

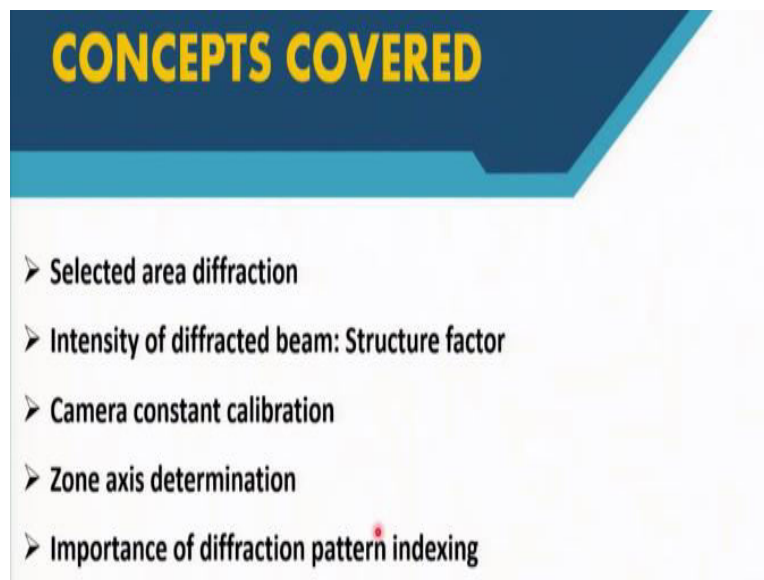
Techniques of Materials Characterization
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Lecture – 23
Electron Diffraction in TEM - Continued

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. So, we are in module 5 that is in fifth week and we are discussing about transmission electron microscopy. And in transmission electron microscopy we have discussed about image formation and then we started discussing about electron diffraction. And in electron diffraction in the last class, we saw like how it differs between a single crystal, polycrystal.

And something if it is intermediate between a single crystalline and polycrystalline how the electron diffraction pattern varies. And then we also discussed various other concepts related to electron diffraction reciprocal lattice, Ewald sphere construction, how the Ewald sphere construction is important to identify several features in diffraction pattern from finite crystals so on and so forth. So, we will continue that discussion today also.

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And we will be discussing here now one very important technique of getting electron diffraction pattern that is called selected area diffraction and then we will discuss about the intensity of diffracted beam. So, after selected area diffraction, basically we will be trying to understand the diffraction pattern more and finally possibly in the next class we


will discuss how to index the diffraction pattern or how to get the full meaning out of that electron diffraction pattern.

So, in order to get the full meaning out of electron diffraction pattern, we have to understand couple of these issues, couple of these points. That is first of all the intensity of diffraction beam that is structure factor. We will see the importance of structure factor and then camera constant. We discussed briefly about camera constant, now, we will see how the camera constant calibration happens, how the camera constant determination occurs.

Then how the zone axis determination is done. And finally, we will try to discuss about the importance of diffraction pattern indexing.

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Selected area diffraction (SAD)




SADP of a single austenite crystal in a piece of steel

- Because the wavelength of high-energy electrons is a few thousandths of a nanometer, and the spacing between atoms in a solid is about a hundred times larger, the electrons are diffracted, and the atoms act as a diffraction grating.
- A fraction of the electrons will be scattered to particular angles determined by the crystal structure of the sample, while others pass through the sample without deflection.
- The resulting TEM image will be a series of spots, constituting the selected area diffraction pattern (SADP).

Each spot corresponds to a satisfied diffraction condition according to the crystal structure.

If the sample is tilted, different diffraction conditions will be satisfied, and different diffraction spots will appear.



So, selected area diffraction is basically a diffraction pattern only, just a diffraction pattern like the way we used to see. So, we know that wavelength of a high energy electron is few thousandths of a nanometer, it is very small and the spacing of the atoms usually are much larger that is much more orders of magnitude larger than the wavelength of the electron.

And they are very much regularly arranged. So those electrons get diffracted from this scattering center. So, from these atoms which are acting as a scattering center and because of their regular arrangement, those scattering happens at certain particular

directions, certain particular scattering angle, those scattered or diffracted scattered beams they undergo a constructive interference that gives rise to diffraction.

