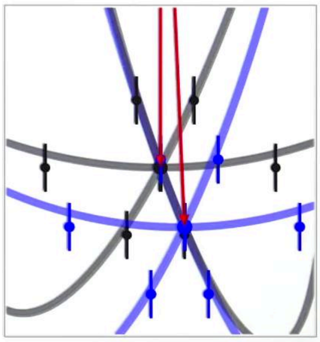
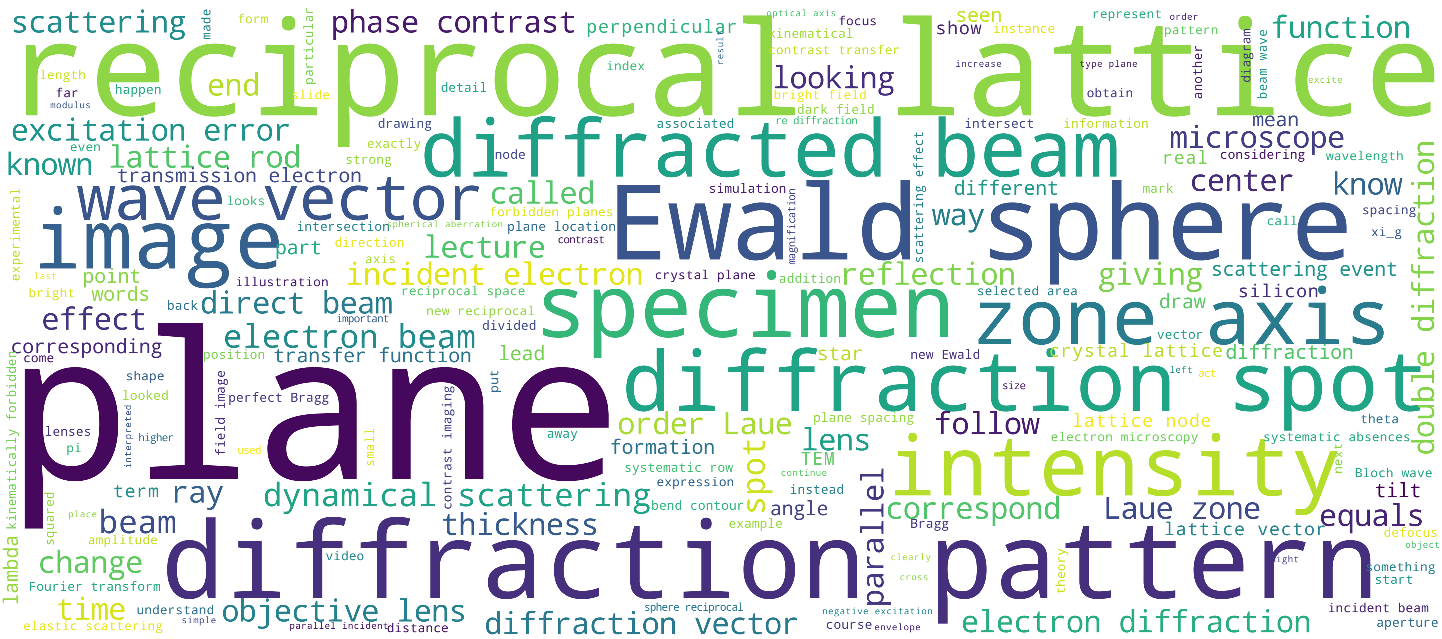


Dynamical effects – double diffraction

Transmission Electron Microscopy

Prof. C. Hébert & Dr D. Alexander



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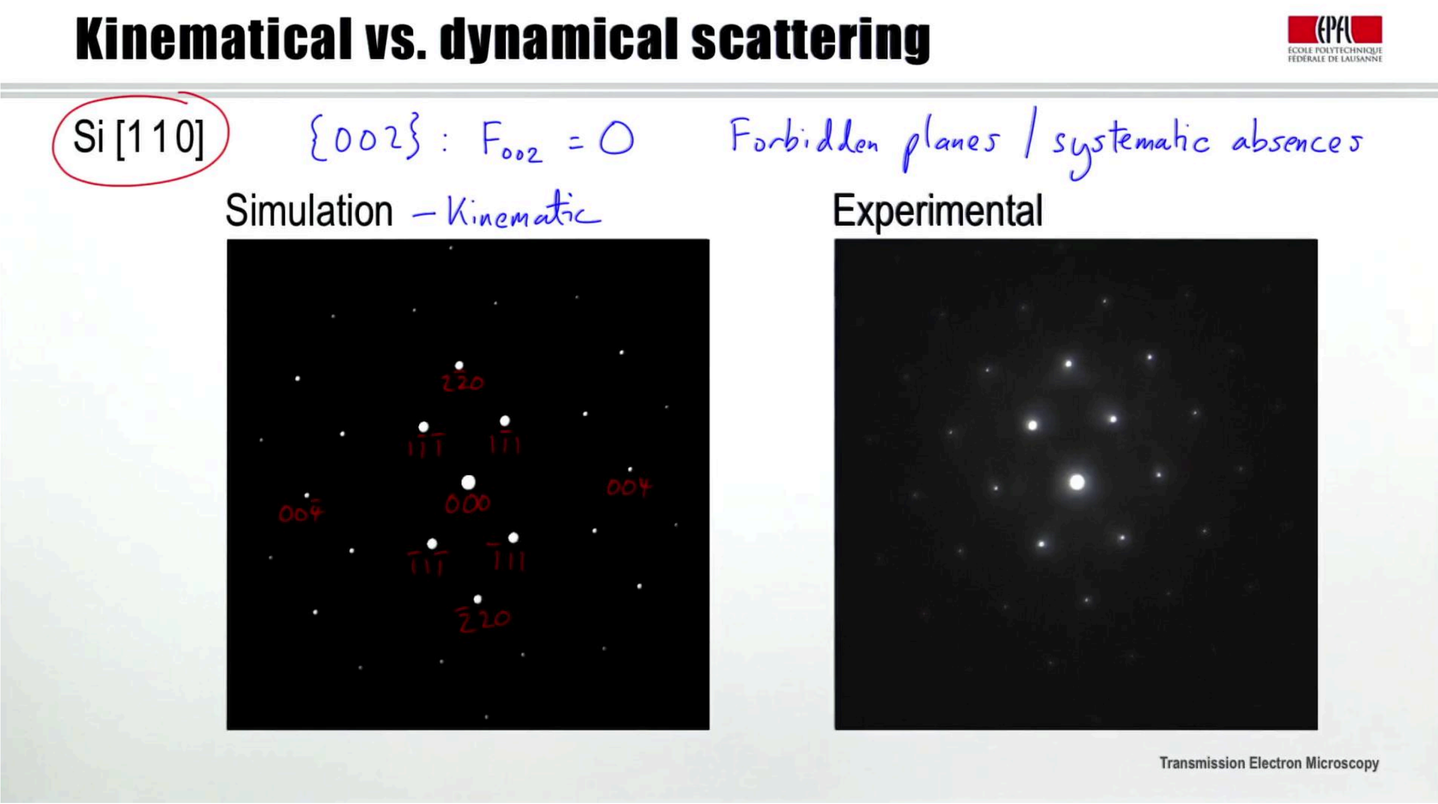
Dynamical effects – double diffraction

