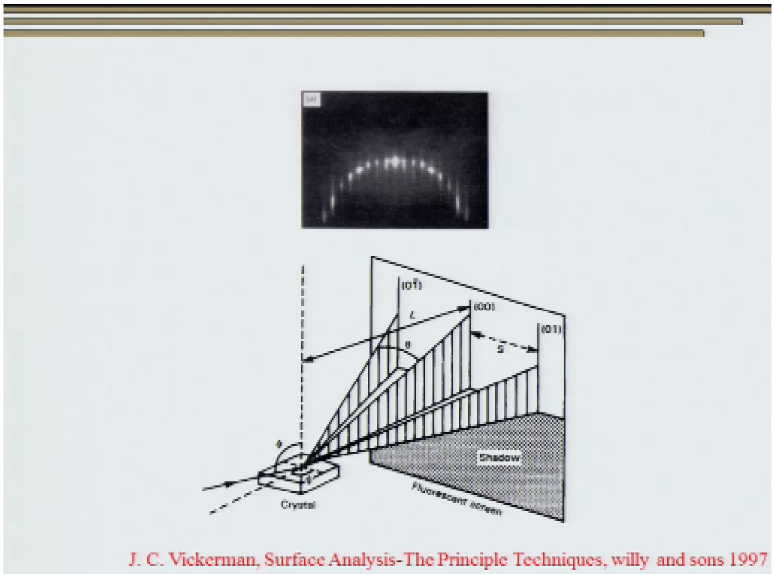

A better figure is given over here and here you can clearly see what exactly I meant. So we have a situation where we have an incident beam which is coming at a very small angle from the surface of the sample and you see how we get diffraction. You see this 00 spot, the 0 bar 1 spot and this 01 spot. You can see that in this plane of diffraction, you do see a lot of streaks which are seen over here also.

Now the distance between the streaks is which is given over here and what we figured out was that the angle what we are having in the plane is essentially proportional to S and the distance between the sample normal and the screen. So going back again we can clearly see that for information in normal direction, ewald sphere must intersect several rel-rods and that is what essentially happens.

Now for a fixed wavelength, if we increase θ, you can ensure that the streaks will be replaced with a row of blurred spots. Now this essentially happens because our diffraction condition itself is changing. So if we go back to this figure, you can see that this particular angle over here which is shown or you can say that the φ angle which is shown over here is changing. And if the φ changes we can see that these diffraction spots are essentially changing.

And from this we can get information about the kind of symmetry we are having in the normal direction.

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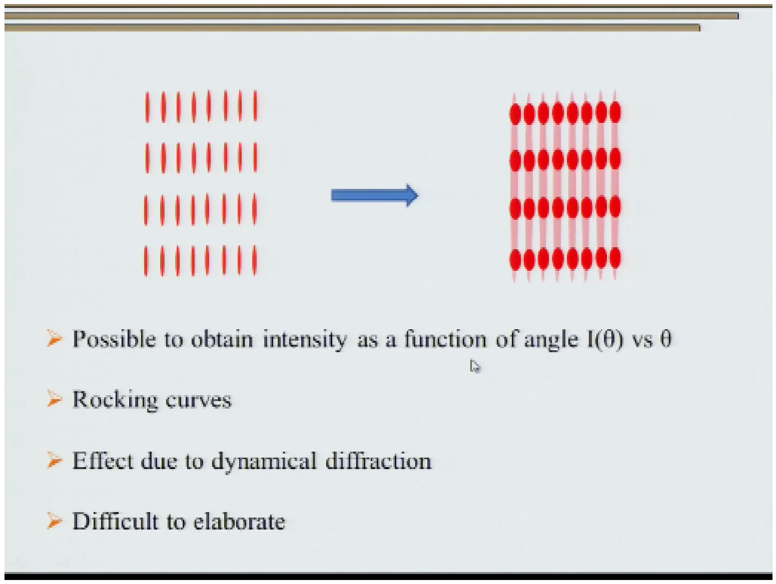

So this is what we mean by changing the angle φ. You see that instead of getting streaks, we do get these blurred spots. Having said that this is a bit of an exaggeration and you can consider that you get only one of the lines over here. So you end up getting only one such sale. Now this therefore by varying this angle, it is possible to obtain intensity as a function of this angle. Now this is very similar to rocking curve.

Now I would like to, we did not touch upon rocking curves essentially but what we do in rocking curve again we have to go back to X-ray diffraction, and if you remember the rocking curve all we end up doing is we go at a particular 2θ, we fix the 2θ, get a peak and now we vary ω right. Just to ensure what is the quality of the crystal or thin film that we have grown okay.

