Techniques of Material Characterization

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Lecture – 20
Electron Diffraction in TEM

Welcome everyone to this NPTEL online certification course on techniques of materials characterization and we are in fourth week now module 4 and we are discussing about transmission electron microscopy. So, in the previous lectures I talked about mostly how image forms in transmission electron microscopy, what are the sources of contrast, various different type of contrast, atomic number contrast, mass thickness contrast, diffraction contrast etcetera.

And we also discussed about various modes of capturing images in a TEM, bright field mode, dark field mode, HAADF mode and STEM mode and so on and we just started discussing the electron diffraction that is another very important aspect of electron microscopy particularly in transmission electron microscope. We just started about it and there we discussed about the definition of diffraction pattern.

We said it is a special distribution or special arrangements of diffracted beams and then we just discussed about the electron diffraction pattern or intensity distribution from an amorphous material we were trying to understand what is the relationship between the scattering center arrangements and the diffracted beam that is what we will be now continuing.

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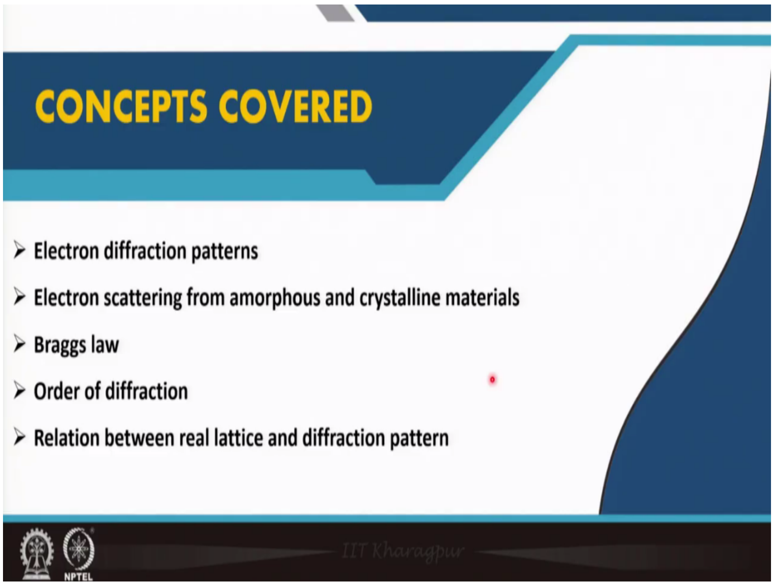

So, in this today's lecture we will be covering electron diffraction patterns more about it. This time it is from the crystalline materials that we will be mainly focus on and then we will discuss about Braggs law and then order of diffraction and then finally we will try to end up with establishing some relationship between real lattice and diffraction pattern.

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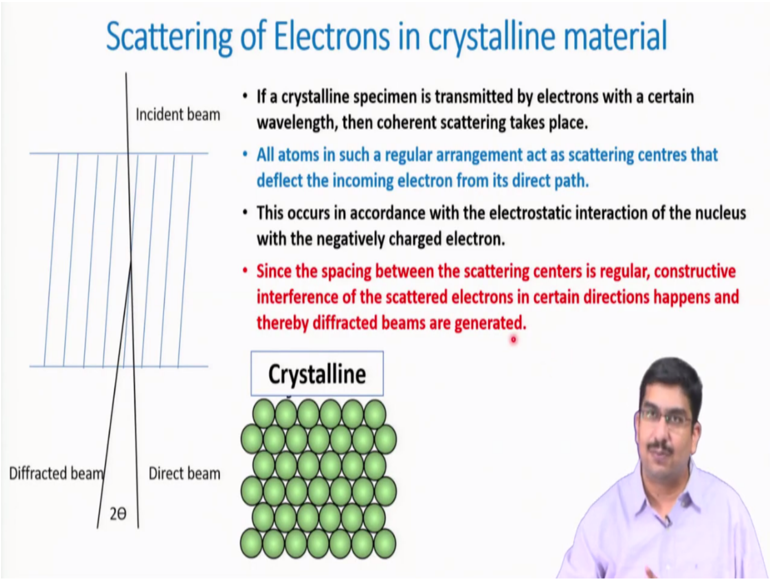

So, we have discussed about amorphous materials and there we saw that like when the scattering centers are all randomly arranged and in that case what happens the chances of constructive interference along certain particular direction is very minimal because the scattering centers itself are very randomly arranged and then they scatter beams in all directions with all possible phases.