Welcome to CIME's lectures on transmission electron microscopy for materials science. In the last two lectures, we have looked at two effects of dynamical scattering on the contrast seen in bright-field and dark-field imaging of crystalline samples; what might be known as diffraction contrast imaging effects. In this lecture, we are instead going to look at an effect of dynamical scattering on diffraction pattern formation, with a particular case known as double diffraction, where the multiple elastic scattering of the beam as it propagates across the sample leads to the formation of diffraction spots for crystal planes which are kinematically forbidden.

Notes

Summary

0m 04s



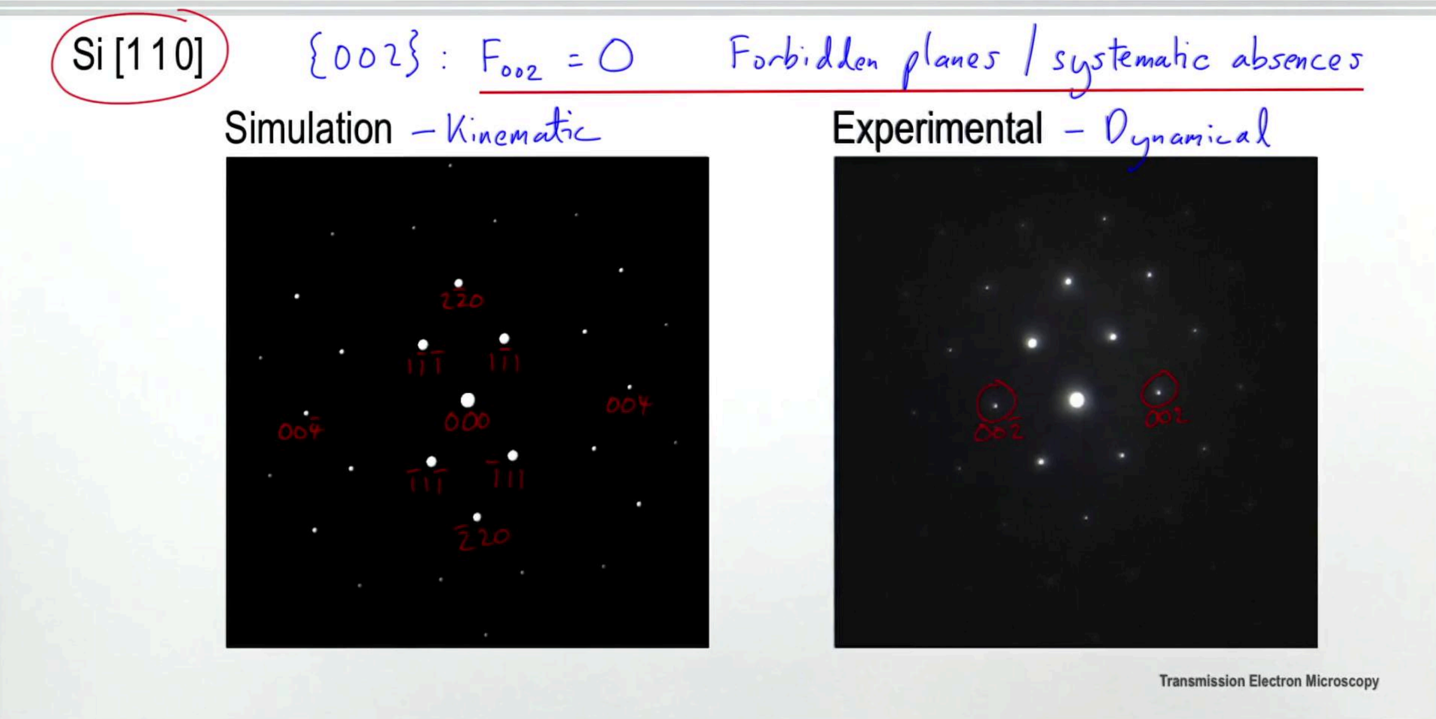
We shall look at double diffraction in what is probably the most common and hence best known case: that of silicon aligned on a [1 1 0] zone axis. Silicon has the diamond structure and for this structure, if you consider the family of {0 0 2} planes, they have a structure factor, say F_002, which equals zero. In other words, if scattering was kinematical, such planes would not be visible as diffraction spots in a diffraction pattern. They are what is known as "forbidden planes", whose diffraction spots are systematic absences. So, if we made a kinematic simulation of a diffraction pattern of silicon on this [1 1 0] zone axis, it would look like this: where by applying the Weiss zone law, h U + k V + l W = 0, we could index the diffraction pattern as follows. In the center, we of course have the direct beam, 0 0 0, and it is surrounded by four {1 1 1} type planes, which we could for instance index as 1 –1 1 here, 1 –1 –1 here, –1 1 –1 here, and finally, –1 1 1 here. Looking at this plane here, we can see that by adding these two diffraction vectors, it corresponds to an index of 2 –2 0. And therefore this one here is –2 2 0. And then looking along this axis we see a spot here, which corresponds to the 0 0 4 plane, and then the 0 0 –4 here.