Similarly here also by changing the φ angle, all we are trying to see is, what is the quality of the crystal, or the thin film that we have grown on the surface. However, there is a considerable effect of dynamical diffraction. That is what was happening when we talked about getting rocking curve in case of thin films using X-ray diffraction. However, as we see again that as rather I had mentioned earlier the theory of reflection high energy electron diffraction is not very well developed.

In fact there are some groups who claim and I will show at the end of the lecture, some calculations which account for dynamical diffractions. But having said that this is more of a qualitative technique rather than getting quantitative information from reflection high energy electron diffraction, and therefore the results are slightly difficult to elaborate.

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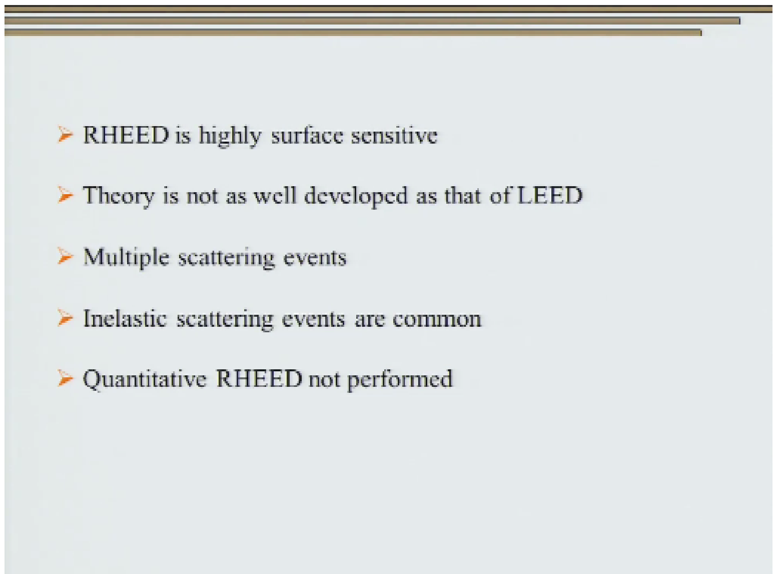

So as I had mentioned and the entire focus of going for reflection high energy electron diffraction is that, the technique is highly surface sensitive but as I had already mentioned, the theory is not as well developed as low energy electron diffraction and does not account for multiple scattering events. Having said that it is also characterized by plenty of inelastic scattering events.

And therefore, quantitative estimate from reflection high energy electron diffraction has not achieved as widespread use as that for low energy electron diffraction.

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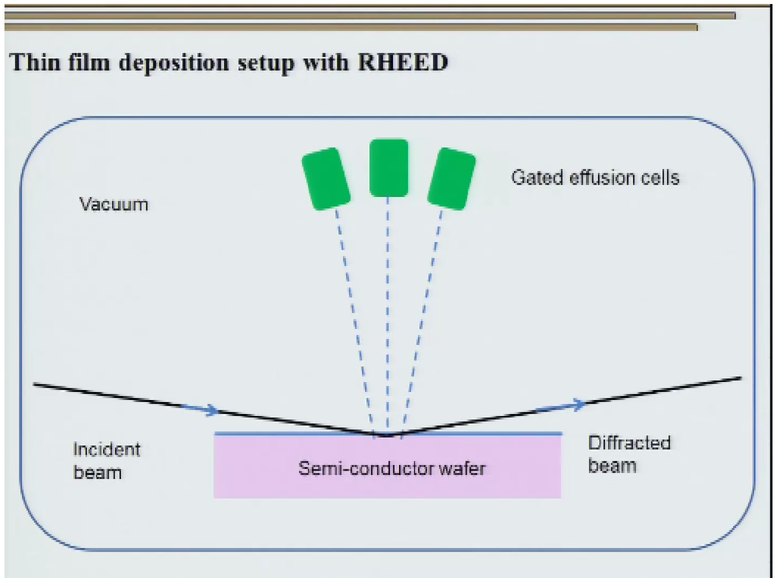

As I had already mentioned that one of the biggest USB that reflection high energy electron diffraction has is concerned with its ability to be incorporated in thin film disposition techniques. So the biggest advantage that reflection high energy electron diffraction offers is that we can actually monitor, in-situ during deformation the kind of layer or rather the kind of film that we are growing, not only the kind but also the quality of the film that we are growing.

Now this gives us a lot of advantage particularly for semi conductor industries where they are very interested in growing thin films of very high quality. Herein rheed offers a very robust tool where you can do in service monitoring of the quality of the thin film which gives us a very handy tool to play with the processing parameters. Now this also ensures that there is no need of doing post processing characterization.

