Techniques of Materials Characterization

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Lecture – 21
Electron Diffraction in TEM - Continued

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. We are now in fifth week that is we are continuing with fifth module. We are discussing about transmission electron microscopy. In the previous week we mostly discussed about the image formation in transmission electron microscope and different type of contrast generation, different sources of atomic contrast.

Atomic number contrast, mass thickness contrast, diffraction contrast, bright field mode, dark field mode, HAADF, STEM all these modes we have discussed elaborately and then we just started with another very important aspect of electron microscopy particularly in transmission electron microscopy and that is electron diffraction. So, there first we discussed about why diffraction happens, what is so special about scattering, how it is related to the crystallography of a material?

So, we saw example for an amorphous material where the scattering centers are all randomly oriented. And then we saw that how the changes happen in case when we have scattering centers all regularly arranged that is in a crystalline material. And then we discussed about the Bragg's law. We discussed about the order of diffraction so on and so forth. So, this discussion we will be continuing this week again on electron diffraction.

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And in this lecture we will be covering this issues this topics that is interrelation between the real lattice and the diffraction pattern. Basically, the interrelation between the crystallography on the corresponding diffraction pattern of the crystalline material. Then we will discuss about a very important concept called reciprocal lattice which will help us to understand the diffraction phenomena.

And later when we discuss about the identification or discussing about the indexing of the diffraction pattern, then this reciprocal lattice concept will be very helpful. So, I put it ahead of discussing about the diffraction patterns and indexing the diffraction patterns. Interrelation between reciprocal lattice and the diffraction pattern and that will help us to understand diffraction phenomena much better.

Concept of Ewald sphere and finally we will see an example of usefulness of this concept reciprocal lattice and Ewald sphere when we discuss about the electron diffraction from a real crystal.

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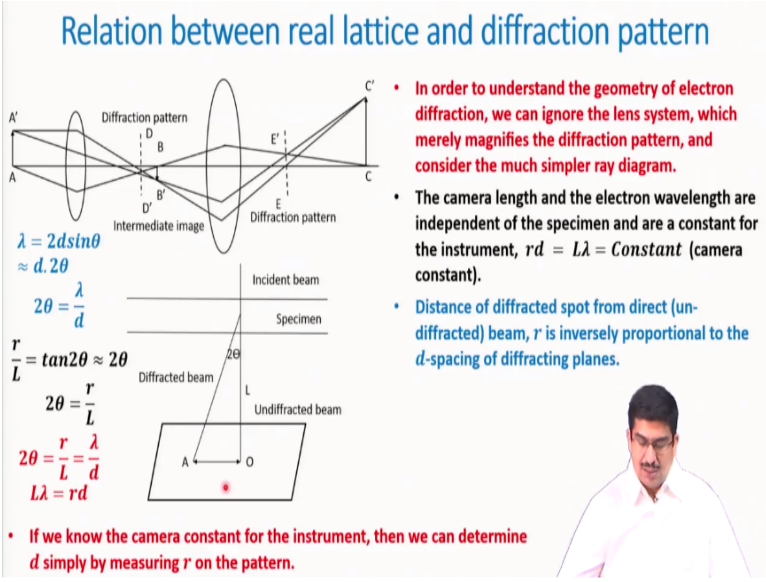

So, first the relation between real lattice and the diffraction pattern. So, what is the relationship between the crystallography of material and the corresponding diffraction pattern? So if we imagine that this is how basically any microscope works, so we have an object and we have the corresponding diffraction pattern of that object. So, diffraction pattern we kept on saying that it is basically captured at the back focal plane of any microscope.

And there are projector lenses and so on and those projector lenses finally magnify this. So, if we essentially remove this entire lens system, we just magnify which helps to capture the diffraction pattern. So, what we can imagine schematically is that we have the specimen. On that specimen there is an incident beam that falls over the specimen and then it hits the image plane where we capture the diffraction pattern.

So, this is the direct beam where the direct beam hits we consider that as the origin and then we have a diffraction spot corresponding to the diffracted beam which is basically diffracted or scattered at an angle of 2 theta. So, this is the incident beam and we could have also drawn these atomic planes which are parallel to this incident beam which are basically deflecting and causing this diffracted beam at an angle 2 theta and finally this is how the diffraction pattern basically forms here.