Notes

Summary

0m 44s

Kinematical vs. dynamical scattering

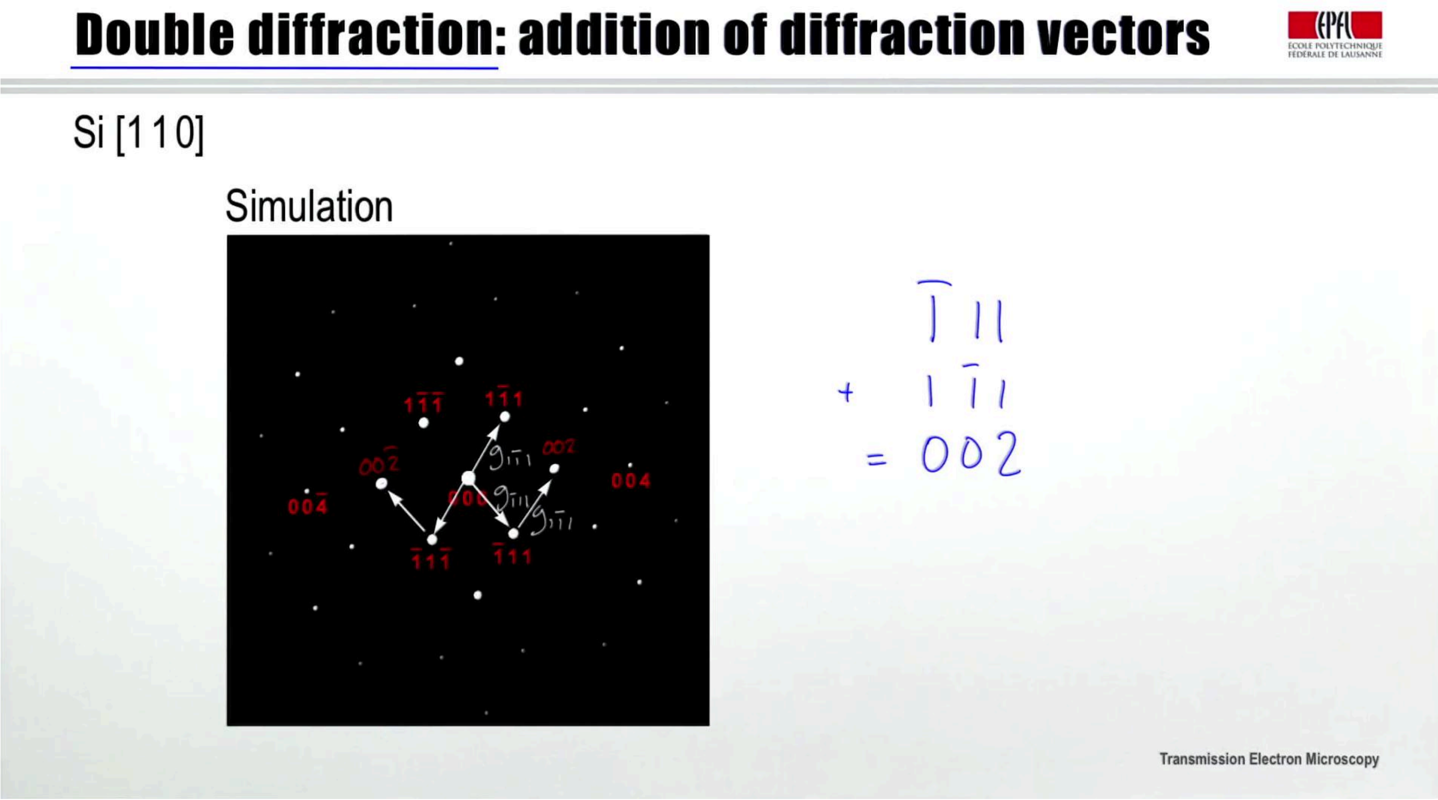


What is noticeable is that there is no diffraction spot here for the 0 0 2, or here for the 0 0 –2, because they are systematic absences – they are kinematically forbidden. Whereas this simulation is kinematical, we know that experimentally, when on a zone axis we have strong dynamical scattering, as the case for this real diffraction pattern here. Comparing this experimental case to the simulation, one thing stands out: that in the experimental case we see intensity for these two forbidden planes. So we have a diffraction spot for the 0 0 2 and the 0 0 –2. We will now look at why that is.