Now this makes life much easier for processing for process engineer. So as I had mentioned, the biggest advantage of reflection high energy electron diffraction is in thin film to position setup. Now the basic advantage of reflection high energy electron diffraction is that it can be incorporated in any thin film deposition setup for say something for various deposition techniques like MOCVD or molecular beam epitaxy.

The biggest advantage that it gives to semi conductor industry is that you can really monitor, in-situ the quality of the film that we are trying to grow. This gives the process engineers a wonderful tool to play with. Therefore they do not have to do process optimization after the processing has been done. Instead we can monitor the evolution of the thin film under consideration while the thin film is growing.

And we can control the quality of the thin film which we are going to get. Therefore, this is the case where reflection high energy electron diffraction gets the maximum importance. A schematic showing the actual assembly having thin film deposition technique as well as reflection high energy electron diffraction is shown over here.

As you can visualize generally reflection high energy electron diffraction is used only as a qualitative tool to determine the kind of films that we are getting. And therefore, you can understand that during the processing of thin films, there is no way we are going to stop and do a entire analysis from the reflection high energy electron diffraction pattern that we are going to get.

And hence generally in most of the practical applications, reflection high energy electron diffraction data is essentially used to get qualitative information about the quality of the thin film.

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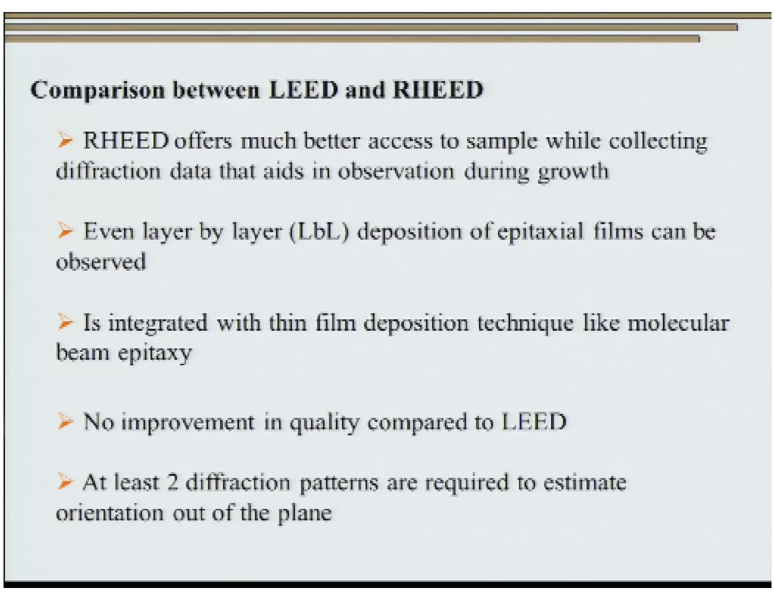

A brief comparison between low energy electron diffraction and reflection high energy electron diffraction is presented over here. As we had seen, reflection high energy electron diffraction offers much better access to sample while collecting the diffraction data that creates in observation during growth. This is the biggest USB of this technique. Having said that we have the ability to monitor layer by layer deposition of epitaxial films using reflection high energy electron diffraction.

I am going to show you some data at the end wherein we see how we can go as small as 4 or 5 mono layers and obtain precise qualitative data from reflection high energy electron diffraction. And as we had seen earlier it has integrated with thin film deposition techniques like molecular beam epitaxy and used on a routine scale in semi conductor industry. However, there are some disadvantages that reflection high energy electron diffraction has over low energy electron diffraction.

And that include the quality of the diffraction pattern that we get is not as good in case of reflection high energy electron diffraction. Having said that, in order to obtain complete information about the film in the plane or the symmetry in the structure of the film in the entire plane, we need to rotate the sample, because if you remember we had got information only in one direction.

In order to get complete information in two direction, we also need to rotate the sample. However, this will give us a lot of information about how exactly the quality of the film is in the plane.