So, ultimately those beams even though that is a elastic interaction, elastic scattering even though then also the scattered beams will have different phase and because of that they are constructive interference along one certain particular direction is extremely difficult, but now when we discuss about the crystalline materials. So, crystalline materials in their case the crystal they are all the atoms as you can see all the atoms are arranged in a specific manner.

And I am not going into crystallography and what the definition of lattice and so on, but you know that lattice is defined as a set of points and arrangement where every point has a identical location, identical surroundings that is what main definition of lattice and when it is extended in three dimension it can fill out the space and it can repeat this we can discuss about the repeating units.

That is unit cells and all which will repeat in three dimension and then come all the way to micro scale, but basically in a crystalline material what you can understand is that the symmetry that means each of these points if you look any of these points here all of them are having exactly identical surrounding A and B the distance between them is also exactly the same.

The distance between two atoms is pretty much the same so they are almost touching each other at least in this arrangement. So, this is a particular plane of course in particular atomic plane this is where they are arranged and they are touching each other, but on an average in a crystalline materials the atoms are arranged in a particular way. They are not randomly oriented they are always oriented along certain particular way.

So, now if we imagine that this is the atomic plane so these are the planes which are scattering where these atoms are lying and this planes are basically scattering. So, now I imagine that an electron beam which is coherent in itself so this electron beam is hitting the specimen and then all of this scattering centers are now producing are now deflecting this electrons scattering this electrons in various directions.

But since the scattering center themselves are arranged in a regular manner so what happens is that those electrons along certain direction they will satisfy a phase relation. They will be all in phase definitely in certain directions, they cannot have a complete random scattering in all possible directions that is not possible certain directions definitely because the scattering centers itself are arranged in a particular manner.

So, this in accordance to this arrangement of the scattering centers definitely the scattered beams will be in phase along certain particular directions and that is what this spacing between the scattering centers is regular. So, constructive interference of the scattered electrons will definitely happen along certain directions. So, we have to just see which direction it will happen and how it will lead to the diffraction pattern and this obviously happens because of the elastic scattering.

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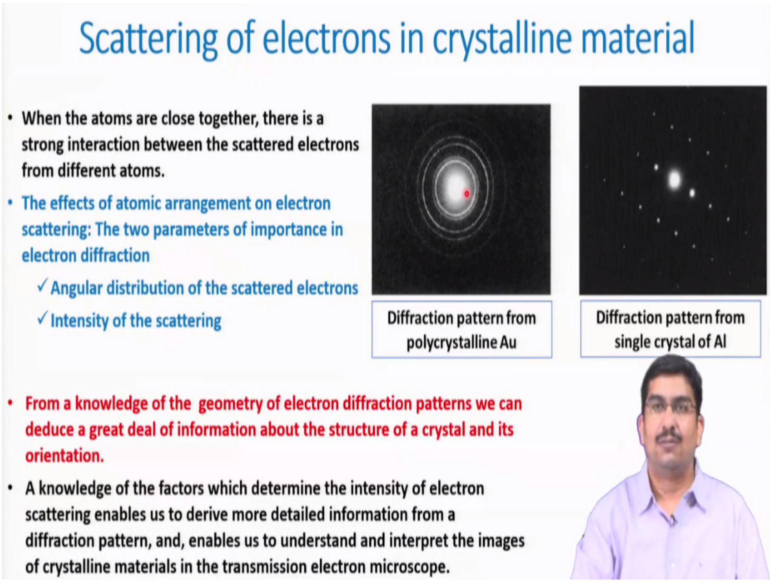

So, the scattering of electrons when this happens the constructive interference along certain direction happens then what it produce is a very regular variation of intensity, we have already seen it for amorphous material even for them the intensity variation with scattering angle they are diffuse maxima and in this case in crystalline material these are much more prominent.

So, there are diffuse maxima definitely and at a certain particular scattering angle that is what the diffraction pattern of any crystalline material should look like. For example if you look at this polycrystalline gold or single crystal aluminum depending on whether they are polycrystalline or single crystalline this nature of or this exact nature of this diffraction pattern will change we will come to that.

