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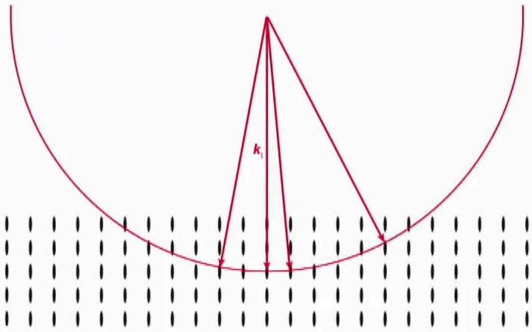
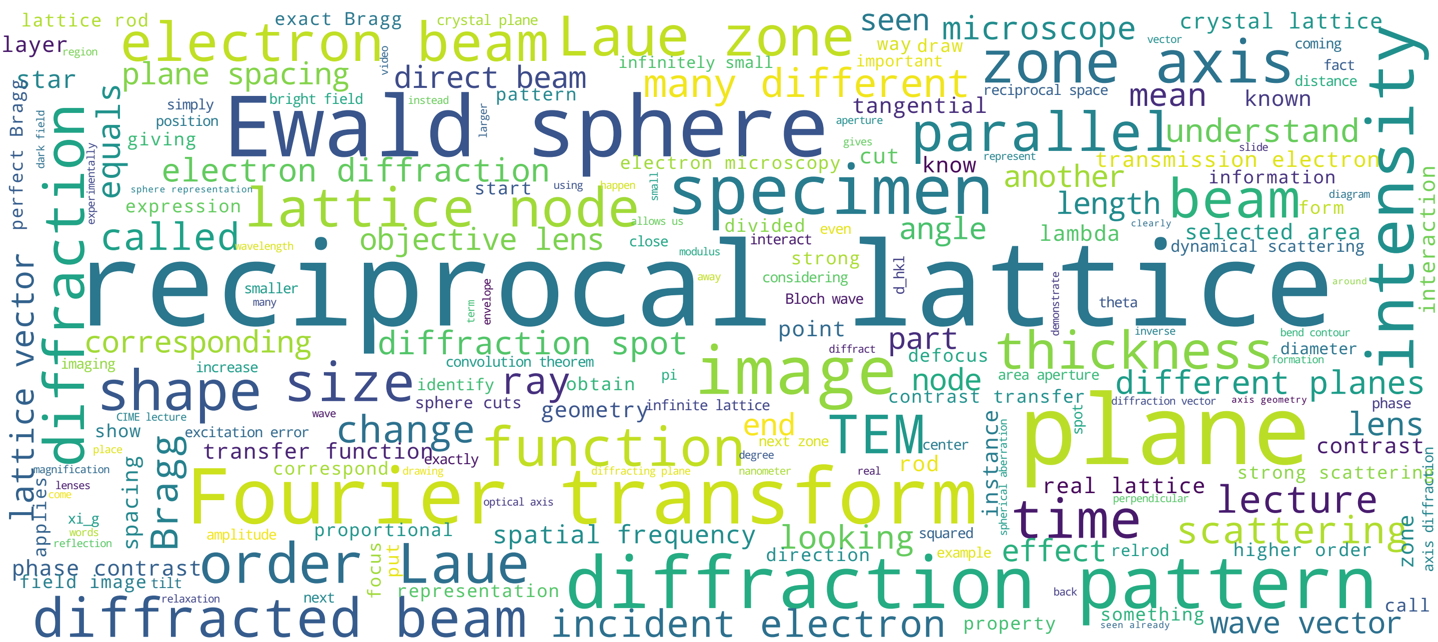


Shape effects in the reciprocal lattice