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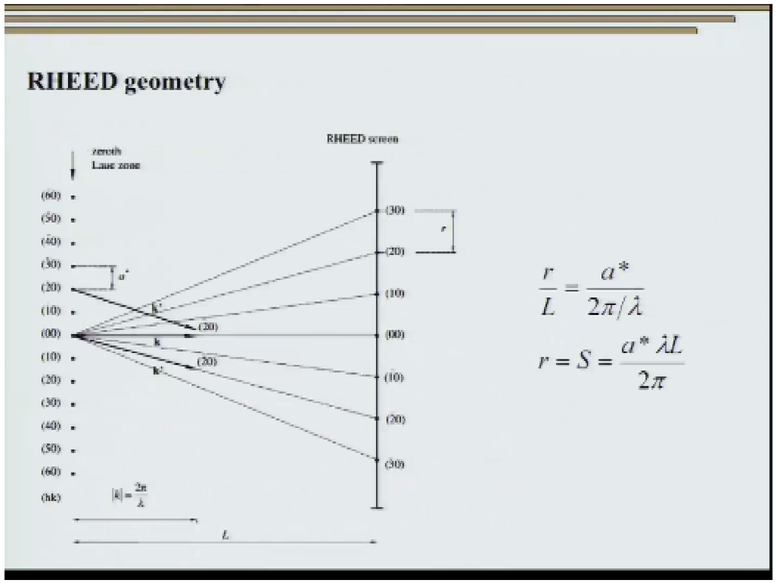

Another important thing regarding the geometry of reflection high energy electron diffraction is enumerated over here in this figure. So this is very similar to the earlier figure that we had seen. But this clearly shows that how exactly we get diffraction from different spots, and on the rheed screen each and every spot over here corresponds to a particular any diffraction spot corresponds to a reciprocal spot which in turn is again related to a real lattice point in the refraction pattern.

So here we see the 0^{th} level zone of the film under consideration and how it gets reflected or rather manifested on the rheed screen. Again similar to what we had seen earlier, the distance between the two spots as shown over here is proportional to A*, which is nothing but the reciprocal lattice parameter and L where L is nothing but the distance between the screen and the Lave zone.

I would like to remind you that this entity that we have shown over here is essentially in the reciprocal space. So let us not get confused. If you got parameter S/L and that is what was equal to R. So the same equation that we had got, the only difference is in this case, I have represented everything in terms of the reciprocal lattice vectors.

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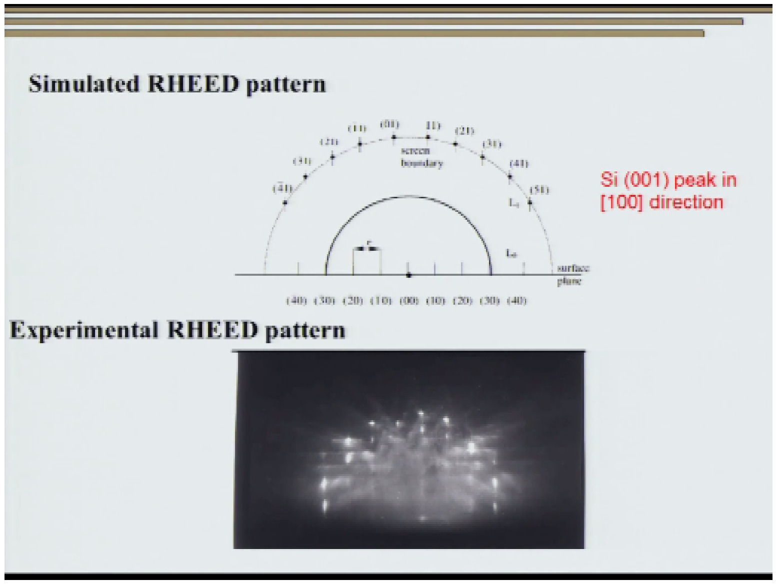

And why do we do a 4 mentioned exercise, these two essentially simulate the rheed pattern that we are likely to get. So herein you can see the kind of read pattern that you would obtain for a silicon 001 peak in 100, this is a silicon 001 crystal in 100 directions. So you can see depending on the different zone axis you get these all spots which you are getting over here and these can be indexed.

Now below is given an experimental rheed pattern. So just by qualitatively comparing these two patterns we can see that what are the diffractions that we are getting in the silicon film, and from this we can comment about the quality of the film. I would also like to mention that here you can see there is only one spot and there is a bit of streaking but here you see there are two spots and this is what I had shown you earlier.

Now this is one of the aspects that happens over here. So you see instead of having just one spot, you are having two spots. So this is what happens essentially because in experimental condition the diffraction criteria is relaxed in one dimension. Having said that I have just showed you, how we can get a qualitative estimate?