But basically what you can see is that you have the direct beam somewhere over here, you have the direct beam somewhere over here and then all other beams here, all other this rings or this spots they are all at a particular distance from this direct beam and also they have certain type of angular relationship with this direct beam. So, these are the two things in there and of course their intensity also changes.

Whether you have this ring pattern, whether you have this spot pattern you can see that the intensity of those beams which are closest to the direct beam these are the most intense one and slowly the intensity is going down as we are moving away from the direct beam. So, these two very important things, characteristics we can make out immediately from any diffraction pattern of a crystalline material.

So, there are two parameters as we said number one the angular distribution of the scattered electrons which electrons these beams are coming. So, the scattered beams along which direction is constructive interference is happening and then as a result we are getting this diffuse maxima in the diffraction pattern. When I capture the diffraction pattern I also discuss how to capture you have to basically image the back focal plane on the objective lens then we can capture the diffraction pattern.

So, number one the angular distribution of the scattered electron which particular angle if they go it will be diffracted constructive interference will happen that and second thing is the obviously the intensity of this scattered beam. What will be the intensity of this diffracted beam in a particular direction. These are the two most important things for the diffraction pattern.

So, if we understand the first that is this angular distribution along which direction this electron beams will undergo diffraction phenomena that means what is the special scattering angle over which constructive interference happens if you know this and as we already discussed that this is related to the arrangement of the scattering centers just this is not now we know that these two theta the scattering angle is a particular angle over which if the electrons are scattering the scattering centers.

If they are scattering those electrons incoming electrons along this particular direction then only the diffraction will happen that scattered constructive interference will happen. So, this is a particular angle and this angle is definitely related to the arrangement of electrons in this atomic planes. So, if we know now we can go reverse, we can do a back calculation, we know this diffraction pattern.

And we can go back and find out what are the arrangement of the scattering centers, how they are arranged along certain particular atomic planes and so on that means we can get idea about the structure of a crystal and of course little extension to that we can get idea about their orientation as well. Second thing we can get from the intensity of this diffraction scattering electron scattering or this diffraction patterns the intensity will tell us again about crystallography.

But little more than that and we can even understand certain image forming contrast forming mechanism just what you understood in the diffraction contrast how they are forming and so on. So, there the intensity will come very handy and that will tell you that exactly where I will get this intensity and how the image is forming because of diffraction contrast and that also will tell you something about the intensity.

We will tell you something about the crystallographic structure as well. So, this is what the importance of diffraction pattern at least for a crystalline materials. This is why we can get the crystallographic structure of any material from this diffraction pattern. If you look at the diffraction pattern we can get an idea about the arrangement of the scattering centers that is this atoms within this crystalline materials.

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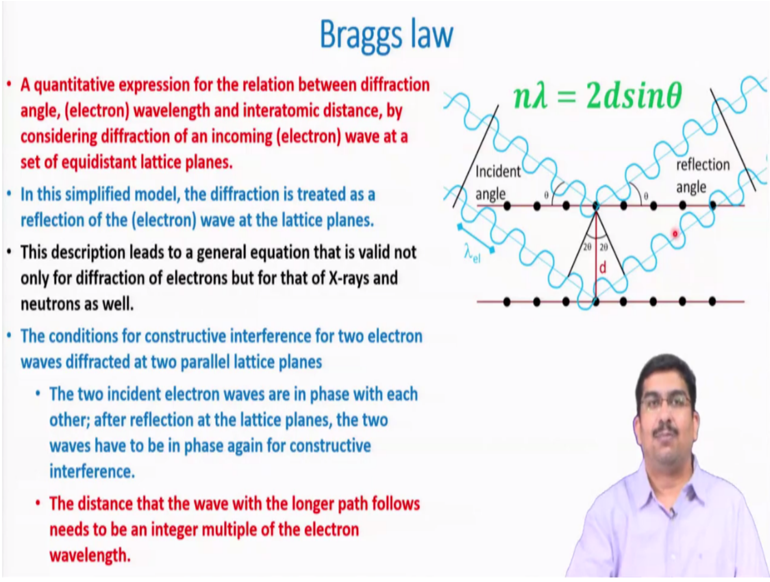

Now the more celebrated part of diffraction 9 out of 10 students of my class if we ask them what is your idea about diffraction the first thing they tells is Braggs law very good no harm in there. Even many people many of you who are listening to this lecture you will also say that diffraction oh that means a Braggs law, but exactly we have to understand the diffraction is beyond Braggs law.