And when we capture this diffracted beam along with the direct beam, we captured these diffracted beams and there are different first order, second order, we know all this order of diffraction and all. Let us say we capture only the first order diffraction beams from various atomic planes, then what we get is basically a diffraction pattern. Now, what is special about selected area diffraction pattern we will come to that.

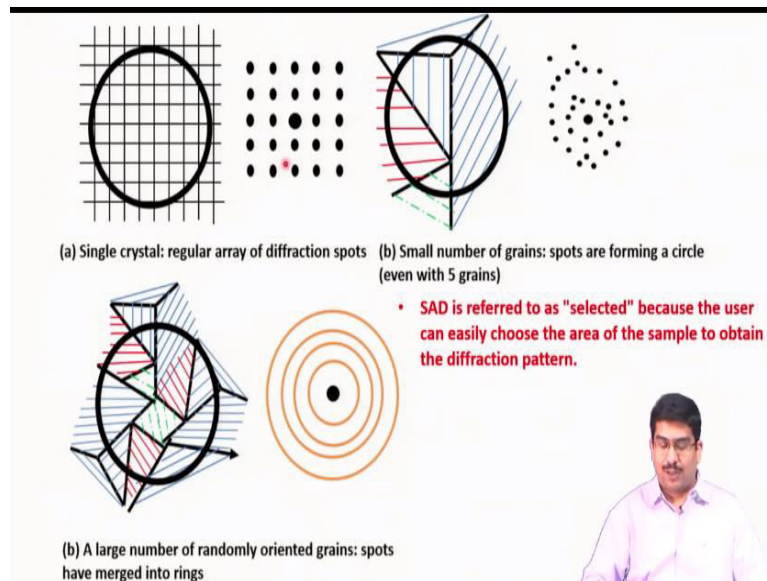
But basically, this also we did that each of these spots is a diffracted beam and it denotes the particular diffraction angle or particular atomic plane from which a satisfied diffraction condition happens or diffraction condition gets satisfied according to the crystal structure. So, if we tilt this electron, basically the sample, the specimen is tilted, this is another way of saying that we are changing the zone axis.

So, we instead of tilting the beam itself or let the beam come from a different zone axis because you know in electron diffraction, the atomic planes which are parallel to the beam that means they are diffracting that means the electron beam comes along the zone axis for that particular set of planes which are diffracting. So, if we tilt the specimen that also means that we are going to another zone axis.

And then the entire diffraction pattern will change and now those planes which are parallel to that particular change modified zone axis they will now satisfy diffraction condition and we will get a completely different diffraction pattern. So, this diffraction pattern if you look at here, this is from iron, and this different fraction pattern basically is a single crystal. Now, we understand that this is a spot pattern and this belongs to a single crystal right.

But actually, this diffraction pattern is captured from a polycrystalline specimen. Now how we are getting a diffraction pattern which is similar to the characteristics of a single crystal diffraction pattern; we are getting it from a polycrystal. Ideally if we capture the polycrystalline diffraction pattern, we should get a ring, but in this case we are getting a spot pattern even from a polycrystalline metal.

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So, in order to explain this, normally my students always get confused with this, I try to give them the same schematic which I use to explain the difference between single crystalline diffraction pattern and polycrystal and diffraction pattern, basically spot patterns, spotty pattern, ring pattern and so on. So, if you remember the spot pattern for that I said that you have this, let us say this is your area that you are scanning basically okay.

So, this is the area from which you are capturing this direct beam and diffracted beams or rather you can also imagine that this is something like an aperture and that aperture this is the size of the aperture. And in regular case when we have a single crystal the single crystal is much larger than this aperture hole or the size from which we are capturing this or we are imaging that diffracted beam and direct beam.

And then within the same size if we now have this many crystallites, multiple number of crystals, not a single crystal but many number of crystals or crystallites or grains then we get a spotty pattern. If we have numerous such crystals then we get a ring pattern. In entire process we are having two different entities, one is the microstructure or the entity that is diffracting so that is changing, in this case that is changing from single crystal to certain some number of crystals to complete polycrystalline numerous crystals.

