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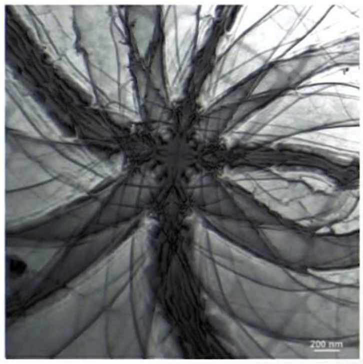
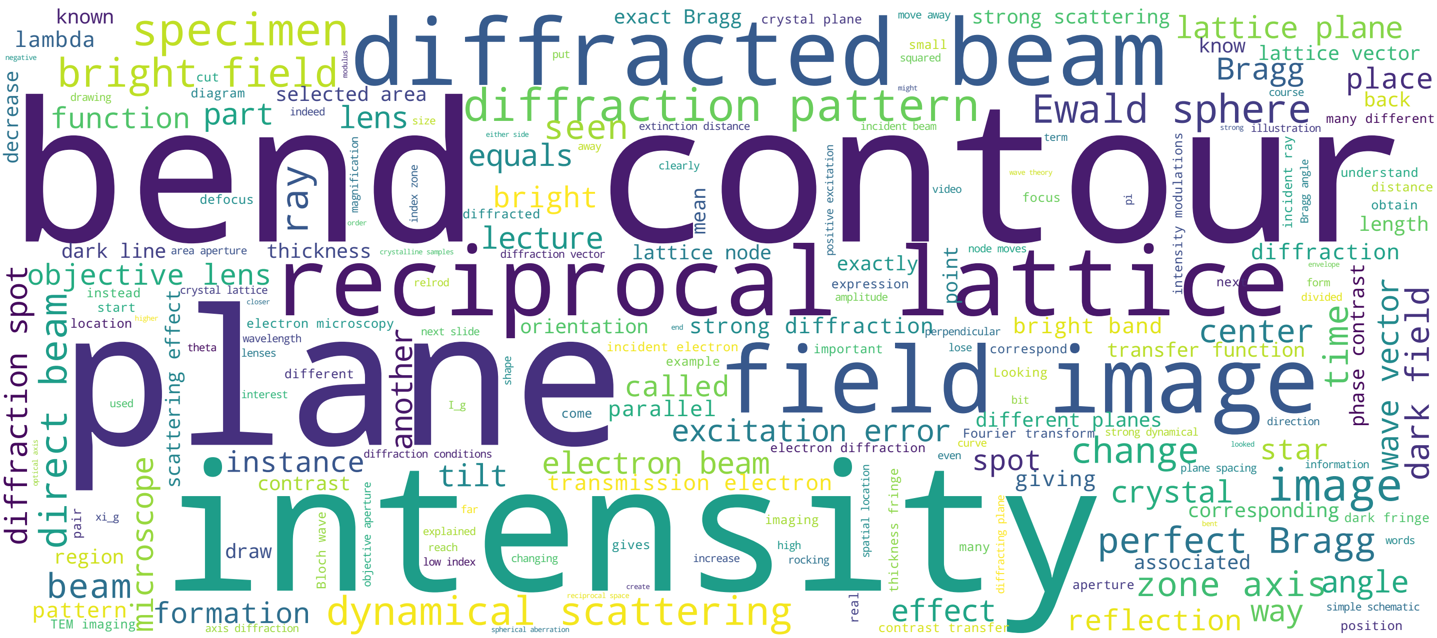