Transmission Electron Microscopy

Prof. C. Hébert & Dr D. Alexander



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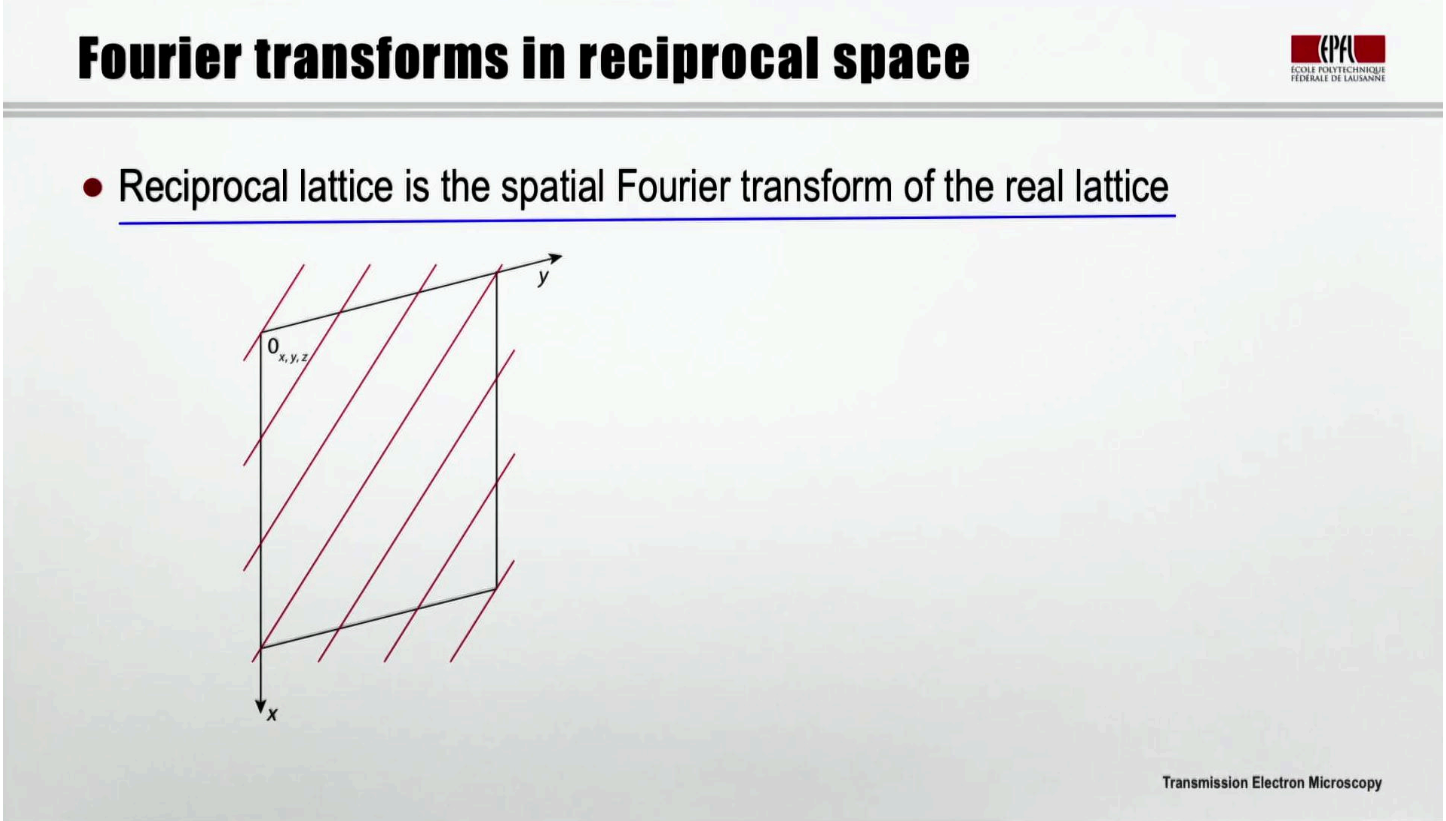
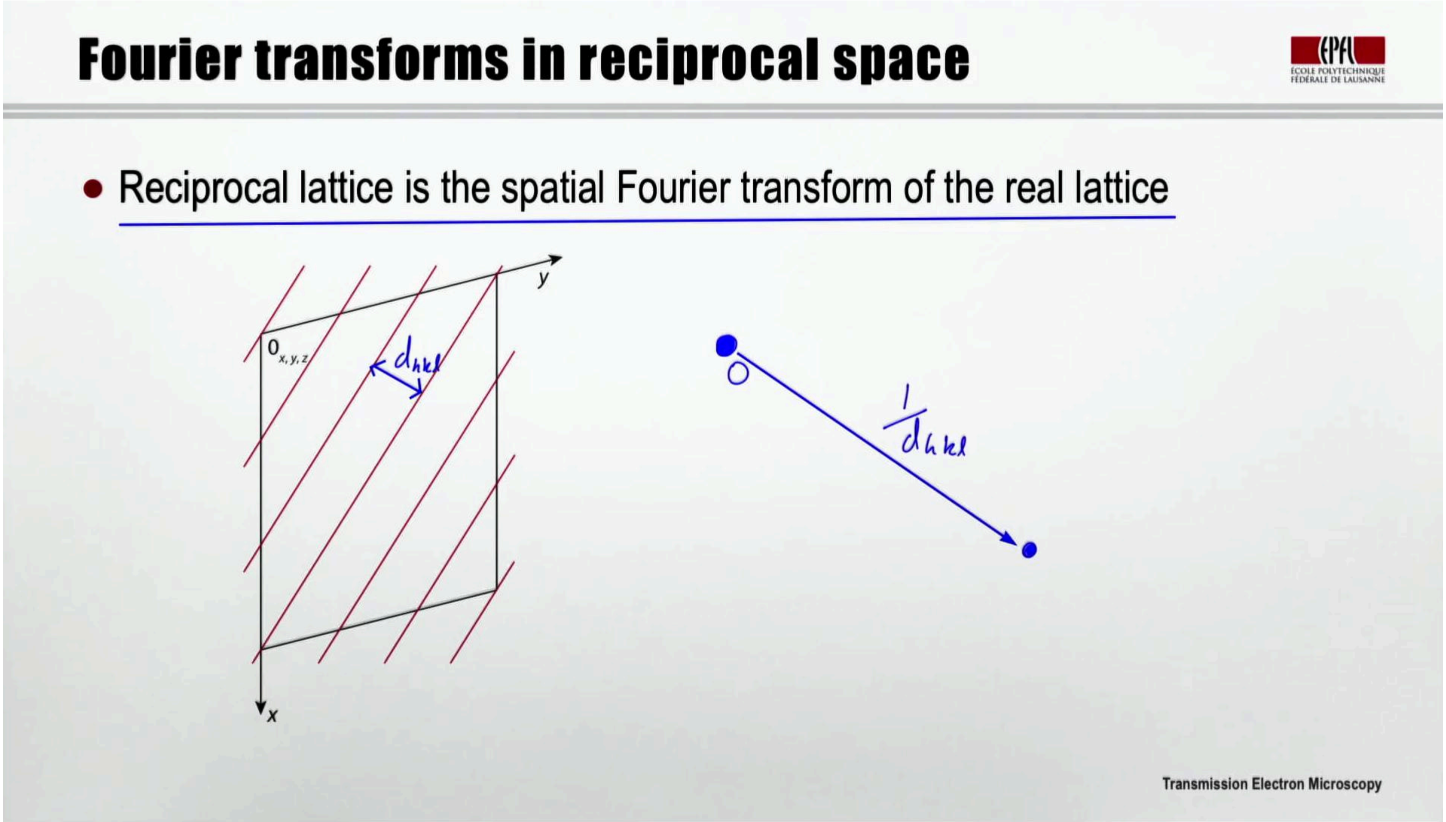