Notes

Summary

2m 44s



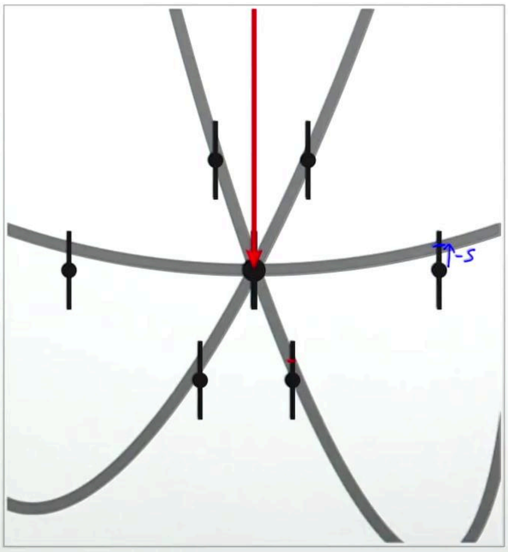
We know that dynamical scattering can lead to diffraction followed by re-diffraction of a beam. This can lead to addition of diffraction vectors – g vectors – as one beam scatters into another beam. We can see that as follows: if we look at our simulation we have a diffraction vector g_1 –1 1 here, and here we have g_–1 1 1. Dynamically, we can have scattering on this g vector, followed by scattering on this g vector. In other words, the diffracted beam for this g vector acts as the direct beam for a subsequent scattering event. When this happens, we can add this diffraction vector to this diffraction vector, giving a diffraction spot here in the 0 0 2 plane location. Equally, we could have scattering to this reflection here, followed by scattering to that reflection there, giving a spot in a 0 0 –2 location. This is known as double diffraction and can be interpreted in terms of the addition of diffraction vectors. In this case, we have g_–1 1 1 plus g_1 –1 1 which equals 0 0 2. This dynamical scattering effect is so strong, that for a typical high tension of two or three hundred kilovolts, you would need a sample just a few nanometers thick to avoid having intensity at these systematic absences.

Notes

Summary

3m 36s

Double diffraction: Ewald sphere/reciprocal lattice



- Diffracted beam wave vector $\boldsymbol{k}_D$ acts as direct beam for new scattering events
- Centre additional reciprocal lattice, Ewald sphere at end of $\boldsymbol{k}_D$
- "Convolution" of reciprocal lattice with itself

Transmission Electron Microscopy

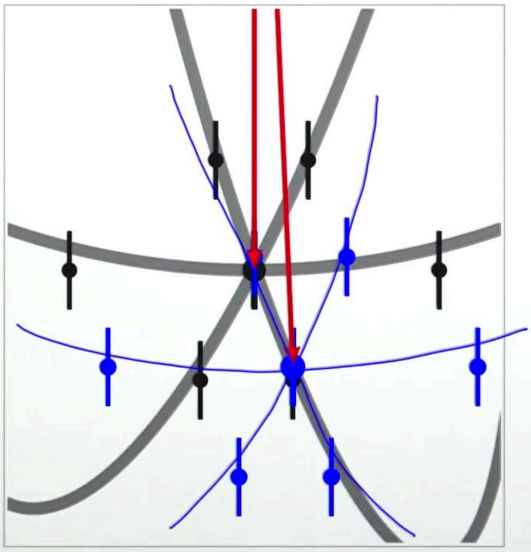
We are now going to consider in more detail the formation mechanism for this double diffraction effect by looking at it within the Ewald sphere/ reciprocal lattice framework. Here we have a schematic illustration of the reciprocal lattice of silicon on this [1 1 0] zone axis, looking in a pseudo three-dimensional projection. These lines represent the elongated form of the reciprocal lattice rods. Because we are aligned on the zone axis, this zero order Laue zone is perpendicular to the incident electron beam. Therefore, we can draw the wave vector for that incident beam like this. Associated with this incident beam is of course the Ewald sphere, which will intersect these different reciprocal lattice rods. To represent this intersection I will draw arcs, which correspond to different circumferences on the Ewald sphere, intersecting different pairs of reflections. So one here, here, and here. So giving a diagram that looks like this. Because these planes are parallel to the incident electron beam, the Ewald sphere does not intersect at the center of the reciprocal lattice rods, but instead at a negative excitation error. Therefore, if we consider this (1 –1 1) plane here, the Ewald sphere intersects somewhere there.