And if you look at this diffraction pattern, this is the diffraction. This is the direct beam and the diffracted beams falls on this and diffracted beams are also represented by spots or also formed some kind of a spots here. The direct beam location is this and we can consider this as the origin and all these other spots are basically this diffraction spots here this A.

Now, from this geometry, we can derive some very important relations between the specimen, the atomic spacing in the specimen and this distances in the diffraction pattern. So, we start with Bragg’s law. Again, we go with the lambda = 2d sine theta. And we know for at least for electron diffraction that theta is very small. So, basically that means this sine theta comes down to theta.

So, we can write it in this way lambda = d into 2 theta. And that means we can again write 2 theta = lambda by d. This is what is Bragg's law. This is another form of Bragg’s law; we derive it from there for electron diffraction okay. Then, here in this simple geometry, if we consider this simple triangle here and this triangle will present this kind of relationship r by L, r is basically this distance that is the distance between the origin and the diffractive spot.

So, this distance is r, L is this distance that is the distance from the specimen to the origin of the diffraction pattern. So, this perpendicular distance are basically the distance between the specimen to the image plane. This is really not a real distance, is a virtual notational distance, we will come about this when we really talk about the indexing the diffraction pattern, then we will understand the meaning of this distance.

For now, we can work with this relationship r by L = tan 2 theta. Again, similar to sine 2 theta for a very small value of theta this comes down to be 2 theta. So, we can write 2 theta = r by L. Now from these two relations; this 2 theta = lambda by d and 2 theta = r by L, what we can get is this relation finally 2 theta = equal r by L = lambda by d. That means L lambda = rd okay. So, now what does this mean basically?

The camera length is L, L is called the camera length okay and most often for most of the microscopes this length is physically constant to the distance between the specimen and the image plane is basically constant, physically it is constant and all it varies is basically we have already discussed enough about these lenses or electromagnetic lenses we basically change the focal length of the lenses very easily and that is how we change the magnification and so on.

But, basically the physical distance between the image plane and your specimen remains almost it is constant for any kind of microscope. So, virtually L is constant. Again the wavelength is constant because that is what will satisfy this Bragg's equation. So, lambda is constant here, for a constant lambda only we will get a diffraction for a constant d because d the interatomic or interplanar spacing is also constant for a particular type of material.

So, altogether what we can imagine that L lambda is basically a constant and that constant is usually called the camera constant, we will again discuss more about this. So, rd is equals to a constant. What does this mean is the distance of the diffracted spot from direct or un-diffracted beam, so, this distance basically from this origin this direct beam to the diffracted beam you can imagine this is a center-to-center distance and this is what you can possibly measure also in a real diffraction pattern.

You can very easily measure the distance from this direct beam to the diffracted beam center to center distance, that distance is inversely proportional to the d spacing of the diffracted plane. The planes which are causing this diffracted beam, this interplanar spacing of those planes that d is inversely proportional to this distance. okay. So, if we know this camera constant, this L lambda.

If this is known, then we can very easily determine the d value that is the interplanar spacing of the specimen for any particular set of planes. The plane that is giving rise to this diffraction spot we can find out that d value from measuring this r, this distance, this distance is the real distance we can measure it in the image and from there we can make an idea about the d value of any particular atomic plane, right.

So, this is how the diffraction patterns are related to the crystallography of the specimen. This is the relationship This is another way of saying the same thing the relationship. We discussed about the relationship of diffraction phenomena with the crystallography of specimen, this is another way of saying this how the diffraction phenomena is related to the crystallography of any material.

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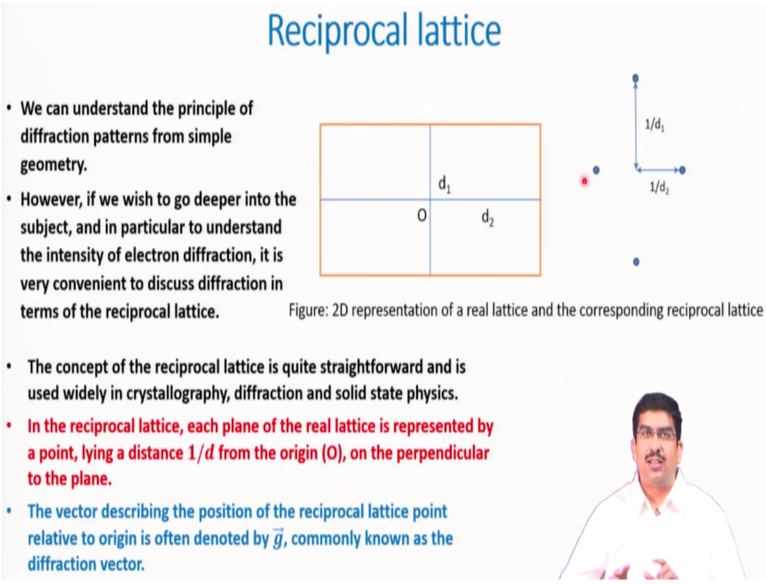