It starts before Braggs law and it can go even further after Braggs law that is what we will be discussing and now you understood that where from this Braggs law basically is coming, the diffraction pattern is related to the arrangement of crystals in the material so that is how the Braggs law from there the Braggs law started. Now what this Braggs law is doing and the absolute form of the Braggs law everyone knows n lambda equals 2 d sin theta.

And this is a very simple equation very powerful and it is most powerful because of its very general nature. So, what does this equation do? This is basically relating the diffraction angle that is this angle theta which is telling us that exactly which angle this incident electron beam is coming and exactly which angle the scattering is happening. So, what is the special direction where this constructive interference will happen.

So this tells this theta so this is related to the incident electron beam and the detection signal that is the signal that is generated that scattered beam that is generated that is all. The second thing is the wavelength. Wavelength is again a characteristics of the source signal itself, the source signal at which wavelength the source signal is coming. So, it is characteristics of this one.

And third thing it has this d which is interplanar spacing that means this one or in some case the interplanar spacing and interatomic distance all of this are related. So, this d is basically telling us about the crystallography of this material, the information about the atomic arrangement in this material. So, the simple one lines very small equation is basically relating the source signal, the detection signal and the crystallography of the material.

So, it is explaining, it is describing the entire diffraction phenomena that is why it is very, very powerful and very effective and very general this equation is not only valid for electron diffraction it is valid for any other kind of diffraction like x-ray diffraction like neutron diffraction and any other electromagnetic wave all you need electromagnetic wave with a particular lambda coming at a particular angle.

And then the crystalline material you can apply this Braggs law. So, if one day if we are able to apply let us say gamma ray or some other electromagnetic radiation and we are having instrumentation to capture their diffraction pattern then definitely there also the Braggs law will be valid that is the power of this equation and that is why it is one of the most celebrated equation in the field of science.

And now we can prove this one all of you know how to prove this I am not going it is very much text book stuff. Basically what happens is that this electron beam is coming we are considering two different plane of atoms and relation that is needed or the condition that needs to be satisfied for a constructive interference between these two electrons basically what it has to do is that electrons are all in phase when they are coming in phase.

So, when they are going or following in this particular scattering direction they has to be again in phase and for that this one has to have wavelength which is a integer multiple of the electron wavelength and then from that condition geometrically from this angle or from this triangle you can geometrically prove this relation that is all about Braggs law.

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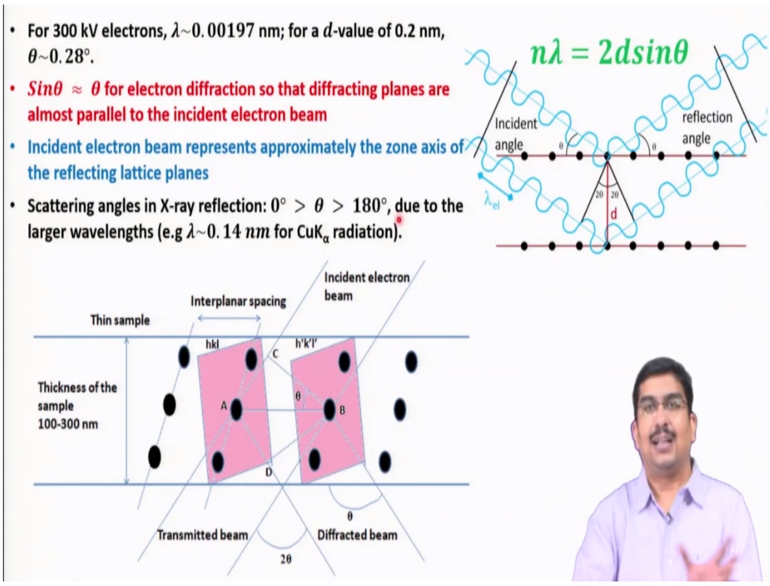

Now let us put some typical numbers. Let us throw some typical numbers in this Braggs law and try to find out what is so special about electron diffraction, how this equation gives us an idea about electron diffraction. So, for let us say a 300 kV electron which is very typical that we use for a TEM instrument. Lambda we know the relationship between accelerating voltage and wavelength.

So from there we can very easily calculate lambda we are getting around this value 0.00197. You can roughly take 0.002 nanometer d value is around for any typical materials d value is 0.2 nanometer that is 2 angstrom which is almost atomic separation distance (15:25). When we put all of this here what happens that the theta turns out to be around 0.028 degree which is very, very small.