Notes

Summary

5m 38s

Double diffraction: Ewald sphere/reciprocal lattice



- Diffracted beam wave vector $\boldsymbol{k}_D$ acts as direct beam for new scattering events
- Centre additional reciprocal lattice, Ewald sphere at end of $\boldsymbol{k}_D$
- "Convolution" of reciprocal lattice with itself

Transmission Electron Microscopy

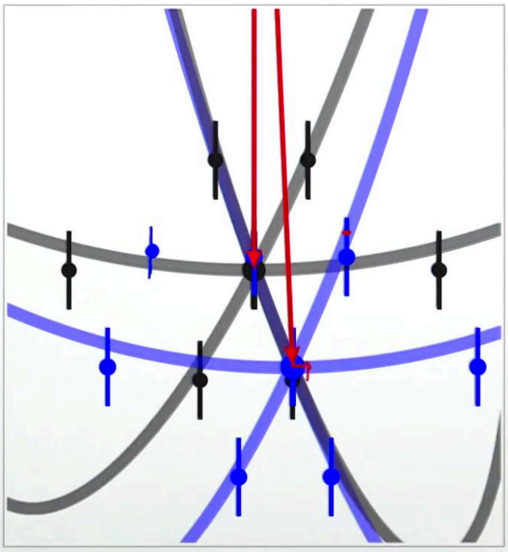
Thus considering that for this illustration, the center of the Ewald sphere is somewhere above the slide, the diffracted beam wave vector for this plane will come down as follows, giving a figure like this. In double diffraction, this diffracted beam wave vector k_D, now acts as the direct beam for subsequent scattering events. To represent this, we first center a new reciprocal lattice at the end of this wave vector here. So (0 0 0) here. Over here, we will have a reciprocal lattice node for a (0 0 4) plane, and the (0 0 –4) here. And then the {1 1 1} planes: here – in line with the original 0 0 0; somewhere here; here; and here. Giving an illustration that looks like this. As well as marking in a new reciprocal lattice, we also have to mark in a new Ewald sphere, associated with this wave vector here. Because this wave vector is inclined relative to the incident electron beam, this Ewald sphere will be tilted compared to the original Ewald sphere. I now draw in arcs for the intersection of this new Ewald sphere with this reciprocal lattice, giving a final diagram which looks like this.

Notes

Summary

7m 23s

Double diffraction: Ewald sphere/reciprocal lattice



- Diffracted beam wave vector $\boldsymbol{k}_D$ acts as direct beam for new scattering events
- Centre additional reciprocal lattice, Ewald sphere at end of $\boldsymbol{k}_D$
- "Convolution" of reciprocal lattice with itself

Transmission Electron Microscopy

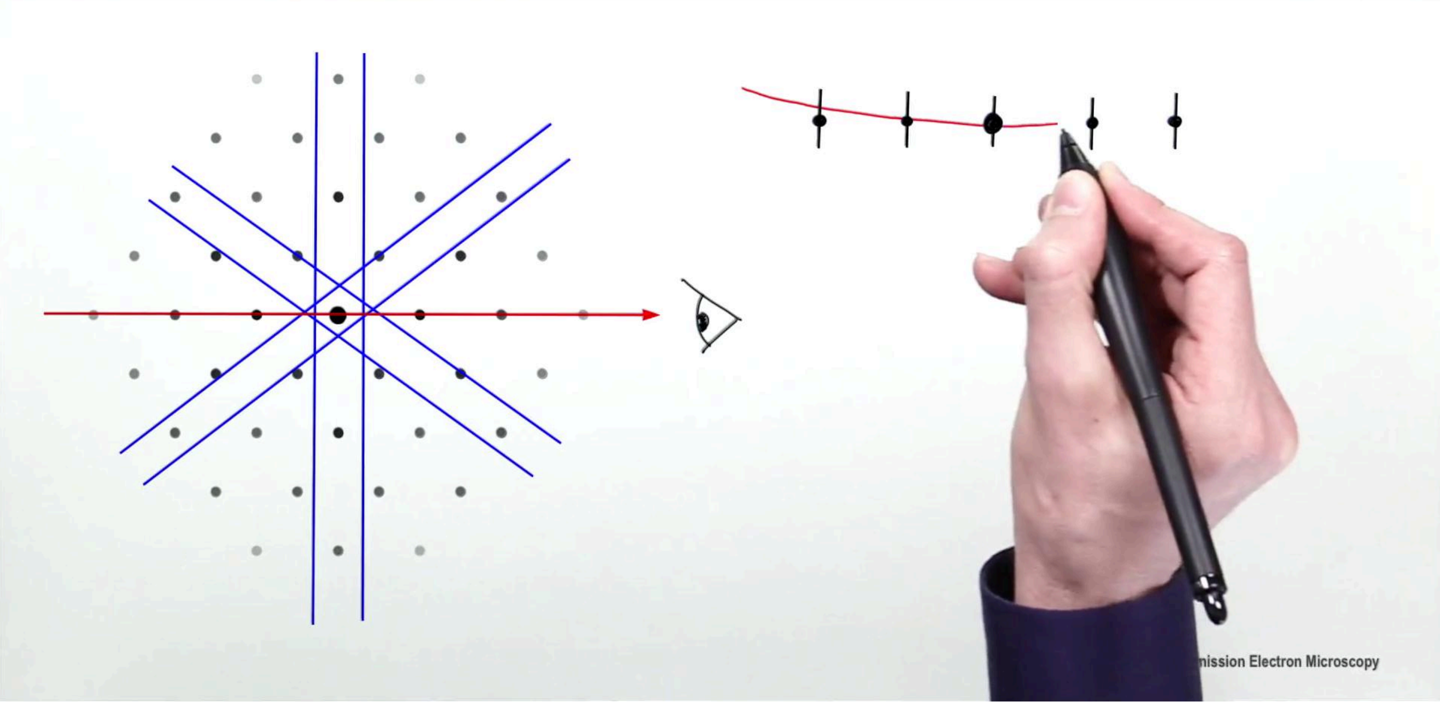
We can see that, in the projected electron diffraction pattern, the intersection of this new Ewald sphere with this reciprocal lattice rod here, will give a diffraction spot in the (0 0 2) plane location. Equally, if we consider re-diffraction from this (–1 1 –1) plane here, we would end up with a reciprocal lattice rod here, corresponding to the (0 0 –2) plane location in the electron diffraction pattern. We can think of this as a convolution of the reciprocal lattice with itself. So here, this new reciprocal lattice was convoluted with a reciprocal lattice node of the original reciprocal lattice. However, it must be noted, that in reciprocal space, this new reciprocal lattice is at a negative excitation error compared to the original reciprocal lattice. In other words, it is higher up.