Now, we go to the concept of reciprocal lattice. First of all, reciprocal lattice is very useful as I said to understand the concept of electron diffraction. Electron diffraction we have understood by now from Bragg's law construction and all, we have understood it very nicely with the geometry. So, if you remember the Bragg's law, we have proved this Bragg's law from simple geometry itself.

But this concept will be much more easier or even more in order to understand the diffraction pattern from a real material, this reciprocal lattice concept is very useful. So, what this reciprocal lattice does is that basically in the reciprocal lattice each plane in the real lattice, so this is a real lattice. Let us say this is one real lattice, here I have this set of planes, two sets of planes I have.

So, I have this one set of planes which are parallel planes, this is one plane, the middle one is one plane and this one is again one plane. So, these three is forming one set of planes. Again similarly, this is another set of planes. These two sets of planes are perpendicular to each other. So, this is one set of planes, the middle one is again one set of planes and this third one is one set of planes.

I am taking a section of this plane. Let us say this all these planes are going through perpendicular to the screen. So, these are just resection on the screen and these two sets of planes are there and the distance between this or interplanar distance that is the distance where imagine this origin O and the distance from this plane to this plane is d 1 and this plane to display is d 1.

So, I have to set of planes which are having interplanar spacing d 1 and d 2. Now what happens in reciprocal lattice, we basically represent the planes with a corresponding point with the origin So, this is basically the origin here and then we represent these three planes. So, we have this set of planes, this one, two and three these planes are represented by this dot and then origin dot of course and this another dot here.

Similarly, these two planes this one and this one if we ignore the origin plane, so this one and this one, these two planes are again represented by these two points right. Another point is the way these points or these planes here are related in the real lattice and reciprocal lattice they are related is that now what I have the distance from origin to this point, these points are corresponding to basically this plane, this atomic plane and this atomic plane.

So, these points, this distance from origin is 1 by d. So, this is how reciprocal lattice is basically inversely related to the real lattice. Similarly, the distance from the origin to this point and this point belongs to basically this atomic plane that distance is 1 by d 1, this one is 1 by d 2. So, this is how these two are related and the vector that basically describe the position on the reciprocal lattice point relative to the origin.

So, from this point to this point; the vector that runs through from this point to this point or from this point to this point that vector is known as g, g vector and that g vector is commonly known as diffraction vector. So, in case of when we use the reciprocal lattice concept to diffraction phenomena that is the diffraction vector.

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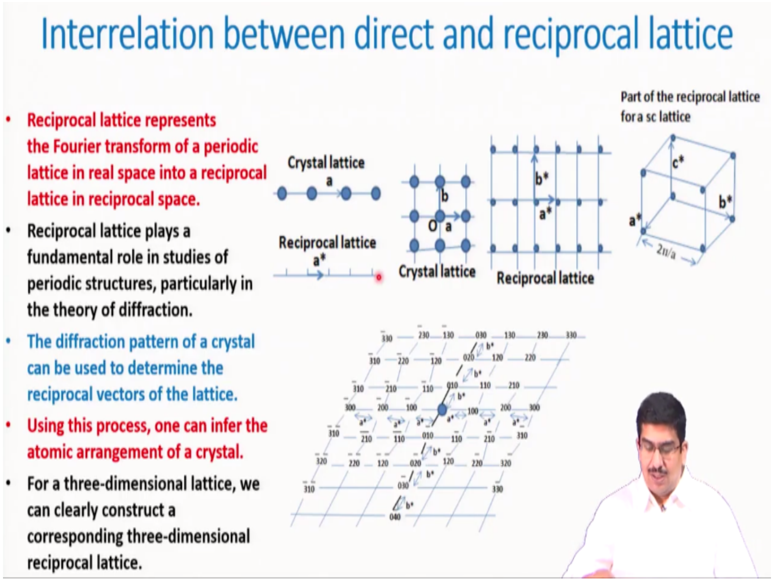

So, this is the relationship between reciprocal lattice and the real lattice and we can go on and derive many relationships between the real crystal lattice or the lattice parameters and interplanar spacing to the reciprocal lattice and all, more or less what it represents is basically in the real lattice the planes which are two dimensional is now represented by a point which is one dimensional in reciprocal lattice.