Welcome to CIME's lectures on transmission electron microscopy for materials science, and the lecture on shape effects in the reciprocal lattice. In the last lecture on zone axis electron diffraction, I identified a problem in our representation of the Ewald sphere and the reciprocal lattice, in that, while experimentally we know that in this geometry – where we have the incident electron beam parallel to a low index zone axis vector – we have strong scattering from many different planes which are parallel to that incident electron beam, this strong interaction was not illustrated in our Ewald sphere/ reciprocal lattice representation. In this lecture, I will resolve this problem by illustrating how each reciprocal lattice node is not infinitely small, but it has a certain size and shape, related to the size and shape of our TEM sample geometry. And it is because of this size and shape, that the reciprocal lattice nodes can interact with the Ewald sphere, and we can have strong scattering from many different crystal planes at the same time. To understand these shape effects, I'm going to use the fact that the reciprocal lattice is the spatial Fourier transform of the real space crystal lattice.

Notes

Summary

0m 04s

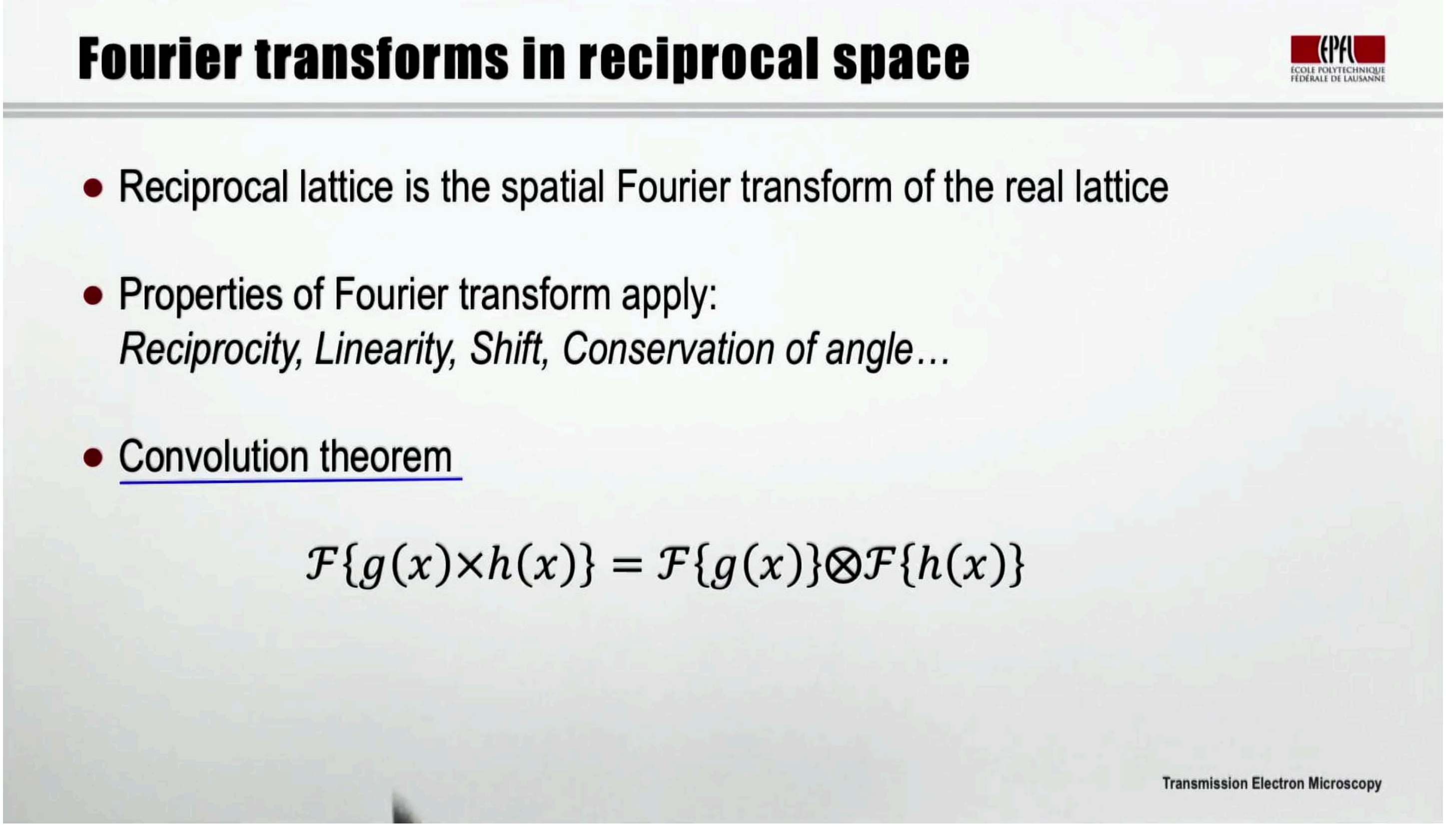


While I say this without proof, we can understand it on a simple level by considering the reciprocal lattice vector for a plane, (h k l), which has a spacing of d_hkl. When we construct the reciprocal lattice vector for this plane, as we have seen, we start at the origin, 0 (h k l), and then make a vector perpendicular to the plane which has a length which is inversely proportional to the plane spacing. Now, if we think about the spatial frequency of that plane, it will be one divided by the plane spacing, and so identical to the length of the reciprocal lattice vector. Therefore, we can see that this reciprocal lattice node – or, in a diffraction pattern, the diffraction spot – contains the information on the spatial frequency of that plane. While Fourier transforms are often used to identify the temporal frequency of waves, if we apply the Fourier transform to our lattice, we identify the spatial frequency components of the lattice, hence we identify the spatial frequency of different planes – so giving the reciprocal lattice, where each node identifies the spatial frequency of its corresponding crystal plane.

Notes

Summary

1m 28s



With the reciprocal lattice being the spatial Fourier transform of the real lattice, all the properties of the Fourier transform apply to this transformation of real lattice to reciprocal lattice. Most obviously is the property of reciprocity. However, other properties apply as well, such as linearity, which means that that the Fourier transform of function (A) plus function (B) is the Fourier transform of function (A) plus the Fourier transform of function (B). Next, there is the property of shift. According to this, if a function is shifted, the only change in its Fourier transform is a shift in phase. Since in diffraction mode we can only measure wave intensities, and not their phases, this means that if the real lattice shifts, then its diffraction pattern does not change, neither in diffraction spot position or intensity. There is also the property of conservation of angle. Because of this, if the real lattice rotates by a certain number of degrees, the reciprocal lattice will rotate by the same number of degrees. However, for this lecture, the most important property of Fourier transforms that we are going to use is that of the convolution theorem.

Notes

Summary

2m 47s

Fourier transforms in reciprocal space

- Reciprocal lattice is the spatial Fourier transform of the real lattice
- Properties of Fourier transform apply:
  Reciprocity, Linearity, Shift, Conservation of angle…
- Convolution theorem

$$\mathcal{F}\{g(x)\times h(x)\} = \mathcal{F}\{g(x)\}\otimes\mathcal{F}\{h(x)\}$$

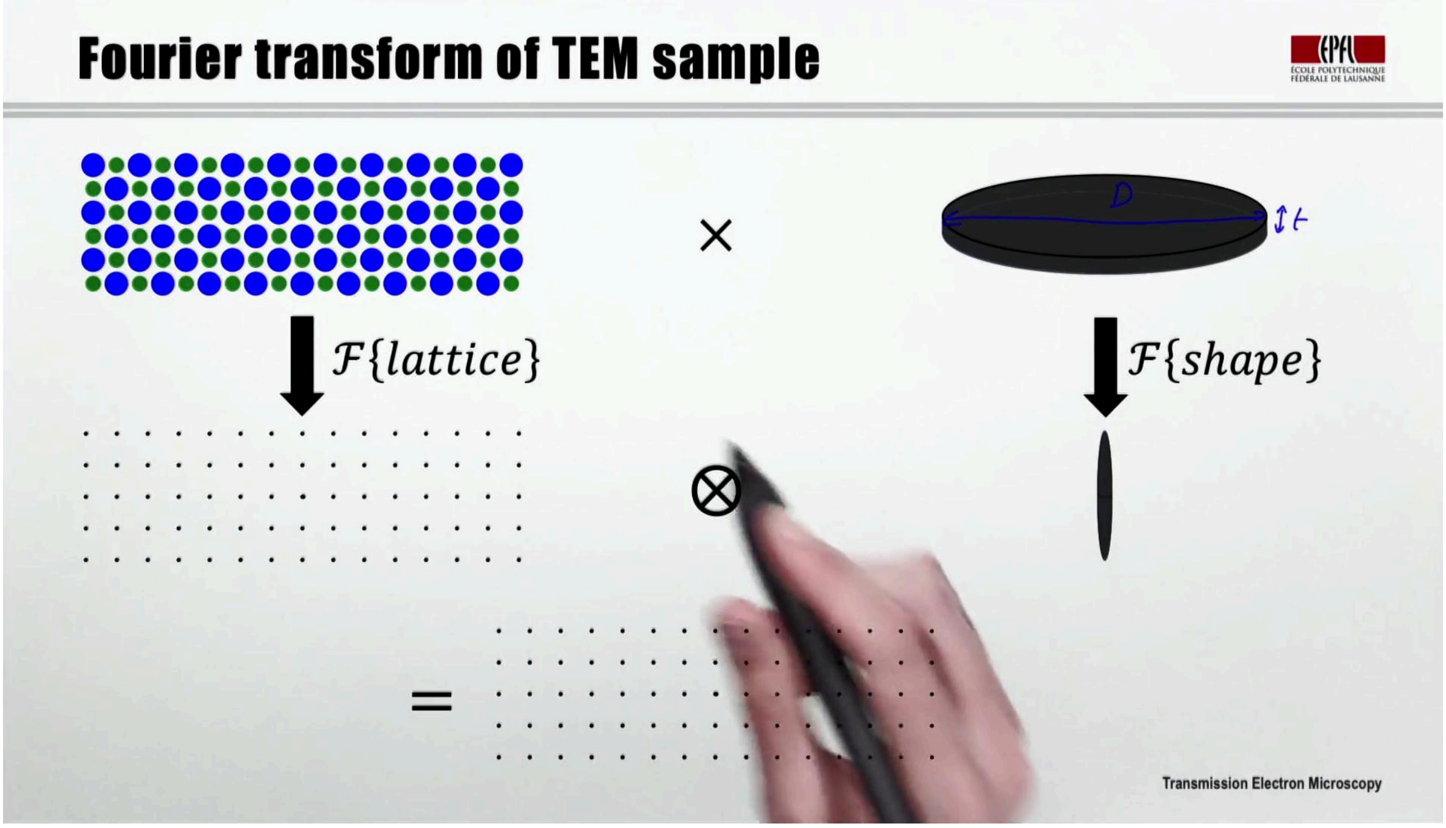
Transmission Electron Microscopy

Convolution states that, if we take the Fourier transform of one function multiplied by another function, the Fourier transform of this is going to be the Fourier transform of the first function convoluted with the Fourier transform of the second function. So here, I'm using this symbol to represent that convolution, and we will now use this convolution theorem to understand the effect of TEM sample geometry on the size and shape of reciprocal lattice nodes.