But we are keeping, what we are keeping is the area which we are analyzing from which we are capturing this diffraction pattern. That is what regular in a normal case we do. Now, what we can also do is something called selected area diffraction and as the name

suggests this term selected is given because here we choose an area of the sample to get the diffraction pattern and that is it.

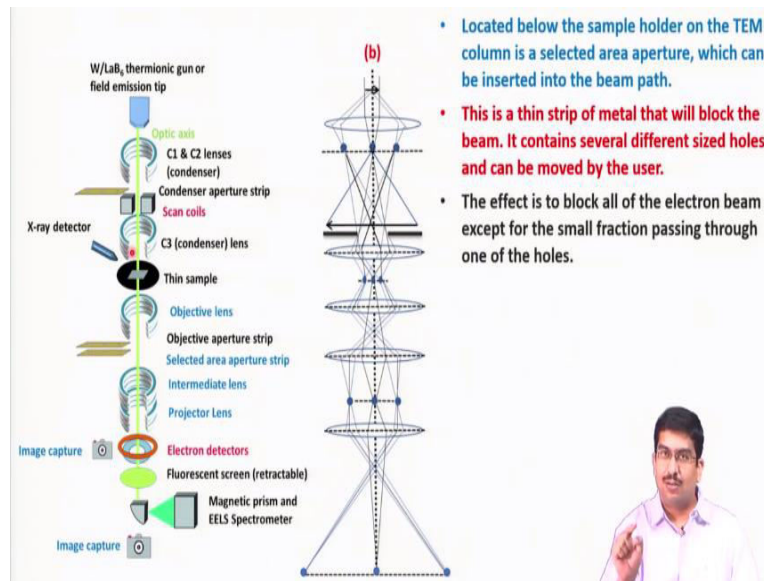
So, it is a polycrystalline material, what now we are doing is we are shortening or we are reducing the size of this area from which we are capturing the diffraction pattern or in other way around we are reducing the size of the aperture. So, we are blocking the beam, all other beams and just focusing, just allowing that part of beam which is coming essentially from one single grain or single crystallite from a polycrystalline material.

So, essentially what happens is that that if we just increase this aperture, our microstructure remains the same. So, this is the microstructure, just understand this different. This is the microstructure it remains the same. All we are doing is now reducing the size of this black ring that is the area we are interested or that is the area from which we are capturing this diffraction pattern that is it.

And we are making it so small, let us say it is becoming so, so small that we are essentially within this one single grain and since we are within one single grain, and every grain, but the way grains are defined is that where there the orientation is the same and orientation only changes in the grain boundary. So, this is one type of orientation, this is another type of orientation with respect to each other, but inside the grain it is essentially a single crystal.

So, that means here we are shortening, we are reducing the size of this ring and in that process we are essentially scanning or we are essentially getting or probing one single grain and that is why we are getting a spot pattern which is characteristics of one single grain. So, this selected area diffraction pattern basically allows us to capture orientation of one single entity in a polycrystalline material, orientation of one single grain on a polycrystalline material in the form of an ellipse spot pattern, spot electron diffraction pattern.

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Now, what do we need to do? Basically, we do not need to do anything. This is there in most of the microscopes. Most of the transmission electron microscope it already is there, this flexibility that you are capturing this form of polycrystalline material, you are capturing a single crystal diffraction, you are just selecting a very small area within that polycrystalline material. And for that what you have? This is again the same schematic of a regular TEM.

What you have is that there is an aperture. Just after the objective lens you have this selected area aperture strip. So, this aperture basically this is located below the specimen holder and sometimes below the objective lens as well and this is a thin strip of metal with different holes. So, there are different size of the hole, and different size of the hole is basically in order to reduce sufficiently this and that different size of hole basically corresponds to the size of the grain.

If your grains are too small, you will be using aperture hole size which is very small and as the grain size is increasing you can possibly use a bigger hole in the aperture strip, your selected area aperture strip. So, what essentially it will do is that it will block all the other grains, all the other part of the diffracted beam which are coming from other grains, it will block all of these and it will just allow the direct beam and the diffracted beams that is coming out of one single grain.