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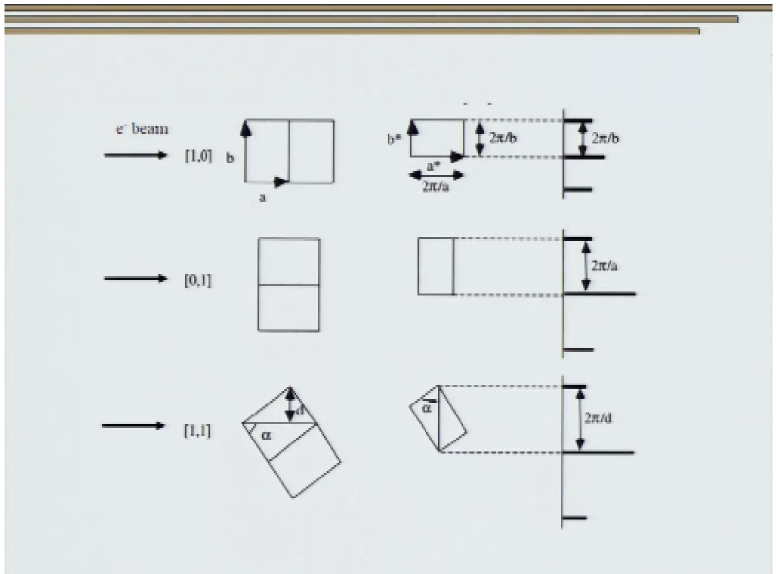

Now let us go ahead and see what all we can do using reflection high energy electron diffraction. But before we do that, if you remember in low energy electron diffraction, we had considered different unit cells and we had seen how they will look like in the reciprocal space. We can do a similar exercise for reflection high energy electron diffraction. But what differentiates read from low energy electron diffraction is that instead of having diffraction spots, we do get diffraction streaks and this is what is shown over here.

So you see different lattices and how they will look. Again this time also they are in the reciprocal space, but because of the geometry of reflection high energy electron diffraction, how do they get modulated? So mind you, this is your real space which is shown over here. This is your reciprocal space and this is how the pattern will look in reflection high energy electron diffraction.

So let us go back and this is why I had shown you this figure, where actually I showed that how your reciprocal lattice which is shown over here gets modulated into your rheed pattern which is shown over here. So the same concept which when you can extend. See this is something that we had covered in the last class and we saw that what actually happens is since here B is greater than A, your B* is lower than A*, which are the reciprocal vectors.

But what do we get in reflection high energy electron diffraction is not actually this much. Low energy electron diffraction we got all the spots over here. But here no, because of the gracing incidence we do get a streak pattern and this is how the pattern looks like. Now similar example for different unit cell configurations is shown over here and these can be easily computed.

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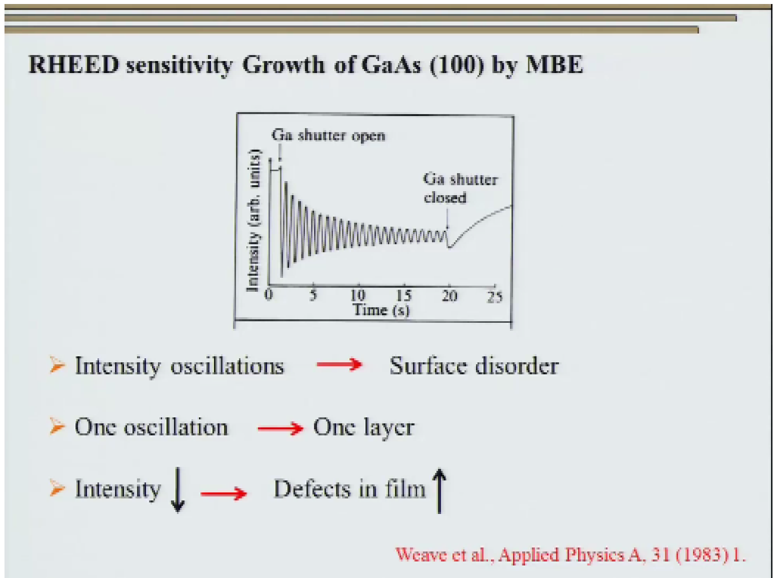

Now let us go and look at some real examples. So just to show you the rheed sensitivity I have given example of growth of Gallium Arsenide by molecular beam epitaxy on a silicon substrate. So here you see this oscillatory pattern, you have these oscillations which are dying off and you see here, you see that the Gallium shutter is open and after a particular amount of time, you close the Gallium shutter and you see as a function of time.

You see that there is damping of the oscillations. Now what does these oscillations correspond to? Well these oscillations actually give us information about the surface disorder. Another important thing that I had mentioned that each oscillation corresponds to each high intensity usage or here the intensity each oscillation or each high intensity actually corresponds to one single atomic layer.

So you can imagine the sensitivity of reflection high energy electron diffraction. So herein we have a very simple technique which gives us information about deposition of single atomic layer, mind you single atomic layer and here another thing that we can see that the intensity is gradually reducing. So now when does the intensity of diffraction reduce? Well, when most of the material that has been deposited is not satisfying the diffraction condition.