Notes

Summary

4m 00s

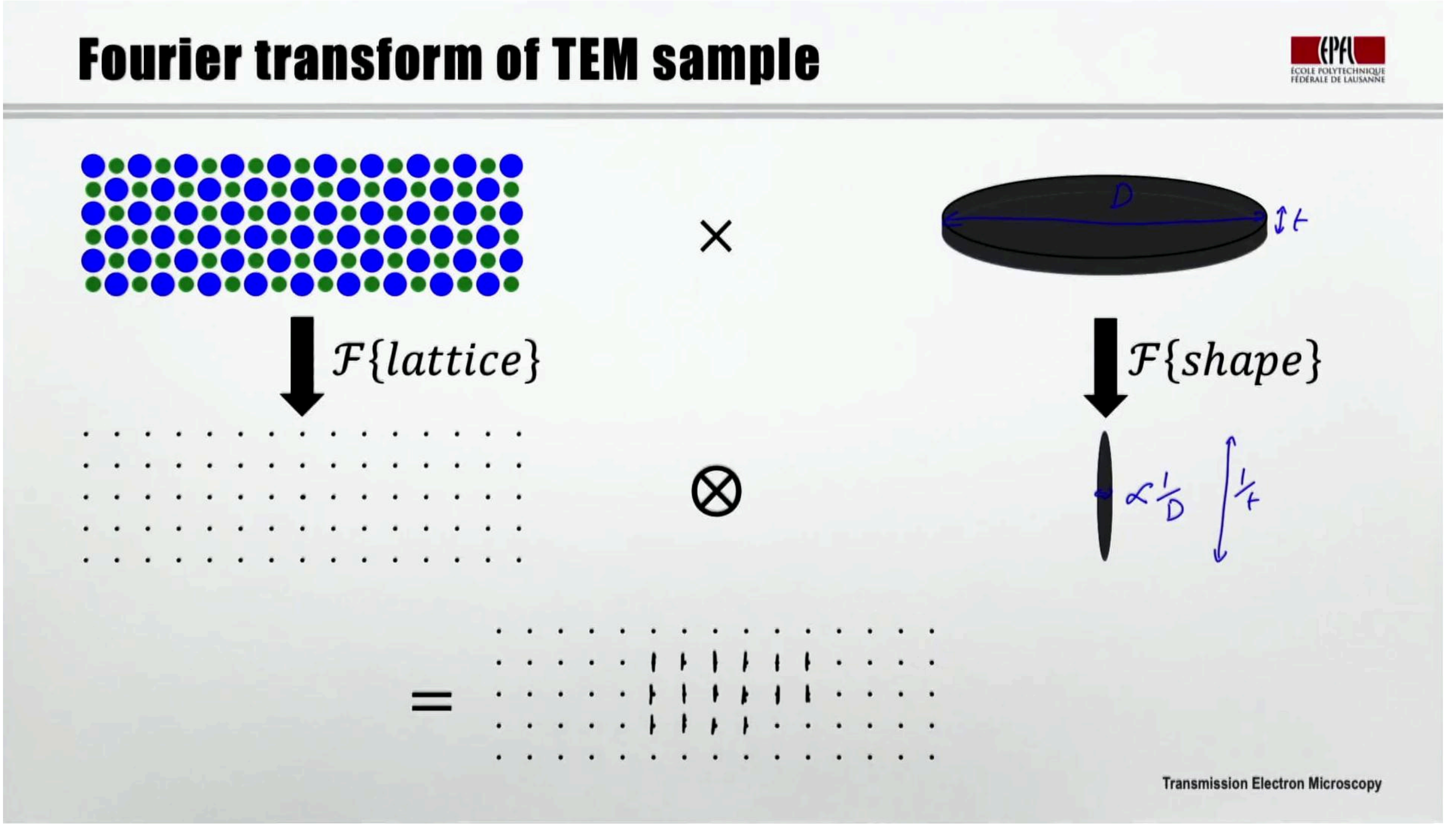


If we consider first an infinite crystal lattice – so an infinitely repeating arrangement of atoms throughout space – if we take the Fourier transform of this infinite lattice, we will obtain the reciprocal lattice that we have seen already, with infinitely small reciprocal lattice nodes. However, it is obvious that our TEM sample is not infinitely large. In fact, it is a bound volume of matter. More particularly, if we do selected area diffraction, the piece of material that we will sample will have a diameter, which here I call D, corresponding to the diameter of the selected area aperture back-projected to the specimen plane. Further, it will have a thickness – here called t – corresponding to the thickness of the TEM sample. This is typically just some tens, or one or two hundreds, of nanometers, in order that the sample be electron transparent. This shape function will multiply the infinite crystal lattice that we have represented here. So to calculate our actual reciprocal lattice, we need to take the Fourier transform of this infinite lattice multiplied by this shape function here. And this is where we can use the convolution theorem.