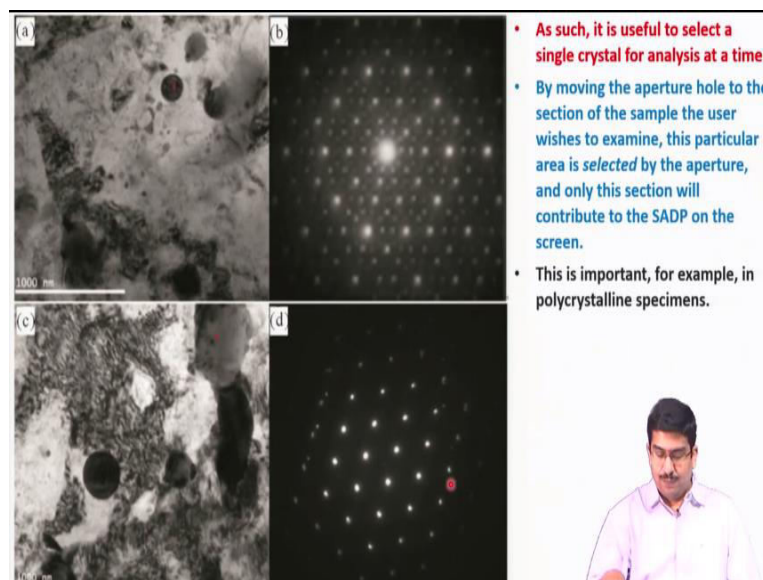
So that is what it will do and it will do right here and after that only that much area or only that much electron beam will be able to reach to the fluorescence screen and

correspondingly it will be magnified afterwards by the intermediate lens and projector lens and all. And finally in the screen you will be seeing a spot diffraction pattern from an area you have selected on your specimen here it is.

So that is how you do basically this selected area diffraction patterns you capture here. Rest of the things remains exactly the same, just this by changing the selected area diffraction by inserting this aperture, selected area diffraction aperture and by using different holes, different size of this aperture you are selecting different regions in the entire specimen. The entire specimen is diffracting.

Direct beam is coming from the entire specimen, you are just now selecting a part of the specimen, you are selecting it from the entire beam, you are selecting only a certain part of it. And that is how basically finally you are getting a diffraction pattern corresponding to that part.

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So if we see certain examples, it will be much more clearer. So, look at this microstructure, in this microstructure now you can understand that it has different entities, may not be a grain, but let us say this is a completely different phase altogether. Looks like it is a completely different phase as compared to these entire other matrices this is some kind of a precipitate some other entity.

So, if you take from the entire region, now if you take a diffraction pattern this looks like to be a very complex one. A very complex one because now even in this diffraction

pattern, you can imagine that there are many, at least two diffraction patterns which are superimposed from these matrices which may be same orientation or may not be same orientation and this diffraction, this other phase.

I am calling it other phase because its chemical identity is determined to be completely different from this. So that is why we will come to that, first you have to determine the chemical identity in order to understand that this is a completely different phase and then you can possibly try to analyze its diffraction pattern selected area diffraction pattern. So, ultimately what happens is that this diffraction pattern here it is very complex, solving this is very difficult.

Plus what happens is you are interested to know whether this is a complete different phase or not or if it is a different phase as I said you can do compositional analysis and try to find out the chemical identity of that and if you do then you are possibly interested to know exactly what phase it is, what is its orientation and something that we will discuss the orientation relationship as well with the matrix.

What you can do now you can just choose a selected area from this microstructure, let us say this is your selected area. This precipitate or this screen that is your selected area and you are just capturing and this is something like you are just within this and you are allowing the beam that is coming out of this area to finally reach to your fluorescent screen. So ultimately, the diffraction pattern that you are getting here is entirely coming out of this phase or this green from here.

So, this is a selected area diffraction pattern and this is how it will be. You are not capturing the diffraction pattern from this entire region. You are just capturing the diffraction pattern only from this region that is why the diffraction pattern looks like it is from one single phase. So that is the importance of doing a selected area diffraction pattern.