Now what does this mean? This means two things number one sin theta comes down to theta for electron diffraction. So, we can safety say this one is lambda equal n lambda equals 2 d theta basically this Braggs law comes down to that is one, but what more it says is this beam is now coming at an incident angle very, very small that means the beam is almost parallel to this plane of atom.

And after diffraction also since these two angles are same it is also going at an angle which is almost parallel to this plane of atoms and this is what it is showing here that this incident electron beam when it is coming from two different planes this planes are almost parallel to the direction of the electron, incident electron beam that is what the specialty of electron diffraction.

And where it is different from x-ray diffraction because if we put some typical numbers again for x-ray diffraction like lambda equals 0.14 nanometer for copper K alpha radiation we get this theta angle now lying within 0 to 180 it is not as small as electron diffraction. It is typically they are the electrons what happens is typically in case of x-ray diffraction the planes which are parallel to the surface they diffract.

And in case of electron diffraction the planes which are perpendicular to the surface of the specimen they diffract that is what the major difference between x-ray diffraction and electron diffraction and there are other differences also, but at this point this is enough for us to remember. So, basically now the message is that electron beam if it is coming parallel or if it is coming perpendicular to the specimen.

So, if I imagine that this electron beam is coming perpendicular to this specimen then this atomic planes which are perpendicular to the surface that those planes perpendicular to the surface means in this case now they become parallel to the incident electron beam. So, electron beam is coming this way so maybe we can go back to this and this tell us much better.

So, electron beam is coming perpendicular to the surface which is specimen surface which is basically perpendicular to this plane to the screen and this incident electron beam is coming here and this atomic planes is parallel to the incident electron beam and in the process they are actually perpendicular to the surface of the specimen that is the specialty in electron diffraction.

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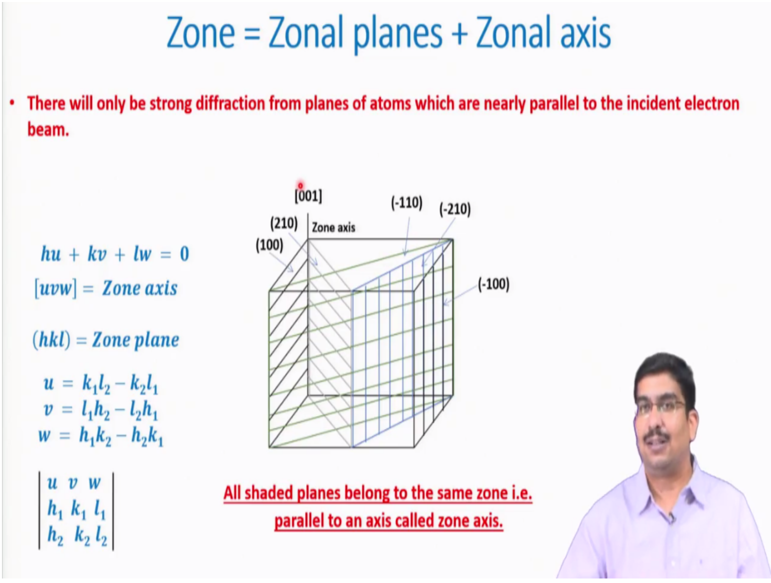

Now we have to learn another quick information from crystallography and that is the concept of zone axis what is the zone axis. So, if we imagine cubic system then this zone axis is basically the common direction which is lying on a family of planes (18:22)or different family of planes. So, if I imagine let us say 001 direction in a cubic system the 001 direction is contained by various set of planes 210, 100 planes bar 110 planes, 2 bar 10 all of them are basically parallel to this direction that means all of this set of planes are having or containing this direction.

This direction is lying on each of this planes and the condition for a cubic system again the condition is for one direction to lie on a plane is this it has to satisfy this basically cos theta has to be 0 so it has to satisfy this and then uvw is the zone axis and we can find out from two sets of planes, we can easily find out the uvw the zone axis for them from this relationship. Now what is this so special about the zone axis?

Zone axis is special because this if we can imagine now this electron is coming along this zone axis. So, if the electron is coming along 001 direction then all of this planes which are contained within the zone that means the zonal planes, the planes which are parallel to this direction they will diffract that is the importance of this zone axis that means incident electron beam is not arbitrary or neither the crystals or crystallographic planes which are doing or which are satisfying the diffraction condition either they are arbitrary.