So, there is a decrease in the dimension in case of a reciprocal lattice from the real lattice. So, similarly, a three-dimension real lattice will come down to a two-dimensional reciprocal lattice and so on and so forth. In some sense, what we can think about is that reciprocal lattice, this kind of if you just imagine that this reduction in dimension, reduction in dimension is basically is similar to projection.

So, if you understand any kind of projection; we have discussed about the shadow formation and all, shadow is basically your projection or your three-dimensional object and you are projecting on a two-dimensional plane as your shadow so that is the projection and that is related to a decrease in the dimension. So, this is similar kind of a concept and the reciprocal lattice whenever there is a decrease in dimension, the mathematical way of representing this is Fourier transformation.

So, we can imagine that reciprocal lattice represents the Fourier transformation of a real lattice, periodic real lattice into the reciprocal space. The reciprocal space as I have said the way it is defined is basically planes going down to the point and the distance is inversely related to the real lattice so that through a Fourier transform, you can change the real lattice to reciprocal lattice and vice versa.

So, you can construct if you know the real lattice, you take a Fourier transform mathematically and then you can construct the corresponding reciprocal lattice. If you know the reciprocal lattice, you can do a reverse Fourier transformation and you can construct the real lattice that is the relationship between them. So, of course that is why reciprocal lattice is so important in studying many properties or many periodic structures or crystalline structures.

They can be very nicely studied by reciprocal lattice and diffraction is just one of these examples. Also, as I said the diffraction pattern is related to the reciprocal lattice, just in a minute we will see how these two are related. Basically the diffraction pattern of a crystal can be used to determine the reciprocal vectors of the lattice and vice versa again. So, diffraction vector, diffraction phenomenon or diffraction patterns can be identified by the reciprocal lattice.

And from the diffraction pattern indexing the diffraction pattern we can identify the reciprocal lattice of any real lattice for a real lattice. So, these three are basically connected; real lattice, reciprocal lattice, diffraction pattern. These three things are completely connected and if you do a back calculation, if you know the reciprocal lattice you can or if you know the diffraction pattern, you know the reciprocal lattice you can infer the reciprocal lattice and from there you can construct the real lattice.

You can know the atomic arrangement in a crystalline material. This is how basically from the diffraction pattern we can construct the atomic arrangement through the reciprocal lattice and the best part of it is that you can virtually create any diffraction pattern for any material. So, you take the element and let us say you know the atomic arrangement of an element you can correspondingly derive its diffraction pattern through this reciprocal lattice concept.

And if get a diffraction pattern, you can solve that, you can construct the reciprocal lattice and from there you can go to the atomic arrangement and construct the crystal structure of any material that is what.

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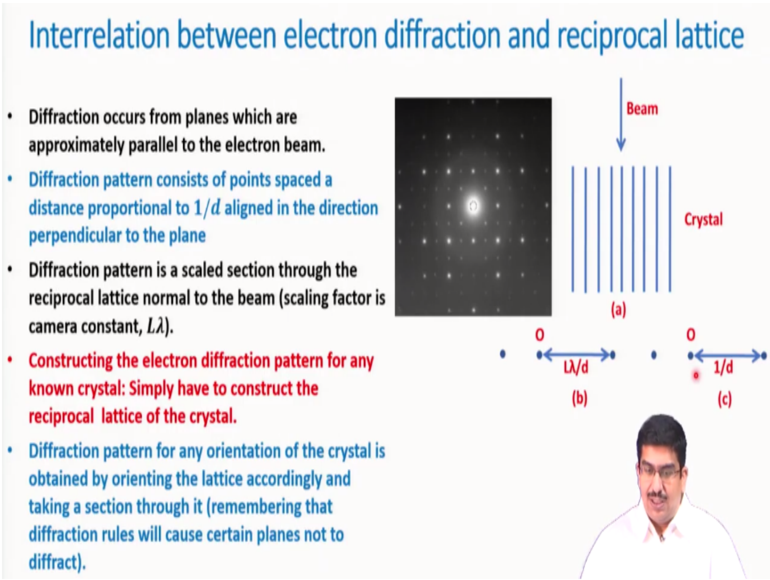