And therefore, we can conclude that, with increase in time there is a decrease in intensity which essentially indicates that the defects which are incorporated or which are getting incorporated in our Gallium Arsenide films are increasing with the deposition time. Now this may affect the quality of the film and we may not get finally good quality epitaxial films. So herein we have a situation or wherein we can monitor in-situ, the quality of the film and control it and use this information that we are obtaining to control the process parameters.

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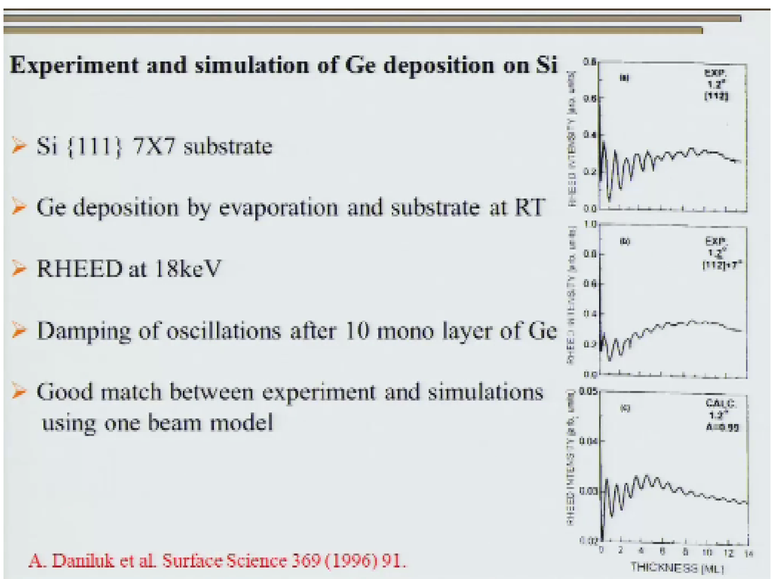

Another important example is for deposition of Germanium on Silicon. Now this classical example where we are going to look at a case wherein we are going to use simulations also. So there are certain groups who have developed something similar to what the simulation tools that we had seen in the last class for low energy for low energy electron diffraction, for reflection high energy electron diffraction also.

So in this case we are depositing Germanium by evaporation on a silicon 111 7 x 7 substrate. I would like to remind you that this 7 x 7 substrate is essentially a substrate on which there is restructuring of the first 7 atomic layers because of the dangling bonds. Having said that, the rheed or reflection high energy electron diffraction was carried out on the Germanium deposition at 18keV, and herein also you see that how the oscillatory nature is seen experimentally.

Now what all things we can do actually see it is silicon 111 and you see the incident electron beam is at 112 direction, and at an angle of 1.2 degree to 112. What essentially happens in the actual experiment is shown over here where the angle was varied to 7 degree. And here we see that there was damping of oscillations after 10 mono layers of Germanium and what we could do was actually incorporate these things in the simulation and obtain a good match using between the simulations and the experiment using a particular parameter.

I will not go in details of how exactly it is done because that is beyond the scope of this class, but I want to just impress upon you that there are certain groups who claim that we can do not just qualitative but rather a semi quantitative analysis using reflection high energy electron diffraction.

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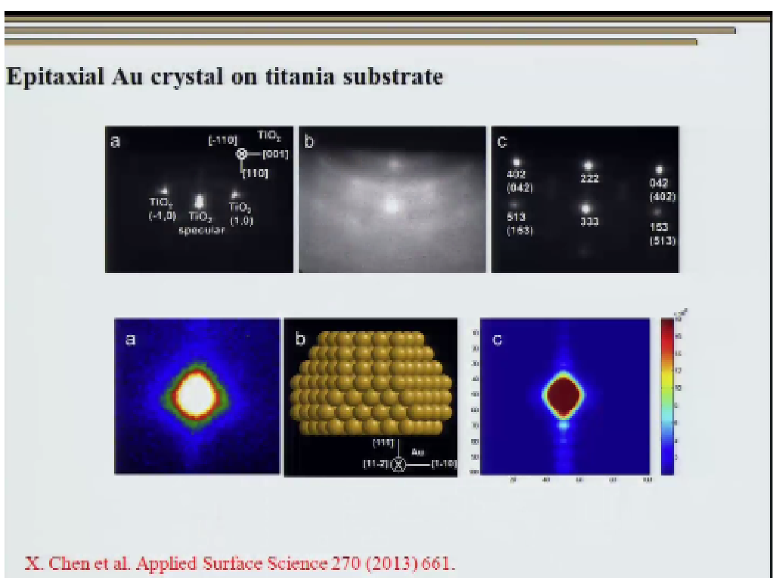

Now this is again another classic example from a paper by Chen et al. So in this case, there was growth of a epitaxial gold crystal on titania that is TiO2 substrate. So that is where rheed is of very, very great use to us, because rheed offer us not just information from the thin film or a quantum dot that we are growing but also from the surface under consideration. So here you see we see nice specular pattern and as well as normal diffraction from the titania.