Dynamical effects – bend contours

Transmission Electron Microscopy

Prof. C. Hébert & Dr D. Alexander



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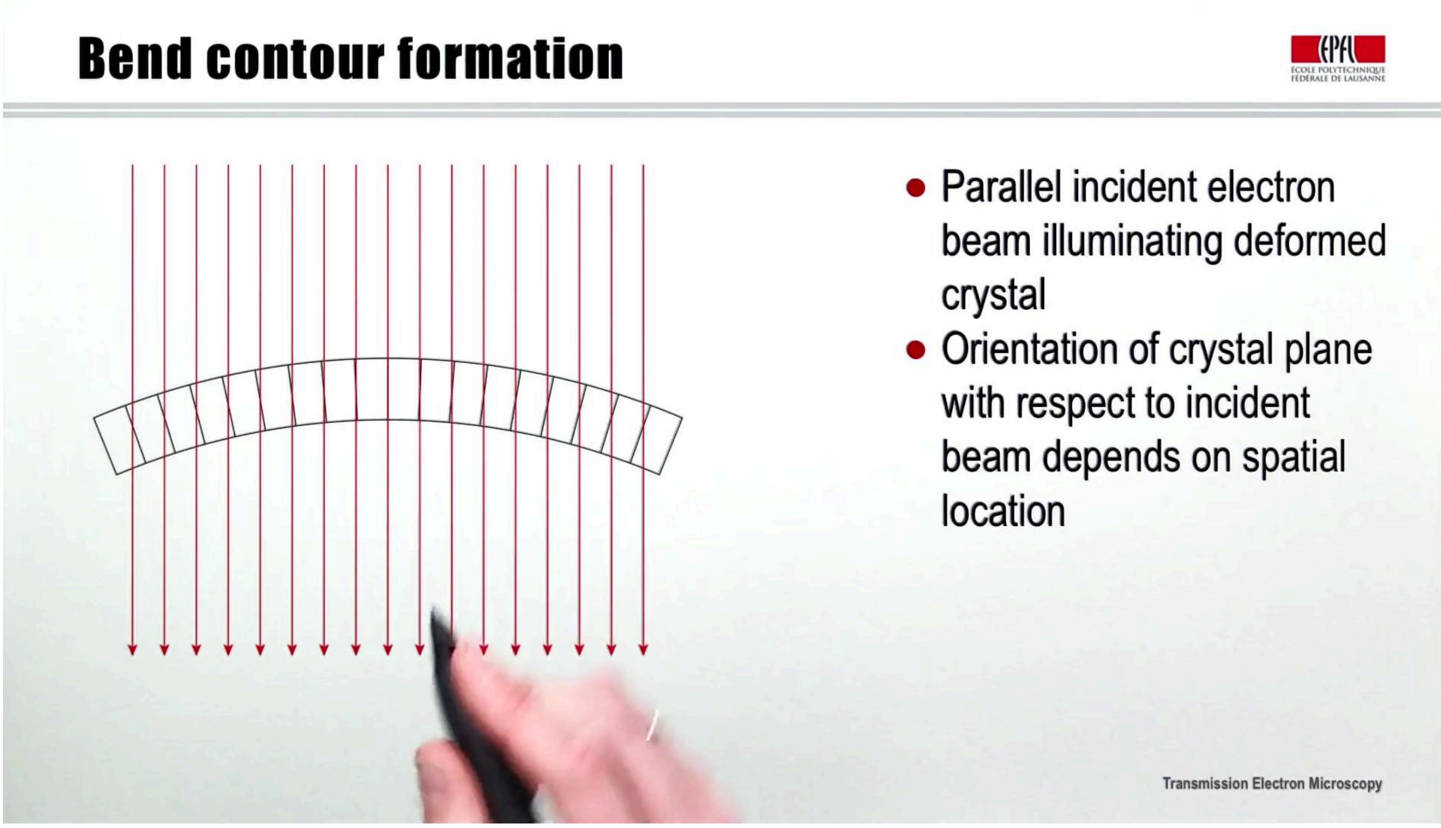
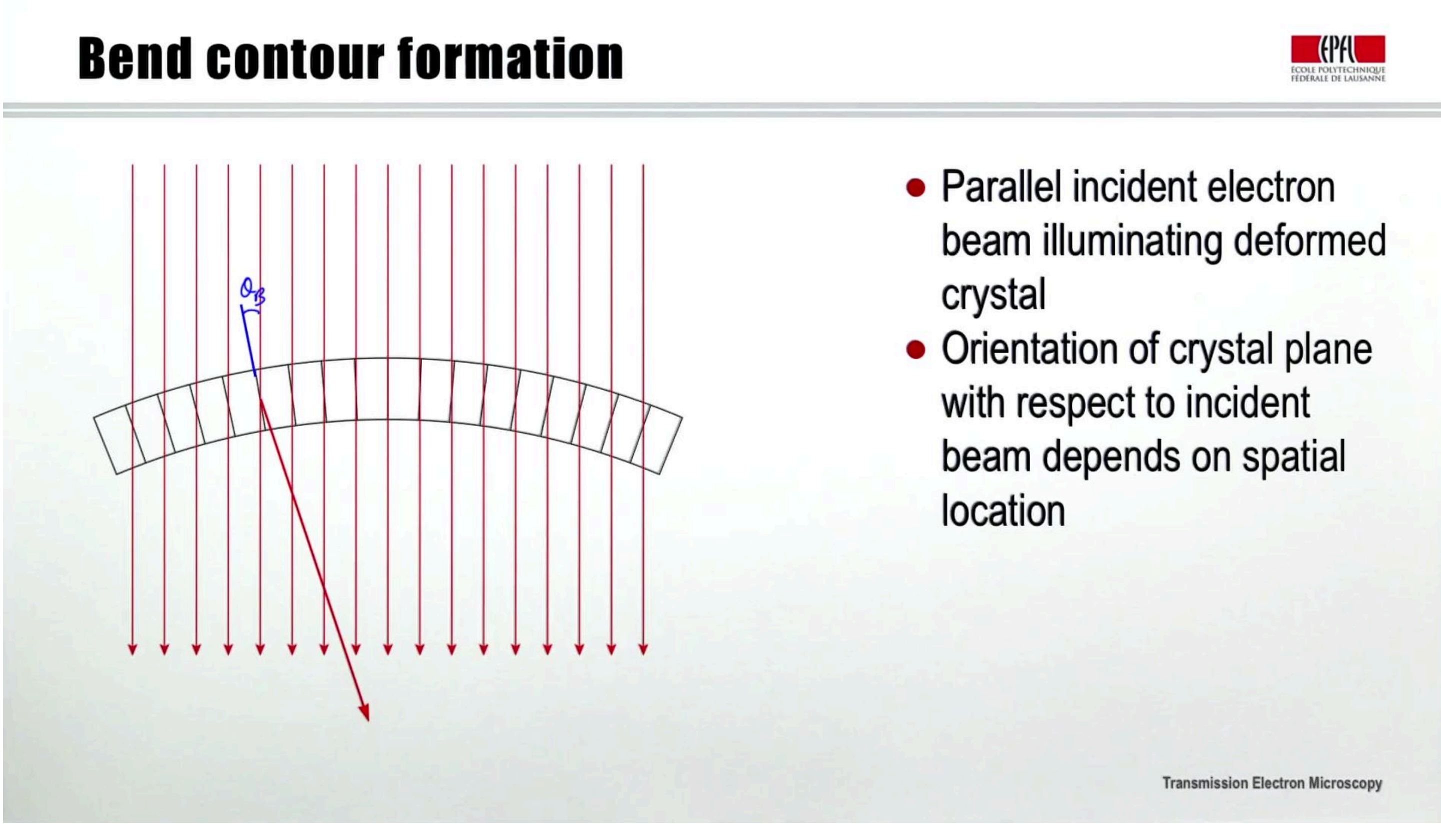