So, definitely as I already explained interrelation between electron diffraction and reciprocal lattice and that relationship is very much true for and it is very direct relationship for electron diffraction because what happens in electron diffraction, we already discussed this that electron diffraction basically because of this lambda because of this very small wavelength of the electrons these diffracting planes are essentially parallel to the incident beam.

So, what happens is that this incident beam comes these parallel crystalline planes are diffracting and from there what I am getting is this diffraction pattern. So, this diffraction pattern if I now imagine that this is basically the origin or this point is this point, the origin point, and already we have discussed the relationship between real lattice and diffraction pattern.

The distance from this direct beam to the corresponding diffraction pattern that distance r I can represent as L lambda by d. Now, again if I take a reciprocal lattice of this atomic planes, these parallel atomic planes, there also there is an origin and from that origin I have the distance of the reciprocal lattice point is 1 by d. So, basically the diffraction pattern is a direct representation of reciprocal space or reciprocal pattern or reciprocal lattice of direct.

So, that is how these three are connected. This one, the diffraction pattern and the reciprocal lattice; these three are completely connected and you can imagine that the diffraction pattern is basically a scaled section through the reciprocal lattice normal to the beam parallel to the beam. So, you have this reciprocal lattice and then a normal parallel to the beam, so you take a parallel section through this reciprocal lattice.

And that is your diffraction pattern with a scaling factor of L lambda that is the camera constant that is what is the relationship between real lattice, reciprocal lattice and diffraction pattern. So, basically as I said that you can construct the electron diffraction pattern for any known crystal if you know this. If you know this atomic arrangement, if you know the d value, if you know the atomic interplanar spacing, the symmetry and so on, you can very easily construct a diffraction pattern for any kind of zone axis.

All you have to know is the zone axis. So, for any kind of zone axis that is the orientation of the crystal you need to know the zone axis and then corresponding lattice you just need to section the reciprocal lattice you have to take, you know the zone axis and then perpendicular to the zone axis you have to section the reciprocal lattice, that three-dimensional reciprocal lattice of course, and you will get the diffraction pattern.

So, virtually I can generate the diffraction pattern for any kind of any zone axis and this is very important in order to create a library that is what, we will discuss about this when we discuss about the indexing of electron diffraction pattern, we will know implication of this that you can essentially you get. Let us say you do not know the zone axis, but you know the crystal, you know what is the d spacing and all, so you can find out the zone axis.

You can just get an electron diffraction pattern and then you match it, you have a library function, you keep on bringing them and you try to match them because you have a library function because you know the reciprocal lattice. So, you keep on do this trial and error and that way you can find out, you can match your diffraction pattern and you can find out that what is this zone axis that is one way of thinking this interrelation as well. So, this is how this relationship between these three works.

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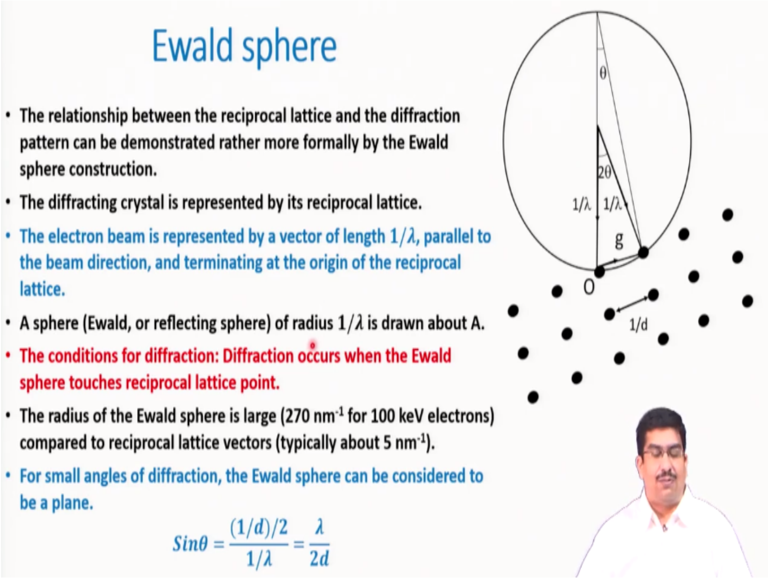

Now, the concept of Ewald sphere. We are going a little ahead and from reciprocal lattice we are trying to connect the reciprocal lattice with a diffraction phenomenon. So, this reciprocal lattice and Ewald sphere construction, this helps us to understand the relationship between the reciprocal lattice in a diffraction pattern even better and even for a finite crystal which we will see in a minute.