Now as we grow you see that some additional spots appear. Once we try to index it, you do see that these corresponds to FCCAU. Not only that, if you look at the pattern in detail which is shown over here, we can go back and assume a particular shape of your particle that is being grown and back calculate the diffraction pattern from it. And if you see that there is a good match between the simulated diffraction pattern and the obtained diffraction pattern.

We can believe that not only the shape, but also the size of the epitaxial gold particle that has grown on the titania substrate. So this is how I talked about getting qualitative information from rheed and herein I am presenting how we can get a lot of quantitative estimate, not only about the shape, but also the size of a single particle using reflection high energy electron diffraction. So this is one technique which is growing very fast.

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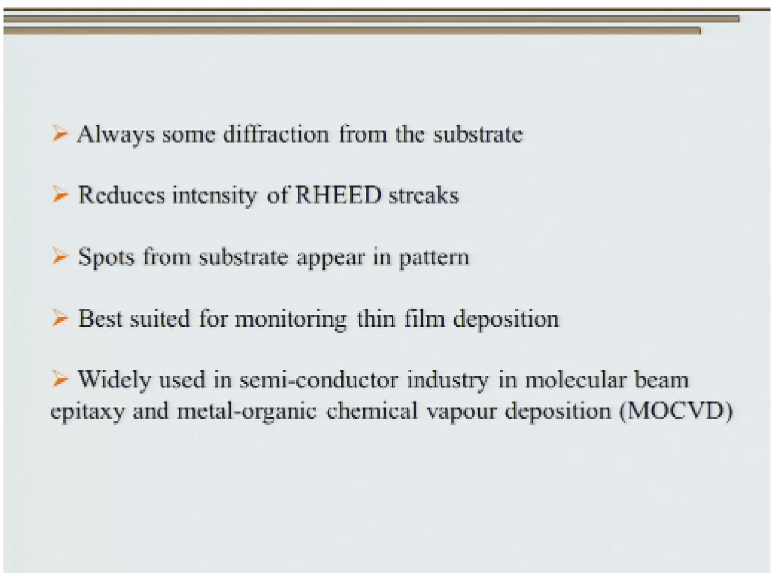

So as I had mentioned, we always end up getting some diffraction from the substrate. So this may be used in a very advantageous way as I showed you in the last example. However, having said that, in most cases in contributes to reduction in intensity. It also leads to additional spots in the diffraction pattern and it is best suited for monitoring thin film. Just a simple qualitative way and other simple qualitative way rather to get more quantitative information, things are a bit involved.

But having said that which comes even closer to reflection high energy electron diffraction when it comes to semi conductor industry where it is used on a routine basis for growing epitaxial films using metal organic chemical vapour deposition or molecular beam epitaxy.

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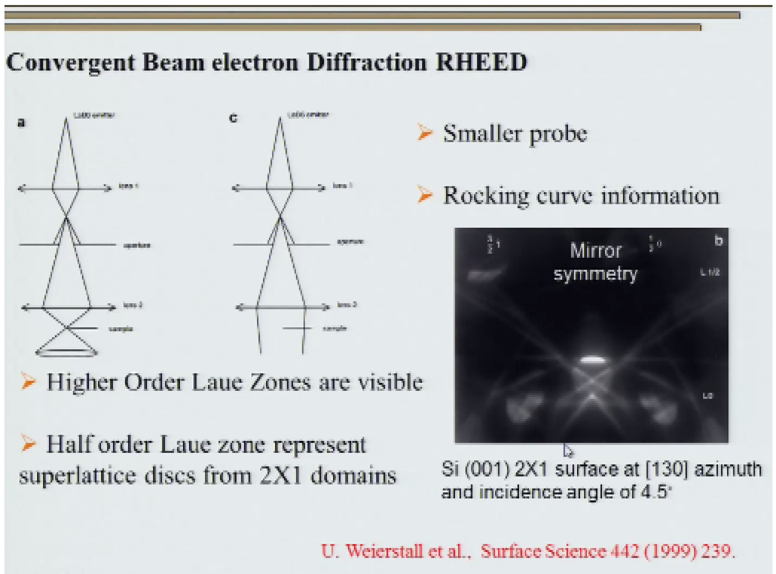

Another important thing, now that we saw in case of low energy electron diffraction that at the end of the day we are having a electron beam, and if you remember we had also used low energy electron diffraction to get some images but having said that the energy of electrons in low energy electron diffraction was very small, 20-500 eV. But now we have energy as high as a low kV transmission electron microscope.