Notes

Summary

4m 35s

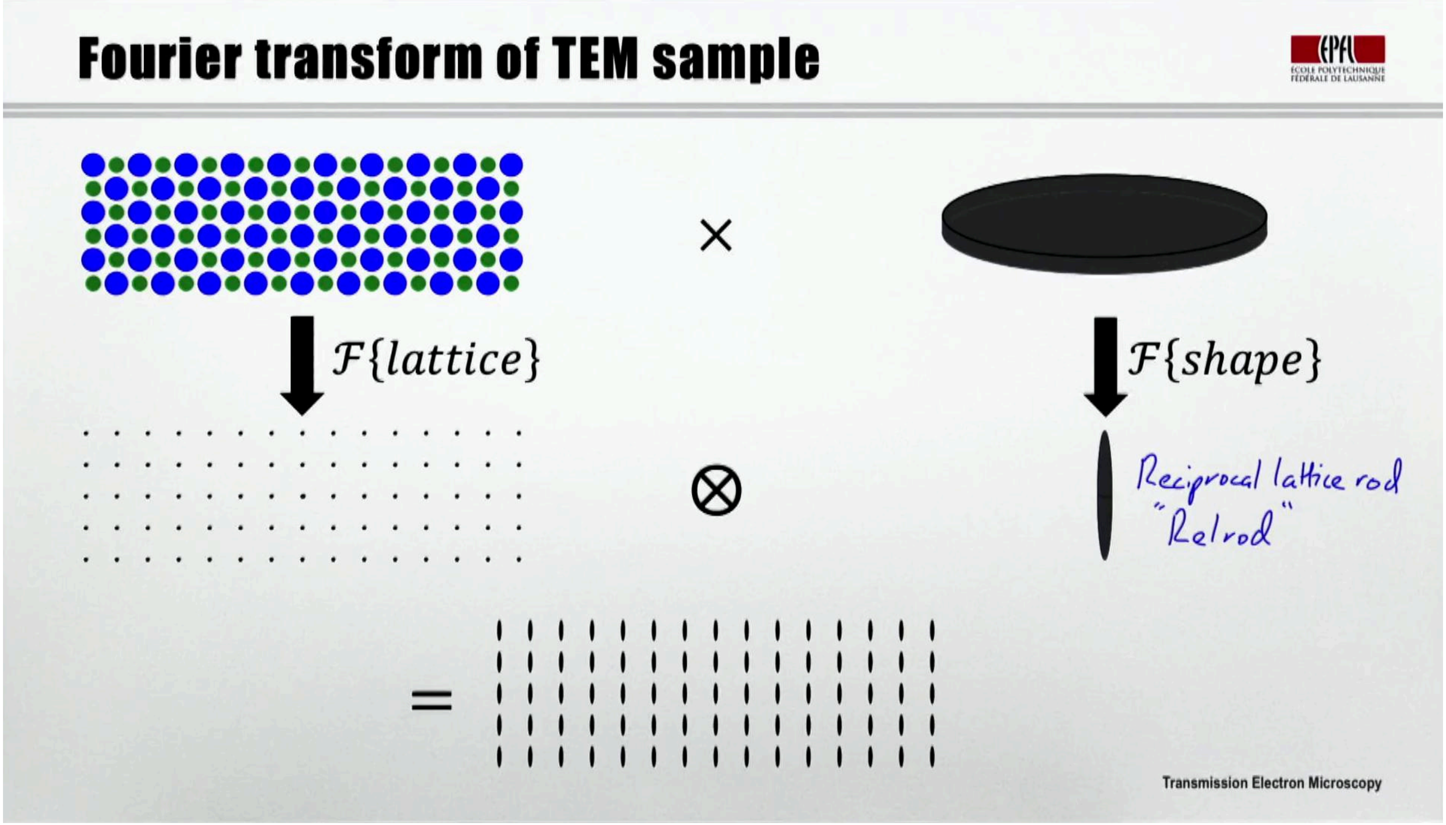


So first of all, we will take the Fourier transform of this infinite lattice, making this reciprocal lattice with infinitely small nodes that we have seen already. And then we take the Fourier transform of this disk shape. On a first approximation, to do that we will simply take the reciprocal of this shape. So, as a shape which has a large diameter relative to its thickness, if you take the reciprocal of that or the inverse, then we end up with this rod-like shape, where the diameter of that rod is proportional to one over the diameter of the selected region of sample; here. And the length of that rod will be proportional to one over the TEM sample thickness. We will then convolute this rod-like shape with our reciprocal lattice here. Now, when we consider it, the size of our TEM sample is much larger than the interatomic plane spacings in our crystal lattice. That means, in reciprocal space, that the size of this rod will be much smaller than the size of the spacings of these reciprocal lattice nodes. So when we perform this convolution, we will end up with little rod shapes on each of these nodes. So we convolute the rod shape with each node in the reciprocal lattice.

Notes

Summary

5m 54s

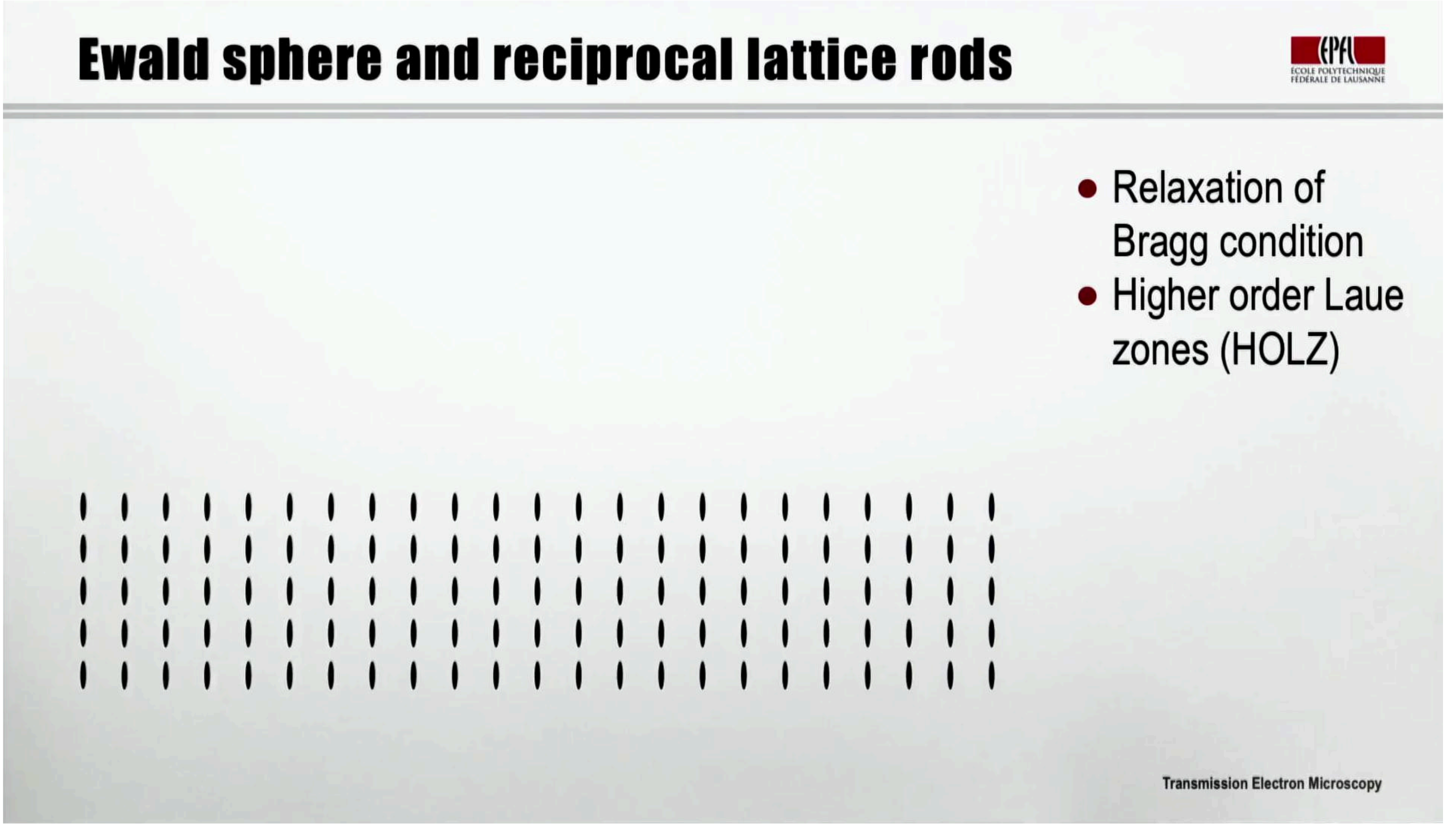


If we keep doing that we will end up with something like this. So now, we see a reciprocal lattice, where each node has a certain size and shape. And, while the spacing between the nodes corresponds to the inverse of the plane spacings, the size of each node is related to the size and shape of our sample. Therefore, we have two length scales in our reciprocal lattice. Because of the importance of this rod-like shape, we call them by a specific term, which is "reciprocal lattice rods" or "relrods" for short.