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Spot diffraction pattern of forged Ti-6Al-4V alloy. Two phases are observed in the pattern, namely, α and β with $Z = [1\bar{2}1\bar{6}]$ and $Z = [012]$, respectively.

- If more than one crystal contributes to the SADP, it can be difficult or impossible to analyze.
- It may also be useful to select two crystals at a time, in order to examine the crystallographic orientation between them.
- As a diffraction technique, SAD can be used to identify crystal structures and examine crystal defects.
- It is similar to X-ray diffraction, but unique in that, areas as small as several hundred nanometers in size can be examined, whereas X-ray diffraction typically samples areas several centimeters in size.



So, there are many things that you can do from selected area diffraction pattern. I am just showing you one single example for like you have this kind of diffraction pattern and if you solve this diffraction pattern, there are two different phases. So, this selective area diffraction pattern is taken from the interface of two different phases. So, this is a titanium 6 aluminum 4 vanadium alloy, which is a two phase alloy.

Which has one alpha phase hexagonal close packed hcp phase and the beta phase which is body centered cubic phase and the zone axis is such that it is for this alpha phase it is this zone axis well for beta phase it is this zone axis. And then from the interface region when you have taken the selected area diffraction then in the diffraction pattern, you will see that the spots are coming corresponding to both the phases.

And if you solve this diffraction pattern in terms of both phases, what you can finally get is an orientation relationship between these two phases. So, either you can take a selected area diffraction pattern individually from these two phases or you can choose the interface region, very selectively you can choose the interface region where you purposefully make both the phases to contribute to the final diffraction pattern on their individual spots.

And from solving that indexing that you can get the orientation relationship between these two phases here. So, this is how some nice studies you can possibly do with this selected area diffraction pattern by capturing selected area diffraction pattern. In some sense it is very similar to X-ray diffraction pattern, but in X-ray diffraction basically

your area that you probe is much bigger than this selected area diffraction the area that you broke in a selected.

So, in X-ray diffraction also possibly you can get diffraction pattern from a polycrystalline material, but if you want to see basically the area that is probed in an X-ray diffraction scan is much bigger than in electron microscopy in selected area diffraction. So, in case you want to determine the structure of any material, then possibly in X-ray diffraction pattern you need to have a single crystal, real single crystal.

While in electron diffraction the advantage is that even in a polycrystalline material you can just utilize the selected area diffraction and you can get the structure of that material, even when it is inside a polycrystalline material. So, that is a very great advantage of this method.

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Intensity of diffracted beam: Structure factor

- Mathematical description of how a material scatters incident radiation.
- Each type of plane in a crystal will have a different spacing and a different density of atoms per unit area, and so we may expect that the intensity of electron diffraction will be different for each type of atomic plane.
- It is obtained by adding up the contribution to electron scattering made by each atom on a given plane taking into account the phase of each wave that is scattered.

Real space
Direct space
Crystals

3D lattice in direct space

Reciprocal space

Diffraction pattern
3D lattice in reciprocal space

$$\rho(xyz) = \frac{1}{V} \sum_{hkl} (|F(hkl)| \cdot e^{-2\pi i(hx+ky+lz-\theta(hkl))})$$

$$F(hkl) = \sum_j f_j e^{2\pi i(hx_j+ky_j+lz_j)}$$

$$\rho(xyz) = \frac{1}{V} \sum_{hkl} \left(|F(hkl)| \cdot e^{2\pi i(hx+ky+lz-\theta(hkl))} \right)$$

Structure = Amplitude Factors and phases

Amplitudes Phases

Real space Direct space Reciprocal space

Now, we will be discussing about another aspect before we go and try to index this diffraction patterns and get more meaning out of it. This is also another very important factor in diffraction. We will have this intensity of diffracted beam and something that is called structure factor which determines the intensity of the diffracted beam that is coming out of a complete atomic plane.

So, we will have a much deeper discussion about this structure factor, intensity of diffracted beam and structure factor, how it comes and what is the form of it, how it gets changed for different type of crystal structure and so on. So, this entire discussion we

will do and we will take some few examples, we will try to solve them and try to calculate the structure factor and so on.