Notes

Summary

9m 20s

Experimental proof: tilt to systematic row



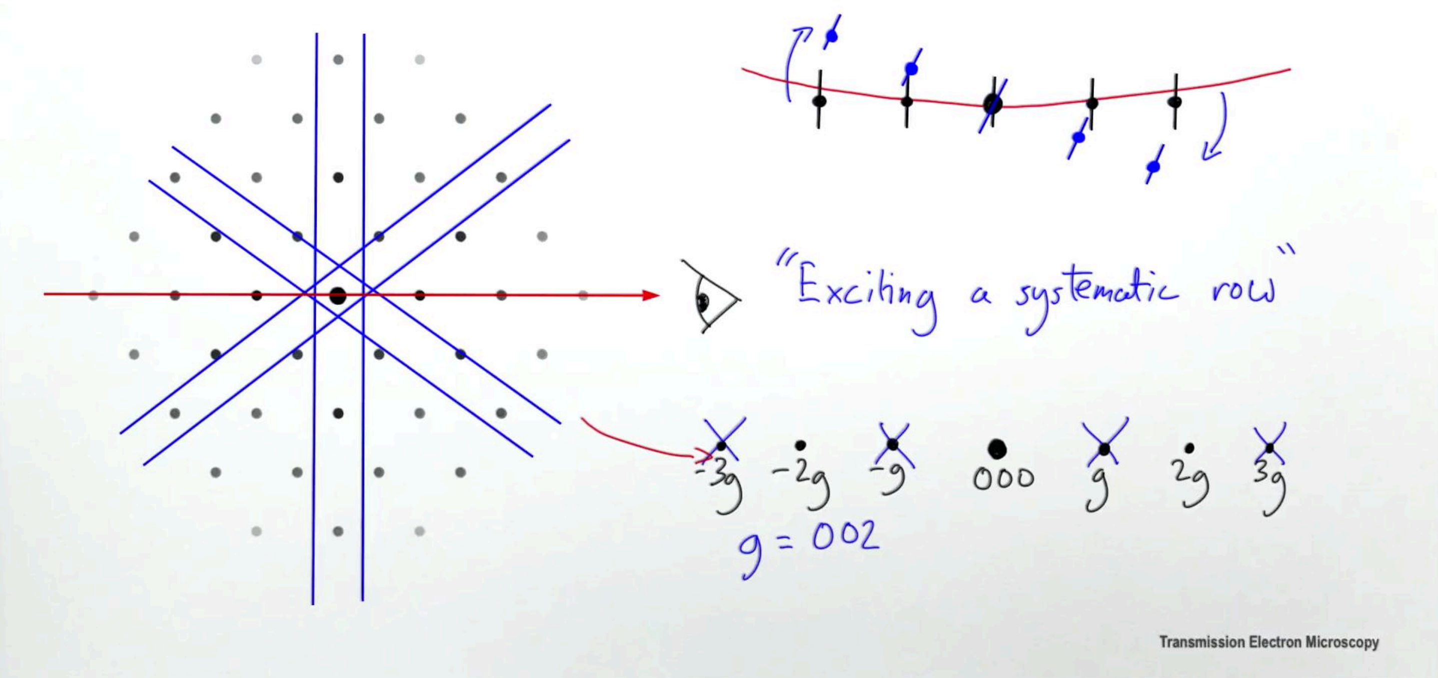
We are now going to look at a way that we can experimentally prove that the diffraction spots for these forbidden planes are the result of dynamical scattering. If we look at the diffraction pattern, we know that each spot is associated with a plane which is perpendicular to its diffraction vector. So for this spot here, we could mark in a plane like this. And for this one here, a plane here. And then planes for this spot here. And then this one here. And this plane here. And another here. And you could continue for all the different spots in the diffraction pattern. Now we know in this zone axis geometry, in the zero order Laue zone, all these planes are parallel to the incident electron beam. Then what we are going to do is tilt the sample on this axis here, as follows. If we look down that tilt axis at the zero order Laue zone, we would see something like this. Where this is looking down the tilt axis where we would see reciprocal lattice rods, both for the 0 0 0, but also these other planes here. And these are reciprocal lattice rods for planes out here or out here. Being in zone axis orientation, the Ewald sphere is tangential with the zero order Laue zone, so will intersect the reciprocal lattice rods as follows.