Notes

Summary

7m 17s

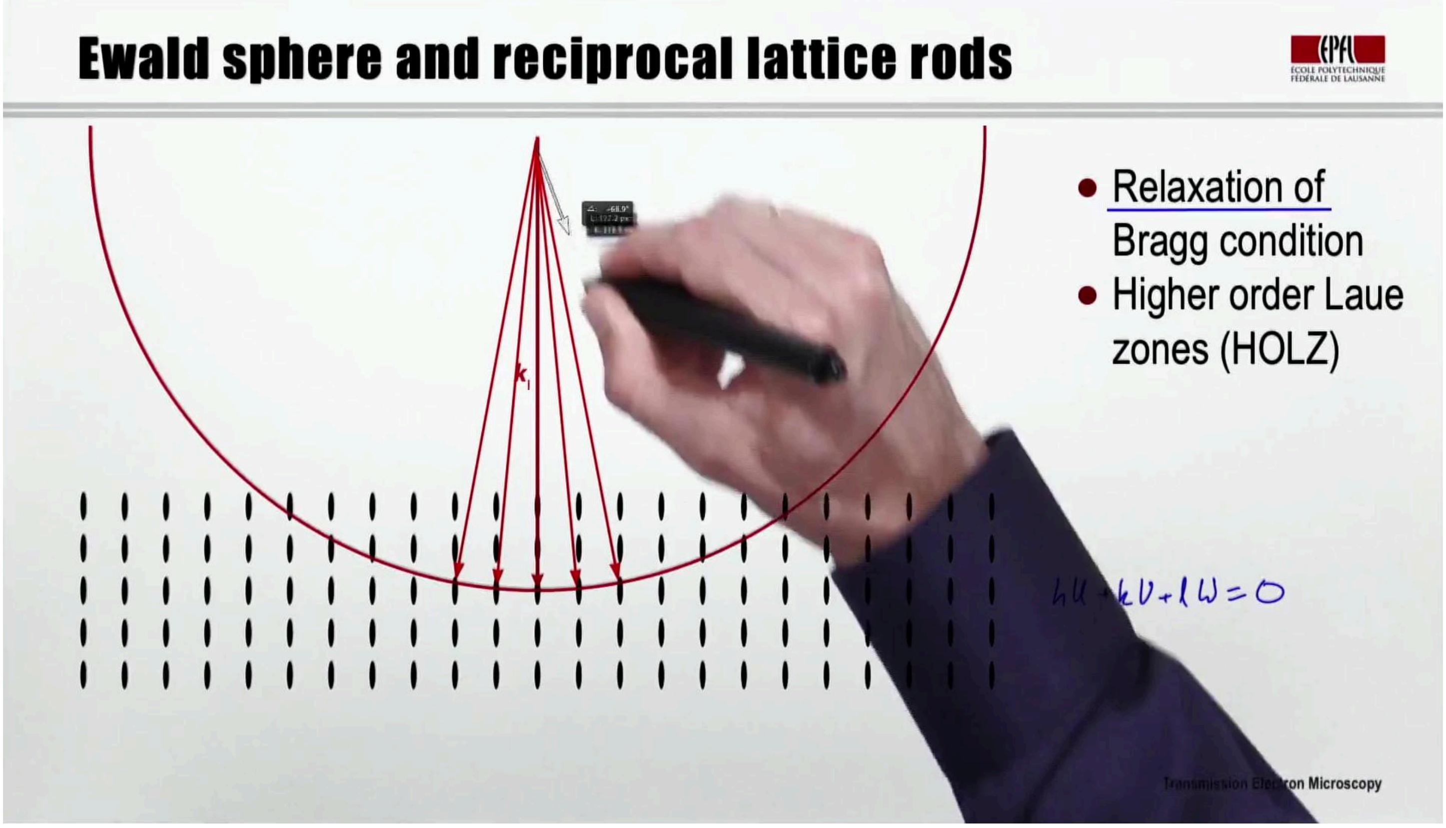


Now, we have this representation of the reciprocal lattice. we are going to interact that with the Ewald sphere in the zone axis geometry where, in the reciprocal lattice, we have one layer or zone of the reciprocal lattice tangential with the surface of the Ewald sphere; and we will see what happens.

Notes

Summary

7m 59s

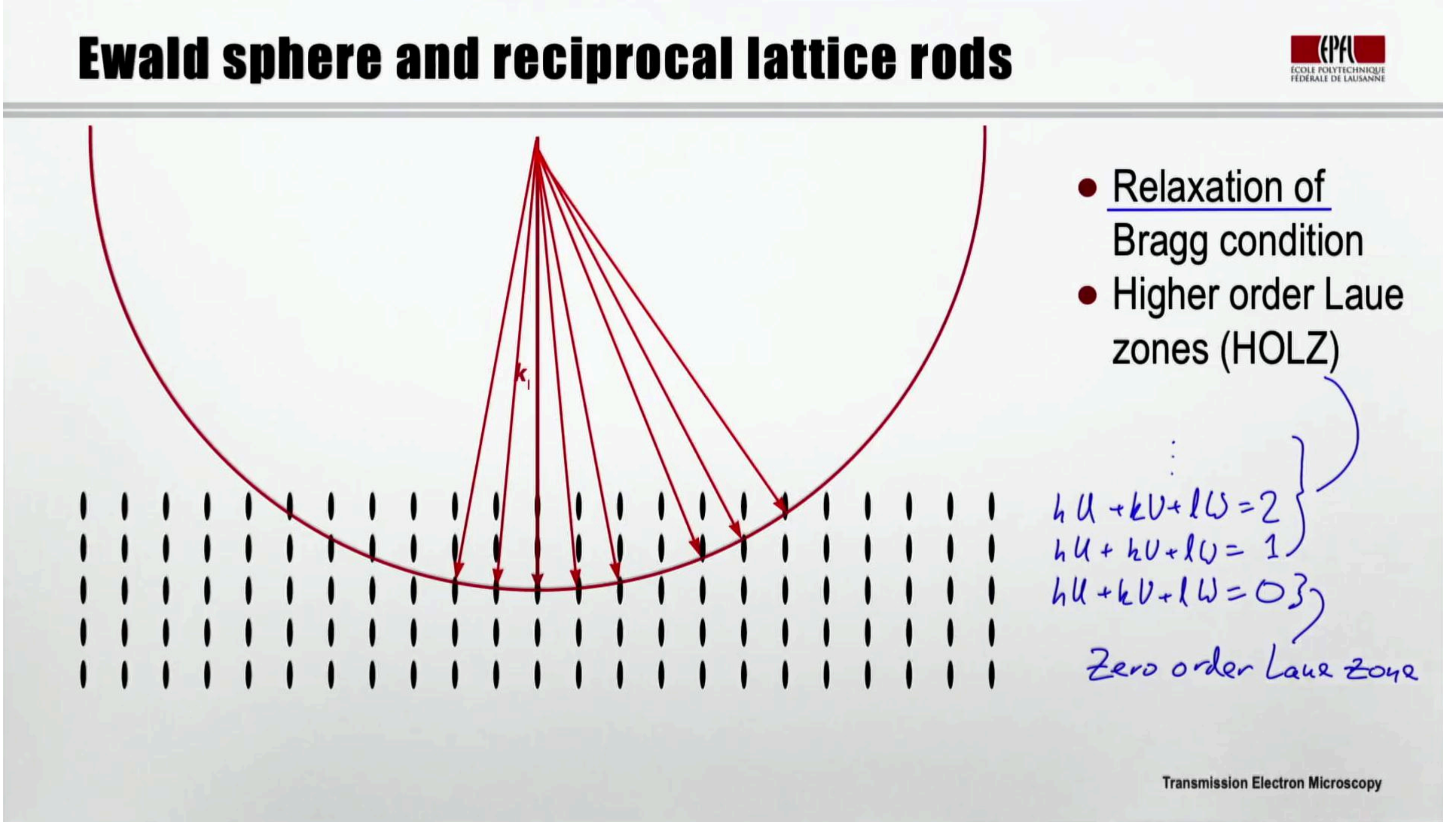


So here I put on that Ewald sphere representation, with the incident wave vector parallel to some important direction in the reciprocal lattice. And we can see straight away that now the Ewald sphere cuts through many of these relrods. Every time it cuts, it meets the diffraction condition, so we see that we can have many different diffracted beams. For instance, we can have one here, you have another here, another here, and another here. So, all these different planes which are parallel to the incident electron beam can diffract because of this relrod geometry. We have what is called a relaxation of the Bragg condition. In order to diffract, planes do not need to be at the exact Bragg condition. They can simply be close to the Bragg condition, and it is because of this that we can observe scattering from many different planes at the same time in a zone axis geometry. For this layer or zone of the reciprocal lattice which is tangential to the Ewald sphere – and for which the planes are parallel to the zone axis vector [U V W] – we can remember that the Weiss zone law h U + k V + l W = 0 applies. However, we can also see that the Ewald sphere can cut other layers or zones in the reciprocal lattice.