That means if the zone axis if the electrons are coming along certain zone axis then all the planes which contains the zones they will diffract. So that is what the importance of zone axis in the case of electron diffraction and from this relationship quickly immediately we can understand or we can what is the zone axis if we know the zone axis we can find out the plane if we know the planes we can find out the zone axis because that is how they are related.

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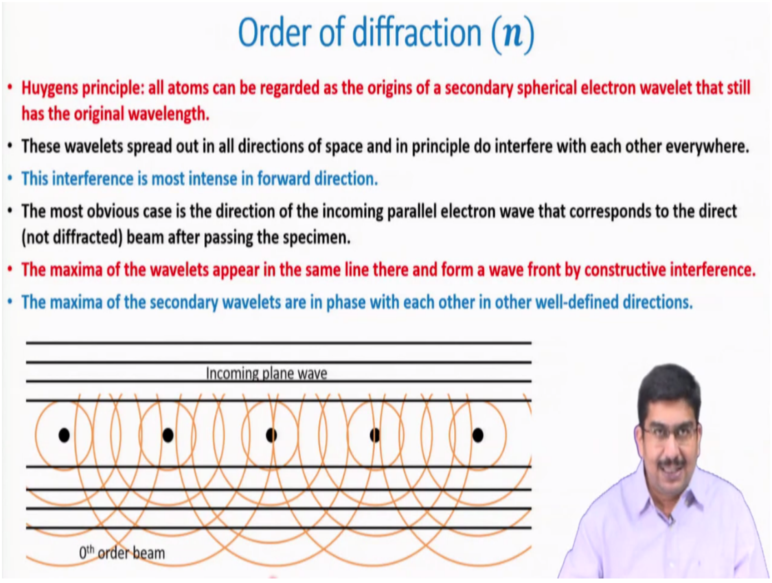

Now another thing that we need to understand here is the order of diffraction we will go one by one. So, this order of diffraction is basically that n in the Braggs now n lambda equals 2 d sin theta so what is that n here. In order to understand that I found there are many ways we can understand, I found the best way to understand this is by going this one called Huygens principle.

And Huygens principle is basically it is very valid it is given for optical microscope it is given for light diffraction, but we can very safely use it for any other kind of diffraction like electron diffraction here. What does it say is that you have this incoming plane wave with a plane wave front and after it gets scattered by this atoms. So, this black dots here are the atoms which are regularly arranged.

We are just considering one set of atom for simplicity they will generate some secondary wavelets and those secondary wavelets are now shown in this as a circle concentric circles those secondary wavelength. So, basically this is the wavelength for them this distance is basically the wavelength for this secondary wavelength and remember since this is an elastic interaction the wavelength does not change.

So, it has the same distance this concentric circle the radius is increasing in the same way as this incoming plane wave. So, this is what is signifying their wavelength and this lines are basically the maxima in those waves. So, secondary waves that is generated those are shown by this concentric this lines and they have exactly the same distance same radius I mean radius is increasing in the same way that is the wavelength.

This secondary wavelets now they are spread in all direction in space in three dimension and if we take a section along this plane waves out as a line and this comes out to be some concentric circles that is it. This secondary wavelets obviously will undergo some kind of a interference and that interference will be most intense in the forward direction that means this interference they will have the interference.

So, this incoming plane wave will of course some part of it will just move through that is forming the 0th order beam and this secondary wavelets will also have a interference with the primary this 0th order beam and that will be the strongest one. So, there will be a constructive interference along the direction of the 0th order beam that is the first thing then also along certain other well defined directions the maxima of the secondary wavelets will be in phase.

That means the maxima of this atom, maxima means this line basically here the maxima of this atom the secondary wavelets generated by this atom maxima of secondary wavelets generated by this atom this atom, this atom all of them will coincide along certain other direction other than this 0th order beam other than this direction this direction of the plane wave propagation.

Other than this some other directions they will also have this interference or they will also overlap and those are the direction where constructive interference will happen that means those are the direction where diffraction will happen.