So, here now, we have the diffracting crystal which is represented by its reciprocal lattice. So, we have the reciprocal lattice, completely two-dimensional reciprocal lattice which is coming out of this three-dimensional real lattice. From there we are constructing, we are knowing or we are getting this reciprocal lattice here. So, this point is say origin here in this part.

What we do is that we represent the electron beam by a vector of length 1 by lambda, electron beam in this construction, in the Ewald sphere construction, we are in reciprocal space. We are constructing a sphere and that radius of the sphere is basically 1 by lambda and this is the point of this origin point of that sphere, the sphere has a radius of 1 by lambda and that sphere is called an Ewald sphere.

So basically, if I know the electron, wavelength of the incident electron beam in the reciprocal space I can easily construct a sphere with a radius of 1 by lambda right. What happens is that and I imagine that okay fine that 1 by lambda that radius will meet the direct beam definitely because that is the direct beam that is how the electron is coming and they are falling on the direct beam on the origin of that reciprocal lattice that is 1 by lambda.

The condition for diffraction in this construction is basically that diffraction occurs when the Ewald sphere touches any of the reciprocal lattice. So, that means, if I construct this 1 by lambda this sphere, Ewald sphere with a radius of 1 by lambda and I make the Ewald sphere to touch the origin, it has to touch the origin. Then whatever other points, reciprocal lattice points it is touching the Ewald sphere is touching those points will be seen in the diffraction pattern.

Or other way round those are corresponding to that reciprocal lattice point whatever the real atomic plane, in the real lattice whatever atomic plane corresponds to that point in reciprocal lattice space those atomic planes are satisfying the diffraction conditions, they will be diffracting. So, this is another way of saying that same diffraction phenomena. Now, earlier we were saying from the Bragg's law that it has to be satisfied lambda equals 2 d sine theta.

When that satisfies then some atomic plane will diffract and they will be seen in the first order if you capture the first order diffracted beam say those are the points. So, basically if you look at this, then this is a single crystal of NaCl. So if you look at this, all of these points are seen because they are satisfying the diffraction condition. Bragg's law is satisfied for them that is what.

Now, we are saying the same thing, but in a different way that this Ewald sphere this is satisfied and that means now this Ewald sphere is touching the reciprocal lattice points and that is how we are getting the diffracted beam. So, this is from corresponding to this atomic plane and we are seeing them in the diffraction pattern. So, if we now look at this Ewald sphere and we imagine that this angle, this theta angle is very small which is true for electron diffraction.

The theta angle, the diffraction angle is very small for electron diffraction. In that case what happens is that the radius of the Ewald sphere is very large because it is 1 by lambda. So, lambda for electron if you remember around very small, around 300 kV electron it was 0.002, around that 0.002 nanometer that was the kind of typical value for lambda in case of an electron diffraction.

So, what happens is that in case of electron diffraction the radius of the Ewald sphere is very large. It is very large, is around 270 nanometer reciprocal for 100 kV of electrons and compared to that the reciprocal lattice vectors which is 1 by d basically, so reciprocal lattice vector which is 1 by d for a very typical material, any typical metal it is almost about 5 inverse nanometer.

So, what happens is that compared to this spots, these reciprocal lattice spots, the Ewald sphere is really big, it is very big and essentially what happens is that the Ewald sphere becomes like a plane for this reciprocal lattice points which are very close to the origin. Reciprocal lattice point very close to the origin or close to the origin, for them the Ewald sphere is essentially a plane because of two factors.

One for the radius of Ewald sphere is for electron diffraction, radius of Ewald sphere is very large. And for electron diffraction again theta is very small. In that construction, what we can write is sine theta, if we imagine this sphere here this radius also, the next point where it touches that radius also is 1 by lambda. So, what we can see is that for this triangle if we consider we can imagine the sine theta comes out to be 1 by d by 2 by 1 by lambda, finally it comes down to lambda by 2 theta.