Notes

Summary

8m 17s

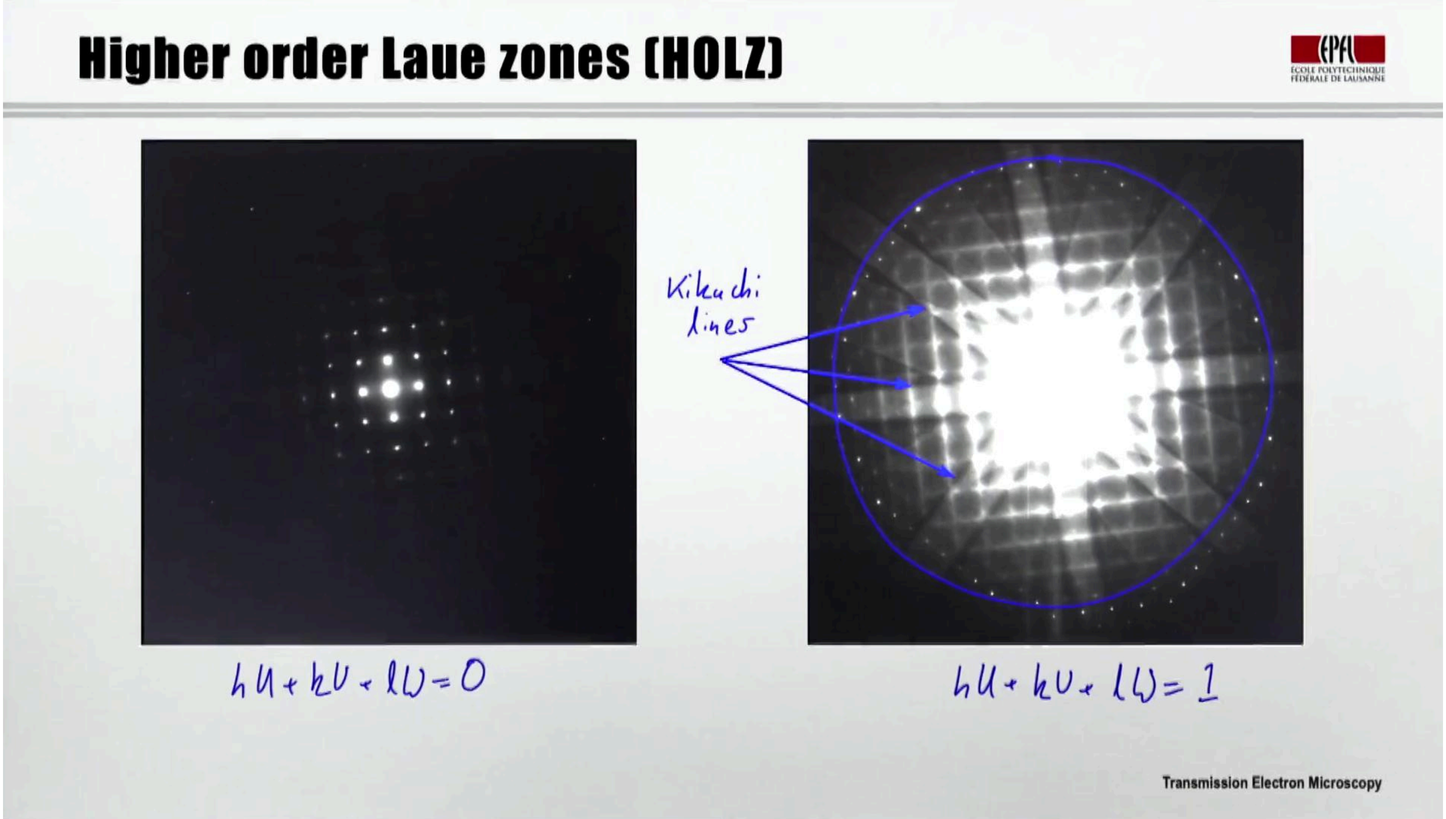


For instance, if we look at this zone, we could have diffraction from that plane and from this plane, and then looking at the next zone up, we can have diffraction from yet another zone. Now, the planes in these zones are no longer parallel to the incident electron beam. They are inclined to the electron beam; and they will have their own zone formulae. So in this zone, h U + k V + l W = 1 applies. And then in the next zone h U + k V + l W = 2 applies. This zone which is tangential to the Ewald sphere, – with h U + k V + l W = 0 – is known as the "zero order Laue zone". Or these other zones which have planes inclined relative to the incident electron beam are known as the "higher order Laue zones". For instance, with h U + k V + l W = 1 being the first order Laue zone. And 2 being the second order Laue zone. And it is this interaction of the Ewald sphere with the three-dimensional reciprocal lattice, which can allow these higher order Laue zones to be visible in electron diffraction patterns.