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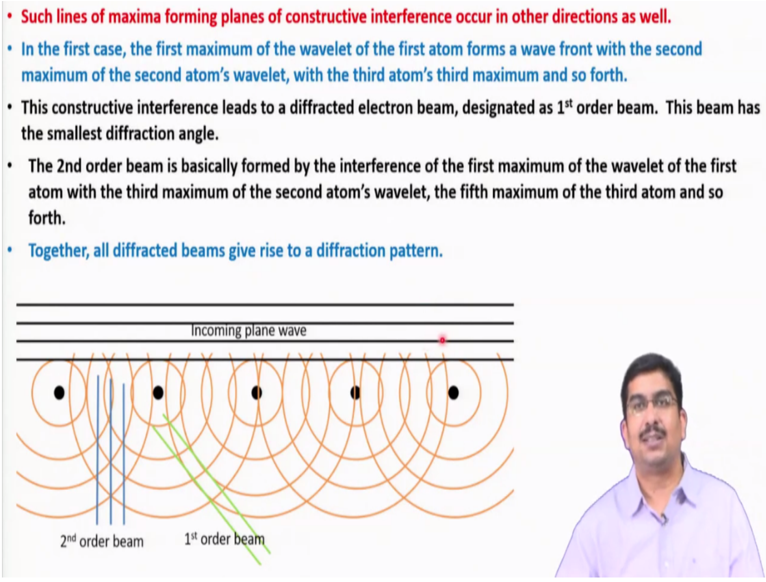

So, now let us see this. So, in the first case what we can imagine is that along this direction the one given with this green color. So, in that direction what is happening is that the first maxima from the first atom the first maxima of first atom is coinciding with the second maxima of the second atom and the third maximum of the third atom and so on and so forth. So, if we imagine this is the first one.

So, then we can imagine that along this direction the first maxima of the first atom coinciding or superimposing or overlapping with the second maximum of the second atom means they are undergoing constructive interference along those directions and then with the third maximum the third atom and so on and so forth that means this is the direction again constructive interference is happening.

Other than this incoming plane wave this direction 0th order beam other than that this direction also again I am getting a constructive interference between the secondary wavelets means between the scattered beams. This is the direction for first order beam when the first maxima of first atom and second maxima of second atom, third maxima of third atom is undergoing constructive interference that is forming the first order beam.

Now, similarly we can find out the second order beam will now be given by the interference of first maxima of the first two atom with the third maxima of the second atom with the fifth maxima of the third atom so on and so forth. You can try to construct this all by yourself this will be a nice task you can do this and go ahead and check out that how the first maxima is forming.

How the second maxima is forming or second order beam is forming and so on and you can move on to other as well with different, different maxima and different, different order of beams here.

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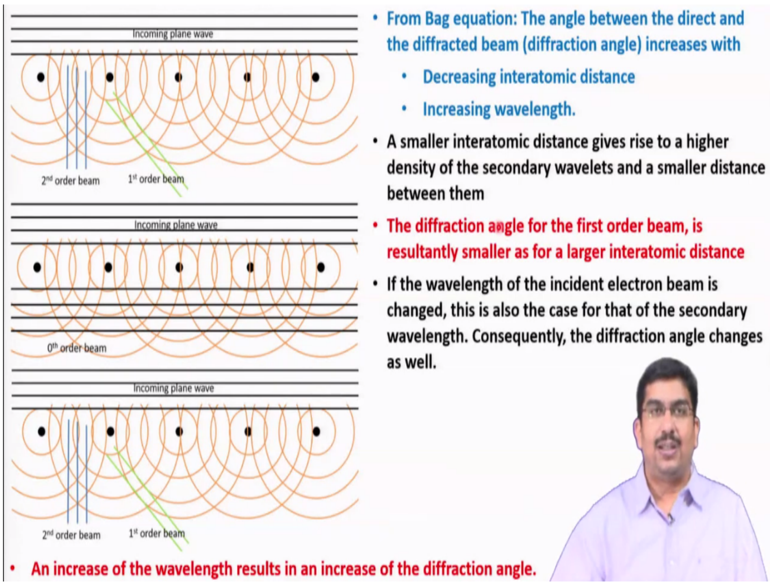

Now let us see some very quick and important observations here from this secondary wavelets and all. So, what we are having is now this from Braggs equation basically from Braggs equation n lambda equals 2 d sin theta from that equation we can find out few relationship between lambda d and sin theta. So, the first one what we can see is that if decreasing the interatomic distance means d and what is the relationship between d and theta.