We will do all of these when we discuss about x-ray diffraction. For now, we will just give a very brief overview about the structure factor, we will not go into details of it, but it will be very helpful to understand the diffraction patterns in general. So, basically, structure factor is a mathematical description of how materials scatter in the incident radiation or rather what will be the intensity of the diffracted beam and you can calculate that from this structure factor of various atomic planes.

Basically, what happens is first you take the intensity of diffracted beam from or intensity of the wave. So, you have this incident electron beam that comes like a wave and after it gets scattered by a single electron, how the wave that is generated, what is the intensity, what is the phase of that beam. Then what you do is that you take a complete atom which consists of many such electrons.

So, you basically take their phase relationship and then you add them, along certain particular direction you add them and find out a relationship, what should be the intensity or amplitude of this resultant wave from one single atom. And finally, what you do is that you take a complete atomic plane, add up these waves which are now coming from individual atoms with a certain phase relationship.

So, you take those atoms on a complete atomic plane, sum them up and find out the intensity of the diffracted beam from one single atom scattered it up. And this is very important because in different atomic planes the density of atoms is not same. So, I hope everybody who are attending this course is aware of crystallography. So, you have atomic density, you know if you take a cubic crystal for example, you have this close packed planes and close packed planes means the atomic density is highest on that plane.

So, if you take for example a BCC crystal then it will be 110 planes which is the highest density. If you take FCC crystal it will be 111 planes which has the highest atomic density. So, that means even within the low index planes 100, 110, 111 even between them their atomic density changes. So, they do not have the same number of atoms. So, ultimately what will happen?

The diffracted beam which is coming out of these different planes will have different intensity and the structure factor for these different planes that will basically denote this difference of atomic density and the final what is the difference between the intensity of the diffracted beam from these different atomic planes. So, the general form of this structure factor or the intensity of the diffracted beam that is basically this.

So, you have a summation of all the atoms present on different atomic planes and then you have the summation in terms of the amplitudes and the summation in terms of their different phases. And these phases are expressed in terms of their Miller indices. If we are just considering this to be a cubic system, so then this Miller indices will tell you that what is the phase relationship between this, finally intensity of the diffracted beam and from there you can calculate what is the intensity.

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- In some cases, the individual terms can sum to zero, i.e. the planes give rise to no diffracted beam.
- These are known as forbidden reflection.
- Structure factor and Bragg's law together decides the nature of the diffracted beam.

Face centered cubic (FCC)

$$F_K = f \left[e^{-iK \cdot 0} + e^{-iK \cdot \left(\frac{a}{2}\right)(x+y)} + e^{-iK \cdot \left(\frac{a}{2}\right)(y+z)} + e^{-iK \cdot \left(\frac{a}{2}\right)(x+z)} \right]$$

$$= f [1 + (-1)^{h+k} + (-1)^{k+l} + (-1)^{h+l}]$$

$$F_{hkl} = \begin{cases} 4f, & h, k, l \text{ all even or all odd} \\ 0, & h, k, l \text{ mixed parity} \end{cases}$$

Body centered cubic (BCC)

$$F_K = f \left[e^{-iK \cdot 0} + e^{-iK \cdot \left(\frac{a}{2}\right)(x+y+z)} \right]$$

$$= f [1 + e^{iK \cdot \left(\frac{a}{2}\right)(h+k+l)}]$$

$$= f [1 + e^{i\pi(h+k+l)}]$$

$$= f [1 + (-1)^{h+k+l}]$$

$$F_{hkl} = \begin{cases} 2f, & h+k+l \text{ even} \\ 0, & h+k+l \text{ odd} \end{cases}$$

First 12 allowed reflections in the fcc crystal structure and their interplanar spacings