So, again the same Bragg's law is meeting. So, this is another way of showing that diffraction happens when this condition is satisfied and when this condition is satisfied that means Bragg's law is satisfied. So, this is proving the same thing, but in a different way.

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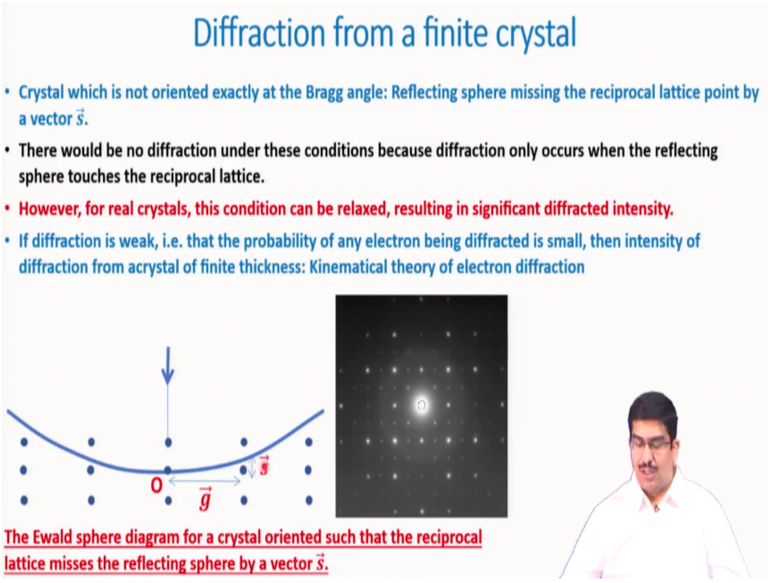

Now, the Ewald sphere concept we can very easily use it for understanding the diffraction phenomena from a finite crystal, at least for one or a couple of things in the finite crystal. So, first thing to understand here is that the reciprocal lattice, so we have an atomic plane and corresponding to that we have a point, now the point is a zero-dimensional entity remember one thing.

So, reciprocal lattice that points are zero-dimensional entity, but the diffraction pattern is a real thing and there we are getting a finite size for the diffracted beam or diffracted spots, right. So, that means if we imagine the diffraction patterns are just a mere representation of reciprocal lattice and vice versa, then these diffraction points should have been a point.

An ideal point without any dimension without any spread around that, no spread should be there, but they have a finite dimension. That means we can imagine that there is some kind of a broadening happens for this reciprocal lattice point. So, what is that broadening? How we are getting a spot with a finite size in diffraction point or diffraction pattern that we can explain by using this Ewald sphere and reciprocal lattice concept.

So, what happens is in case of a real finite lattice or real lattice or real crystal with a finite size, we can imagine that the crystals which are not oriented exactly at the Bragg angle, they still can diffract. So, in those cases what happens is that as just now we told that reciprocal lattice the condition for diffraction is the Ewald sphere should touch the reciprocal lattice point and we have shown that that condition, Bragg's law also gets satisfied.

So, what happens is even if Ewald sphere is not touching the reciprocal lattice point, it is missing the reciprocal lattice point even in that condition, we will be getting diffraction conditions satisfied. That means there is a relaxation in the Bragg's condition and because of the relaxation there is a finite width for this diffraction spot. Ideally this point should not diffract, but still because the Ewald sphere is not touching them.

They are missing them by a certain angle or a certain amount, still we are getting diffraction from the spots and that is what is causing this spread. So, that means the Bragg's condition is relaxed for real crystal and that result in a significant diffracted intensity from any diffractive spots, this spread, this intensity shows a real. We are getting a diffracted intensity from any atomic plane.

Because sort of even if it is slightly deviating from the exact Bragg condition, exact Bragg angle, exact scattering and still it is diffracting that means some amount of the spread is happening and this concept is for a real specimen. This real specimen concept this is called, this diffraction that is even if we miss the reciprocal, Ewald sphere is missing reciprocal lattice point, Bragg's condition gets relaxed and so on, this is called the kinematical theory of electron diffraction.