Notes

Summary

10m 25s

Experimental proof: tilt to systematic row



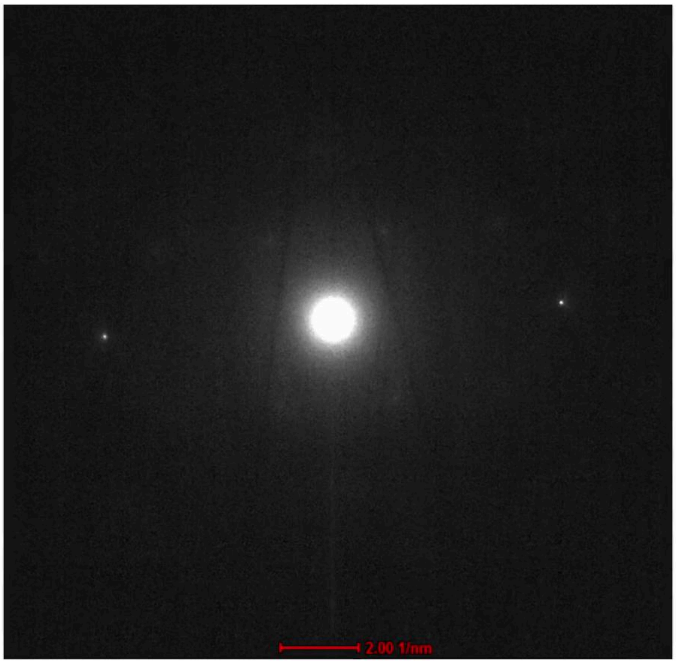
When we now tilt around this axis, we will tilt this zero order Laue zone around this point here, as follows. We can see that, if we tilt far enough, the Ewald sphere no longer intersects the reciprocal lattice rods of these planes. However, the Ewald sphere does still intersect the planes on the axis of rotation. That is because these planes remain parallel to the incident electron beam. Doing this is called exciting a systematic row. That is because, in the diffraction pattern, we will obtain something like this. The direct beam in the center, then a spot for a plane that we will call g, then another spot, the 2 g – a plane which is parallel to the plane for g but has half its spacing – and then 3 g and so on, and their negative counterparts. However, there should be very little intensity in the other diffraction spots, because it is only these planes which are parallel to the incident electron beam. Now, in this case, if we say g equals 0 0 2, then by exciting this systematic row, we will cut out the {1 1 1} planes which are responsible for its formation by double diffraction. Therefore, as we tilt to excite this systematic row, we should see g and minus g disappear. And also, if we included them on the diffraction pattern, then 3 g and minus 3 g. We should be left only with 2 g and minus 2 g, corresponding to the (0 0 4) and (0 0 –4) planes respectively.

Notes

Summary

12m 33s

Demo



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To show this, we look at a video of tilting in this way. At the beginning, we are aligned precisely on the [1 1 0] zone axis, with the (0 0 2) and (0 0 –2) planes strongly excited. Now, we start tilting around this axis. And as we do this, we will see intensities change as the Ewald sphere cuts planes at different excitation errors. And then gradually, we will see that these 0 0 2 planes disappear when the 1 1 1 reflections are completely de-excited. So there in fact they have already disappeared. They come back again. And then now as we tilt even further to excite the systematic row very precisely, the 0 0 2 planes have completely disappeared, thus proving that they were a consequence of double diffraction.

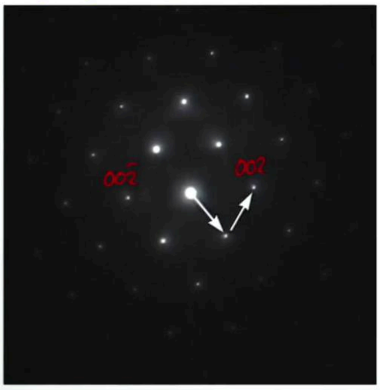
Notes

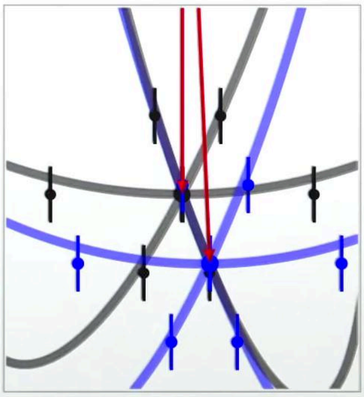
Summary

14m 48s

Dynamical effects – double diffraction summary

Si [1 1 0]





- Double diffraction: particular case of dynamical scattering
- Gives diffraction spots for forbidden planes
- Diffracted beam acts as direct beam for new scattering events
- Demonstrated double diffraction in single crystal