Notes

Summary

9m 40s

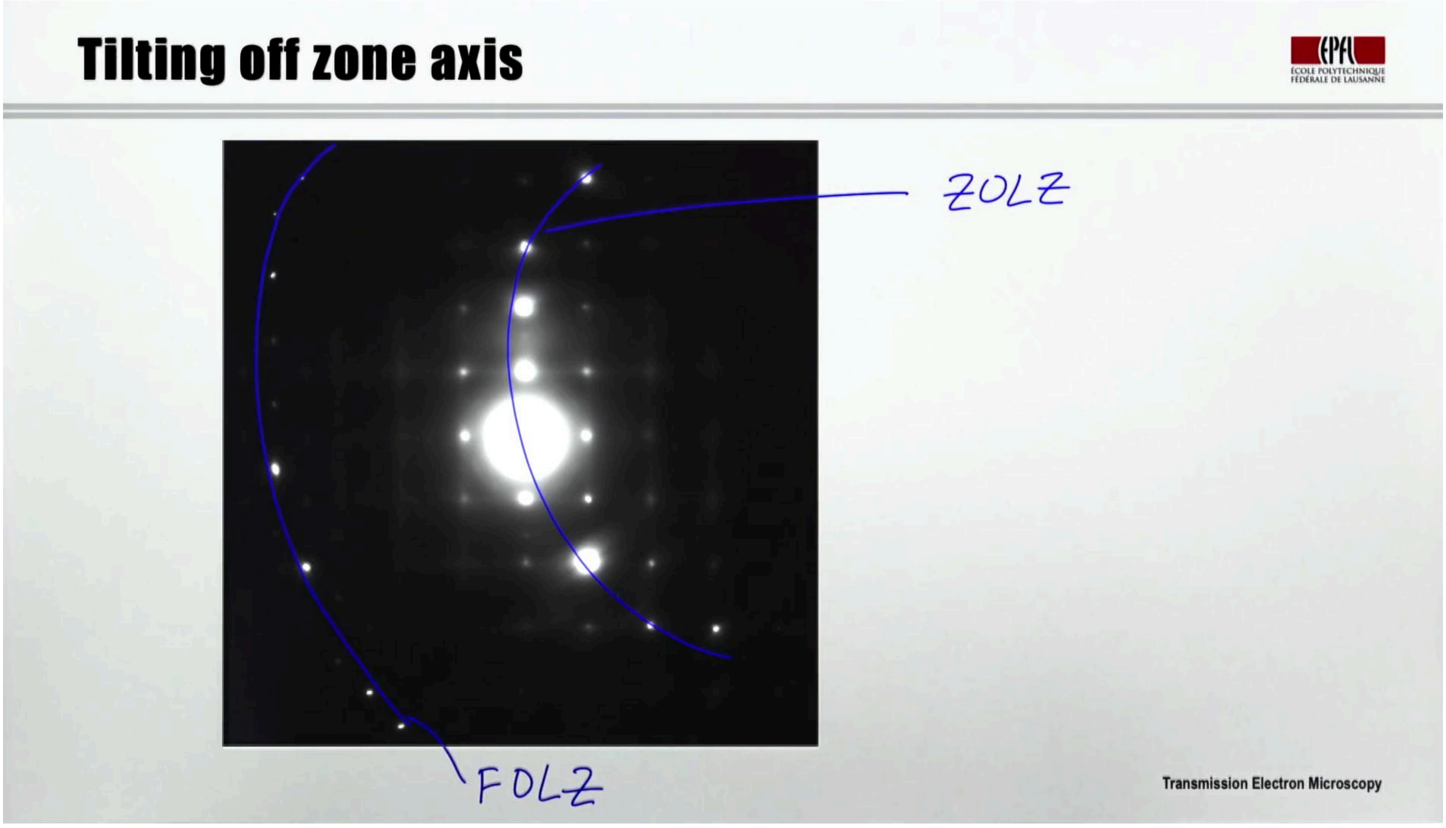


On this slide, I demonstrate this experimentally. Here I show a diffraction pattern from silicon taken on the [0 0 1] zone axis. And here, we are looking at the diffraction spots or reflections immediately around the central beam, the 0 0 0. So in this part of the pattern, we are tangential with the Ewald sphere, with h U + k V + l W = 0. And as we have seen, we have diffraction from many different planes because of the relaxation of the Bragg condition resulting from the form of the relrods. However, looking further out, we can see some little bright spots. If we now take the same pattern but increase the intensity to bring out the intensity in those little spots, we see a circle of diffracting planes. And these are the planes corresponding to the first order Laue zone where we have h U + k V + l W = 1. So for these planes, the Ewald sphere is cutting through the next zone in the reciprocal lattice, giving this scattering at higher angles. By increasing the intensity on that diffraction pattern, we can see something else. You can see all these pairs of dark lines. These lines are known as Kikuchi lines. However, their formation mechanism is a subject for another time.

Notes

Summary

10m 58s

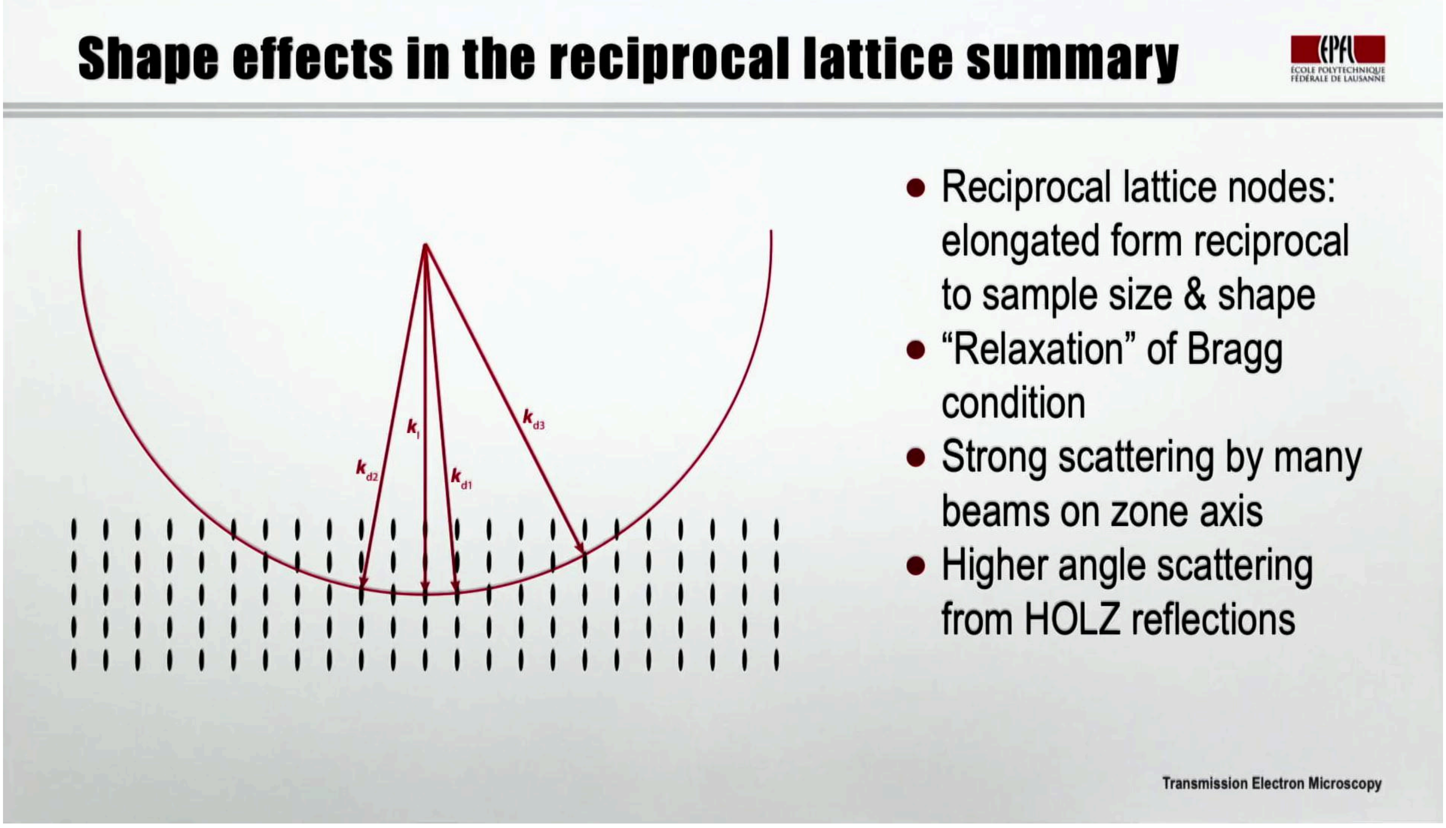


To demonstrate this interaction of the Ewald sphere with the reciprocal lattice, I'm going to tilt away from this nice zone axis diffraction condition that we see here. As we tilt away, you will see how the Ewald sphere cuts through the zero order Laue zone making arcs, and also these higher order Laue zones, such that we can see how this Ewald sphere representation provides an accurate description of the real diffraction phenomena. Looking again at that interaction, here I have a diffraction pattern from the silicon crystal tilted slightly off the [0 0 1] zone axis. And we can see here the Ewald sphere cutting through the zero order Laue zone, and then further out another arc, where the Ewald sphere cuts through the first order Laue zone. Once again, demonstrating how our representation of the Ewald sphere and reciprocal lattice can be very effective for understanding real TEM diffraction.

Notes

Summary

12m 36s



To summarize, in this lecture, I have shown that, because the reciprocal lattice is the Fourier transform of the real lattice, each reciprocal lattice node has a size and shape which are the reciprocal of that of the selected area of TEM sample. And because of this region having a disk-shaped form, we end up with reciprocal lattice nodes which are elongated, the so-called relrods. Because of this elongation, the Ewald sphere can cut through many different relrods at the same time, leading to diffraction from many different planes in one diffraction pattern – a so-called relaxation of the Bragg condition because a plane no longer needs to be at the exact Bragg condition to diffract. This allows us to understand the strong scattering from many different planes at the same time in a zone axis diffraction pattern. And further, it allows us to explain the diffraction at higher angles, which is coming from higher order Laue zones in the reciprocal lattice. In the next lecture, we will look more in detail at the reciprocal lattice rod in order to start understanding intensities in the electron diffraction pattern.

Notes

Summary

13m 40s