So, we will not go into any details of this theory, but we will just see what is the implication of this entire theory here. So, what we can imagine also that if we look at this reciprocal lattice, the section of reciprocal lattice and the Ewald sphere that is passing through them, so this is the diffraction vector. We already know that the distance between the vector that is representing this origin with the reciprocal lattice point that is the diffraction vector, g vector.

Now we are getting another vector, we are defining another vector basically that vector tells us that what is the distance between the reciprocal lattice point and the Ewald sphere that passes very close to it but not touching and this point is still diffracting. So, that distance is represented by another vector which is called as s vector.

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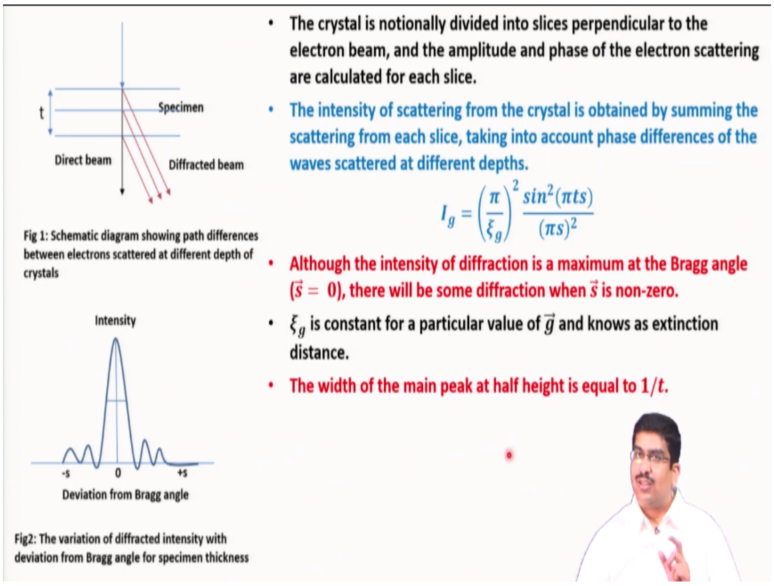

with this now, we will be seeing again this kind of a construction. So, we now imagine that I have this real specimen here and this real specimen is divided into; notice notionally, it is not really or we can imagine that these atomic planes are there notionally, this specimen is divided into certain slices; all of those slices are giving the diffracted beam with a particular kind of angle.

With this definite phase relationship, this diffracted beam is finally coming out of this specimen because of the finite size of the specimen. So, this is the direct beam and these are that diffracted beams coming out from various slices with a particular phase relationship between them. Now, the intensity of the this scattered, intensity of this diffracted beam, we can get it by summing up these diffracted beams coming out from individual slices.

Different sized slices from the specimen these beams are coming out and we can just sum them up considering their phase relationship and we can finally get the intensity of this entire diffracted beam, remember the angle theta is same. So, for each of them Bragg's condition is satisfied at a particular scattering angle. So, finally if we look at that intensity, I am not going again how it is derived and all, so if you just look at this intensity here and intensity is expressed by few of these terms.

The xi term is here, this term is there and then this s, s is the vector that by which this reciprocal lattice points are missing the Ewald sphere and t, another vector t, t is basically the thickness of the specimen finite, the specimen this crystal the thickness of that finite crystal. So, now if we see this intensity distribution with respect to s that s vector and remember xi is here, it is a constant for a particular value of diffraction and this is actually known as the extinction distance.

This represents the distance over which or after which this condition this relaxation is no longer valid, this extinction condition. So, just like we will not go into details of this xi, what is xi that is not the purpose anyway. So, what we understand here is that this one the maximum intensity of course occurs at 0 at exact Bragg's condition that means when Ewald sphere is touching the reciprocal lattice point that is where we are getting maximum intensity definitely.

But with this intensity has a finite width and we will get if we move on to plus s or minus s direction, plus s and minus s means whether this one is missing it in this direction or this direction that is it that is the plus and minus s. So, if we even go to plus and minus then we are still getting some finite amount of intensity and that is what is causing this finite size for this point the diffracted beams.

Another very important thing that we can understand from here, again derivation I am not going, the width of this main peak, the width at half maxima basically at half height; so the width at half height for this intensity peak is equal to 1 by t and t is the thickness of this specimen. So, this has a big implication on the diffraction pattern that we finally obtain from a real finite crystal this relationship, but that we will be discussing in the next class. And for now, we are stopping it here and goodbye.