Indices	$h^2+k^2+l^2$	d/a	Indices	$h^2+k^2+l^2$	d/a	Indices	$h^2+k^2+l^2$	d/a
111	3	$1/\sqrt{3}$	222	12	$1/3\sqrt{2}$	440	32	$1/4\sqrt{2}$
200	4	$1/2$	331	19	$1/\sqrt{19}$	531	35	$1/\sqrt{35}$
220	8	$1/2\sqrt{2}$	422	24	$1/2\sqrt{6}$	442	36	$1/6$
311	11	$1/\sqrt{11}$	333	27	$1/3\sqrt{3}$	533	43	$1/\sqrt{43}$

Now, there are certain cases where this sum, sum means summation of all the beams which are coming from different atoms, because of the phase relationship this summation will give you 0. So, that means those beams, those atomic planes, the diffracted beam or intensity of the diffracted beam coming out of those atomic planes is 0. There is no intensity for the diffracted beam.

These are called forbidden reflection and this is completely dependent on the crystallography. This has nothing to do with the diffraction geometry or diffraction condition. So, Bragg's angle may get satisfied for those planes, but still since this

structure factor restrict any intensity for the diffracted beam that is why finally in the diffraction pattern that you record, there you will not get any diffraction or any spot corresponding to those atomic planes.

So, that is what is the importance of structure factor. Basically, structure factor and Bragg's law together decides the nature of the diffracted beam or together decides the nature of the diffraction pattern itself. Remember this Bragg's condition is a necessary, but not sufficient condition for any atomic plane to diffract and ultimately show in the diffraction pattern.

The structure factor determines ultimately that whether you will be able to see the diffracted beam on the diffraction pattern corresponding to any h, k, l plane. And as I said, the structure factor completely depends on the crystallography. So, for a face centered cubic if we take, this kind of calculation we will do in X-ray. When we discuss about X-ray, then we will do this kind of calculations.

For now, just remember that if it is a face centered cubic, then this is what the amplitude and just corresponding to this equation only, here this is the result and final diffracted from any arbitrary h, k, l plane. So, from any arbitrary h, k, l plane, the amplitude of the diffracted beam will be given by this formula and you will be able to get certain conditions that will have an intensity if h, k, l are all even or all odd and 0 is taken as an even number, do not get confused.

So, if we get for an FCC crystal, this means that if we have something like 111 plane where all of these h, k, l indices are odd, we will get an intensity but if we have something like 001 or 100 where 1 is the odd, 0 is the even, we will not get any intensity from the diffracted even if the beam is coming in electron diffraction, even if the beam is coming along the zone axis which is satisfied for 100 planes will still not get it because structure factor restrict any intensity for the diffracted beam.

Same thing again, we can derive the structure factor or we can derive the conditions for this forbidden reflection as this if h, k, l . If it is $h + k + l$ is even we will get an intensity, $h + k + l$ odd we will not get any intensity. So, this we can do it for any kind of crystal structure and as we have seen already that cubic crystals are more symmetric. Therefore,

if we go for any other complex crystal systems it will be just getting the structure factor expression will just getting much more complex okay.

And finally, you can see that these are the first 12 allowed reflection in the FCC crystal structure and their interplanar spacing. So, if you have first diffraction pattern or in the diffraction pattern the first spot will correspond to 111 plane it will not correspond to 100 planes. But we will get a spot and neither 110 plane, it will be 111 plane, the first one and then the second set of spot will be corresponding to 200 because that is again 2 is the even 0 is even 0 is even.

So, that will be the second spot and correspondingly 220, 311 and so on. So, we can find out in a diffraction pattern if we see the diffraction pattern and we know that it is FCC crystal, we safely can say that the first set or the closest one from the direct beam will be from 111 and that is a big implication, we will see it in a minute.

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Camera constant

$\lambda = 2d \sin \theta$
 $\approx d \cdot 2\theta$
 $2\theta = \frac{\lambda}{d}$
 $\frac{r}{L} = \tan 2\theta \approx 2\theta$
 $2\theta = \frac{r}{L}$
 $2\theta = \frac{r}{L} = \frac{\lambda}{d}$
 $L\lambda = rd$

- If we know the camera constant for the instrument, we can determine d simply by measuring r on the pattern.
- In a real microscope, because of the lenses between the specimen and the screen, L is not the physical distance between the specimen and the screen but is a notional distance which can be changed by the microscopist.
- Camera constant calibration: Polycrystalline Au ($a = 0.4078$ nm) foil prepared by evaporation of Au on thin carbon film with a grain size small enough to give rise to continuous ring pattern.