So what we can do essentially is actually use this high energy electron beam to get convergent beam electron diffraction. So here again I have shown you different geometry. You see what kind of geometry we are having over here when we are doing normal reflection high energy electron diffraction. The beam is not at all converged. However, if you want to do convergent beam electron diffraction, as we do in a TM, what do we do?

We take the electron beam and using the voltage of the lenses we actually converge. Now what are the advantage that we get? Well, we do get a smaller probe. Not only that, this is equivalent to getting or rocking curve information and you see unlike the read pattern that I had shown of till now here, you can see these nice lines. Now these lines are in fact the same thing that we get in a convergent beam electron diffraction and carry information about the symmetry that is existing in the entity under consideration.

And herein we can clearly see for a silicon 001 2 x 1 substrate that there is a mirror symmetry. And what actually we are seeing in this particular slide are actually the higher order Laue zones, and what we see here in this particular example is the half order Laue zone that represent the super lattice discs from 2 x 1 domains of silicon 001 which clearly indicates that there indeed is a mirror symmetry for this reconstructed surface.

So you see we started from normal reflection high energy electron diffraction and understood that we are not going to get much of quantitative information. And at the end of the lecture I have contradicted myself and presented to you what all quantitative information that we can get from reflection high energy electron diffraction. However, it is to be mentioned that one needs to take a lot of care before extracting such in depth information from reflection high energy electron diffraction.

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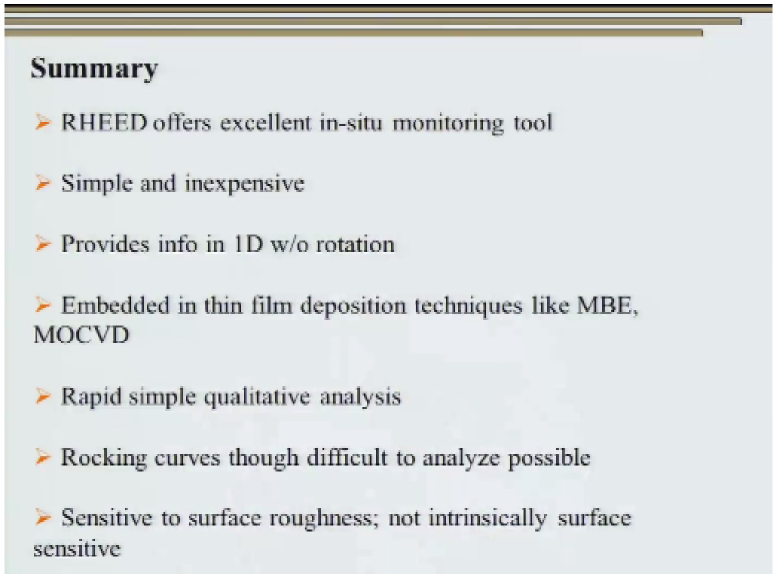

So to summarize we have seen that reflection high energy electron diffraction offers excellent in situ monitoring tool which is very simple and inexpensive. However it provides information only in one dimension. If you want to get information inside the entire plane of the paper or rather in the plane of the sample we need to rotate the sample. We have also seen that how it can be embedded in thin film deposition techniques like MBE and MOCVD and can be used on a routine basis.

It gives us a rapid simple qualitative analysis. We have also seen that we can get information not only about CVD but also rocking curves. However, these things as I had already mentioned are very difficult to analyze but it is something that can be done using reflection high energy electron diffraction. Having said that I should also mention that one of the biggest disadvantage of reflection high energy electron diffraction is that it is very sensitive to the surface roughness.

And therefore, it is very important to see what kind of sample we are using. And therefore, it is very, very useful only for in situ growth. Having said that I hope that I have been able to present you how can we use electron diffraction to get similar and at times even better information than what you obtained using X-ray diffraction.

In the next class, we are going to just change slightly the course of our training and go from diffraction and scattering, we will touch upon scattering, we will leave aside diffraction for some time. Therefore, one lecture we go and study about spectroscopy, then we again come back and do neutron diffraction. Till then have a good time. Thank you.

Acknowledgement
Ministry of Human Resources & Development

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Co-ordinator, NPTEL IIT Kanpur

Prof. Satyaki Roy
Co Co-ordinator, NPTEL IIT Kanpur

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K.K Mishra
Jai Singh
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