Keeping lambda constant the relationship between d and theta that is obviously is an inverse relationship and we are just considering the first order beam so n is 1, lambda is constant d and theta is having an inverse relationship that is that means the theta will increase the angle for the diffracted beam the angle between the diffracted beam and direct beam will increase as the interatomic distance is increasing.

Second one relationship is again n is 1 constant between lambda and theta now d is constant this is the direct relationship if lambda increases theta increases direct relationship. Now, this is what again we can prove it from this construction this Huygens construction what happens is now the smaller interatomic distance means the density of the secondary wavelets is increasing.

And their radius the distance between this maxima the radius of this maxima is also decreasing. So, decreasing the interatomic distance means decreasing d means we are trying to bring now this atoms closer together and that will increase the density of the secondary wavelets and obviously then what will happen is that the angle you can try to do that you can make this construction you will see that compared to the initial case now what we will be having this is kind of an exercise you can try to do it.

Compared to this what will happen the angle between the direct beam and this diffracted beam will now decrease. So, this one will be now more skewed in this direction. Second thing if the wavelength of this incident electron beam is changing basically that means that the secondary wavelength the radius will change again. So, the incident electron beam the wavelength is now we are increasing then this one also we are increasing.

This incident beam this one also will be increasing that means here same thing will happen this will increase the diffracted beam the angle between this diffracted beam and direct beam and vice-versa. So this entire relationship from the Braggs angle you can determine from this Huygens principle and from this construction itself.

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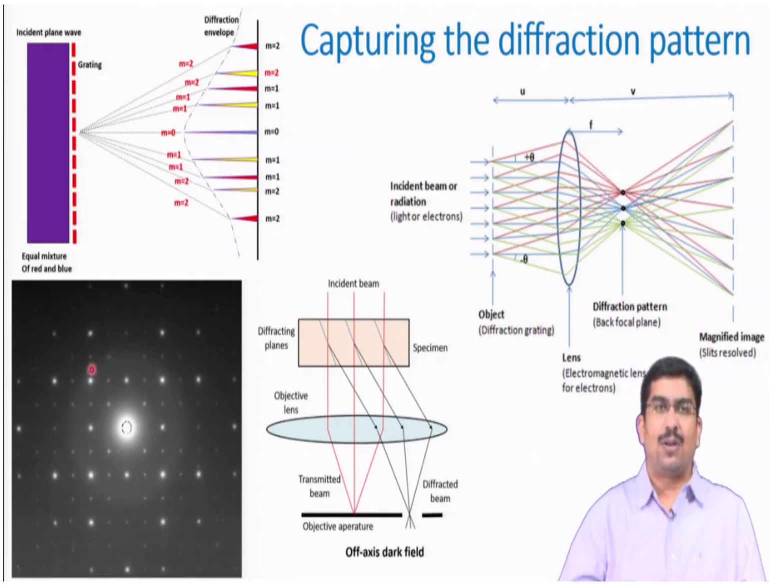

Now the capturing of the diffraction pattern we have already discussed about this that this discuss this one. We can capture this if this is the diffraction the initial schematic so we have the diffraction gratings and we have the 0th order beam and then first order beam, second order beam and so on. Generally, what we do is that we capture the 0th order beam that is a direct beam and one of the diffracted beam usually the first order diffracted beam that is what we capture using some kind of an aperture.

So, you can imagine that again the same dark field what we understood for the dark field mode imaging in the dark field mode we can bring an aperture and now we are capturing the direct beam plus this first order diffracted beam by putting that aperture in the back focal plane and then we can get this direct beam here and all other first order diffracted beams from various atomic planes.

So, this is one diffraction pattern from one diffraction grating you can imagine that this is possibly happening along one atomic plane, but now we understood that electron beams are coming along the zone axis. So there are more than one such atomic planes which are satisfying the diffraction condition and which are diffracting. So, this is basically the diffraction pattern is coming out this is the first order diffraction pattern if you capture the first order diffracted beam from various atomic planes.

And they are obviously arranged in a manner which represent their arrangement crystallographic arrangement in the real specimen that is what is the diffraction pattern and we will discuss more about the diffraction pattern in later classes how to read them, how to index them and so on and so forth and finally we have a relation between the real lattice and the diffraction lattice, but this we will be discussing in the next class. So, we are stopping it here and thank you and welcome.