• Distance of diffracted spot from direct (un-diffracted) beam, r is inversely proportional to the d -spacing of diffracting planes

So, the next thing we need to know is camera constant and camera constant we already have a discussion. We have seen that these equations we have and from that we have derived this relationship $L\lambda = rd$ and then from there we have derived a very important relation that the distance of diffracted spot basically from this origin or the direct beam to the diffracted spot.

The center-to-center distance is inversely proportional to the d -spacing of the diffracted planes of diffractive planes means in the interplanar spacing in this specimen planes

which are parallel to the zone axis or parallel to the electron beam and satisfying the diffraction condition. So, their interplanar spacing is inversely related to the distance from the direct beam to the corresponding diffracted beam.

So, now if we know the camera constant for the instrument, then we can simply determine the value of the d in fact, to determine the value of this interplanar spacing by measuring the r , the distance between the origin and the corresponding diffracted spot, we can simply get the interplanar spacing from this if we know the camera constant. And this camera constant usually we can determine.

What happens is that the camera constant if you see $L\lambda$, so λ is basically the wavelength of the electron which can be determined from the accelerating voltage. We know the relationship and if we know what kind of an acceleration voltage we are using, we can very easily get the λ value. L is not a physical distance because you have the lenses in between the specimen, here the lenses are removed for simplicity.

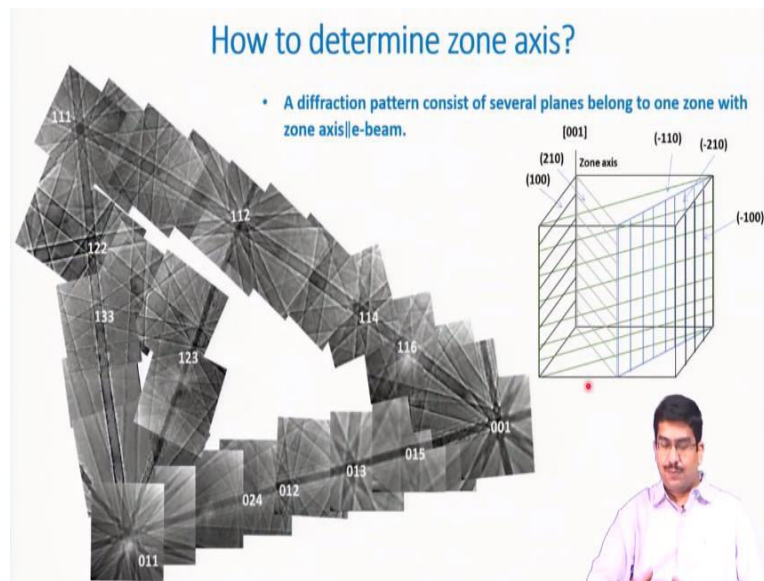
But between the specimen and the screen you have all the lenses, so L is actually not a physical distance but it is a notional distance and there usually used to be a table which says that corresponding to different type of magnification and lenses and then we know exactly and it is a calibration that is done usually. I am not going to how it is done at all. So, it is a calibration because of which we know that this is the length camera length, L is called the camera length, $L\lambda$ is camera constant.

So, we know L , the value of L we can know. And this calibration is usually done with a polycrystalline gold sample which with a very known lattice parameter. So known lattice parameter means we know that it will have a known interplanar spacing. So, we basically know the d value and then we measured the r value, know the λ value, we determine the L .

And that is the kind of calibration we can give for particular different type of magnification, different type of objective lens, projector lens, different type of settings, different type of magnification. We can determine the camera length and then from there we can calibrate for different magnification we have a calibration. So, we can very easily

determine the camera constant for any particular diffraction condition, any particular microscope.

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And then we have to determine something called zone access. This we will be discussing in the next class and then there we will be discussing about how to index this diffraction patterns. And for now, goodbye.