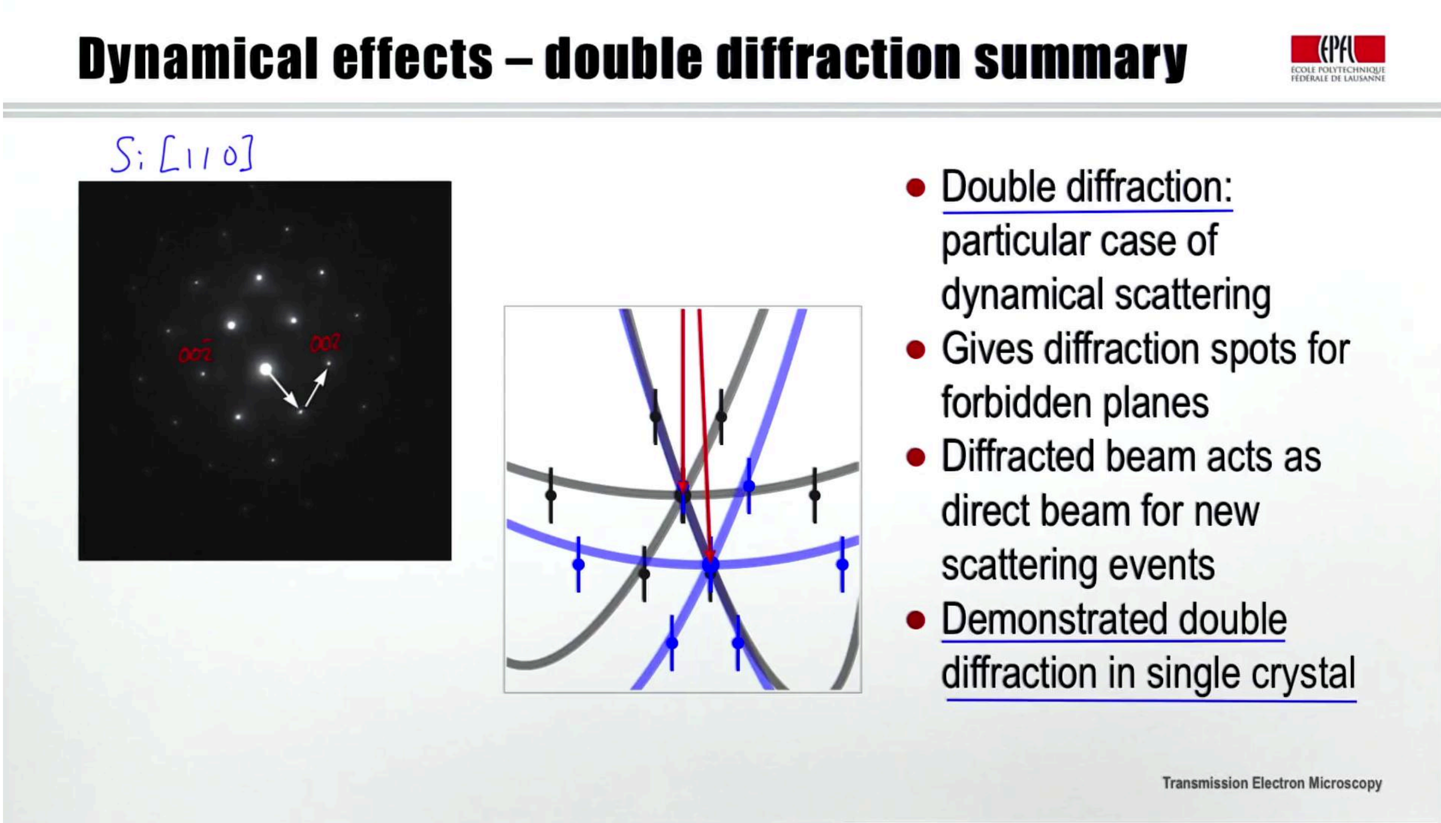
Transmission Electron Microscopy

To summarize, in this lecture we have looked at double diffraction, which is a particular case of dynamical scattering that can give rise to the appearance of diffraction spots for kinematically forbidden crystal planes. To look at this, I have used the case of silicon aligned on a [1 1 0] type of zone axis, where in the experimental diffraction pattern, we see intensity for forbidden planes (0 0 2) and (0 0 –2). We have interpreted this in terms of a diffracted beam from a first scattering event that acts as the direct beam for new scattering events. For instance, in the case of silicon, we can look at scattering on this g vector to this {1 1 1} type plane, combined with scattering on this g vector, adding together to give scattering to this 0 0 2 diffraction spot location. Looking at this in more detail, we can conceive it as an additional reciprocal lattice and Ewald sphere centered at the end of a wave vector for an initial diffracted beam. While here I have demonstrated double diffraction for the case of scattering across a single crystal, double diffraction can also occur when there is scattering from one crystal lattice into and across another crystal lattice.

Notes

Summary

15m 46s



In this second case, it can lead to the formation of what are known as satellites spots which surround the diffraction spots from the initial crystal lattice. It can further be interpreted via the same Ewald sphere/reciprocal lattice construction, where the second reciprocal lattice would instead correspond to the reciprocal lattice of the second phase in the sample.

Notes

Summary

17m 21s

Dynamical effects – double diffraction summary

This brings me to the end of my basic discussion of dynamical scattering effects for a parallel incident illumination. If we instead focus the beam to a spot on the sample, the illumination is convergent. If you then look at the diffraction pattern, you will see what is called a "convergent beam electron diffraction" pattern, or CBED pattern. These patterns can show very strong contrasts from dynamical scattering. And, while not covered so far, this contrast can be highly interpretable to obtain useful information about your sample. Returning to the case of a parallel incident illumination, there again there can be another kind of multiple scattering, this time where the coherent elastic scattering that we have considered so far is preceded by an incoherent scattering event. The combination of this incoherent scattering, followed by coherent elastic scattering, can produce bright and dark lines in a selected area diffraction pattern. These Kikuchi lines can be very useful for precisely aligning a sample to a specific diffraction condition. However, these too are a subject for another time.

Notes

Summary

17m 48s

Dynamical effects – double diffraction summary

Now that we have treated the basics for the theory of electron diffraction, and also seen some examples of the effects of diffraction on TEM image contrast, we will now go onto another important category of imaging contrast in TEM, namely phase contrast imaging. And for this I hand over to Cécile. Thank you Duncan. Phase contrast derives from the interference of different elastically-scattered beams, and it is the basis for high-resolution TEM, also called atomic resolution imaging. We will go through the theory of phase contrast in the coming lectures.

Notes

Summary

19m 10s