Welcome to CIME’s lectures on transmission electron microscopy for materials science. In the last lecture, I introduced thickness fringes: the patterns of bright and dark fringes seen in crystalline samples of strongly varying thickness, which are also in strong dynamical scattering conditions. In this lecture, I'm going to look at one of the other main consequences of dynamical scattering on regular TEM imaging, in the formation of bend contours. These are contours that are observed in crystalline samples which are locally deformed or bent, thus changing their crystal orientation with respect to the electron beam, depending on spatial location. Which in turn gives a spatially-dependent diffraction condition. To understand the formation of bend contours, we can look at this simple schematic of lattice planes in a deformed crystal. If we draw on some parallel incident rays, we can immediately see that the orientation of this lattice plane, with respect to the incident ray, depends on the position of the ray on the sample. Marking on a number of parallel rays, this becomes even more obvious. In the center here, the ray is perfectly parallel with the lattice plane.

Notes

Summary

0m 04s



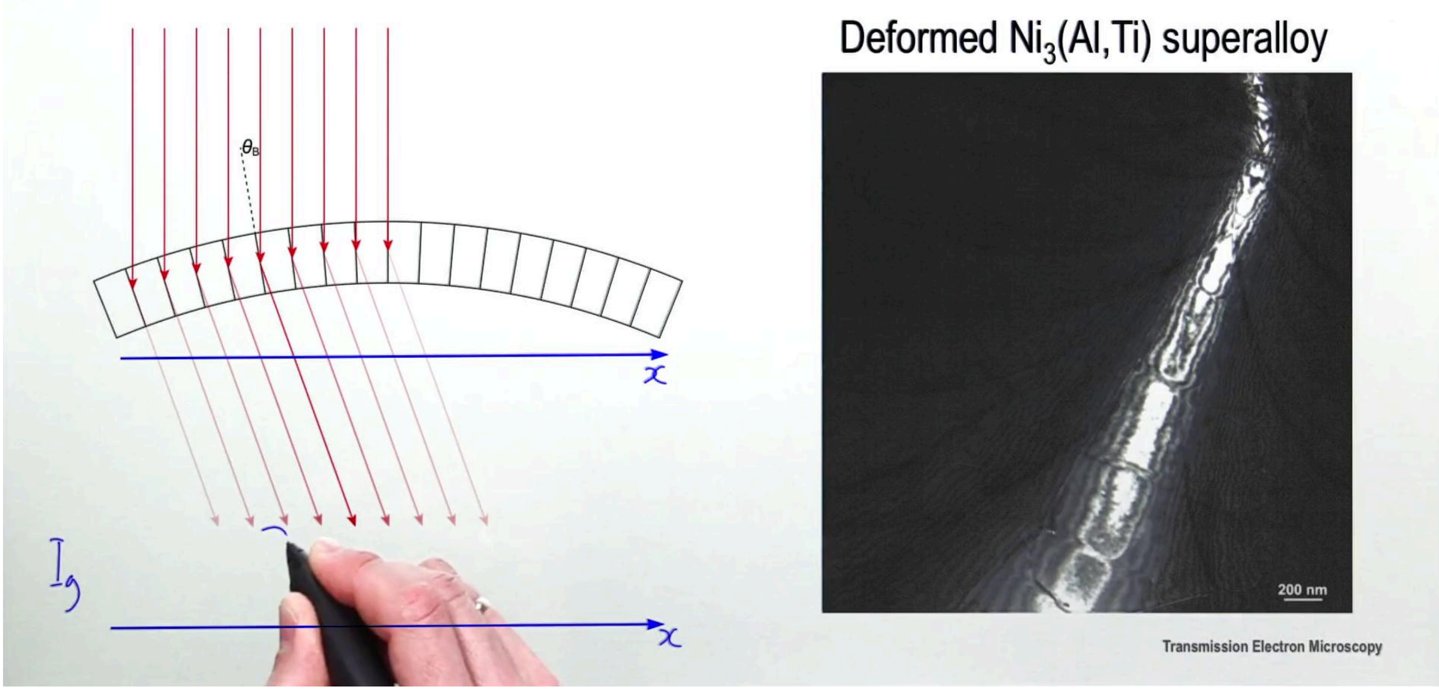
Moving away from that central location in either direction, we can see that the angle between that incident ray and the lattice plane gradually increases. Thus we can see that the diffraction condition of this plane will vary locally as this angle changes. For instance, it might be that here the plane is in the perfect Bragg condition, thus there will be strong scattering of that ray into a diffracted beam.

Notes

Summary

1m 31s

Bend contour formation (dark-field)

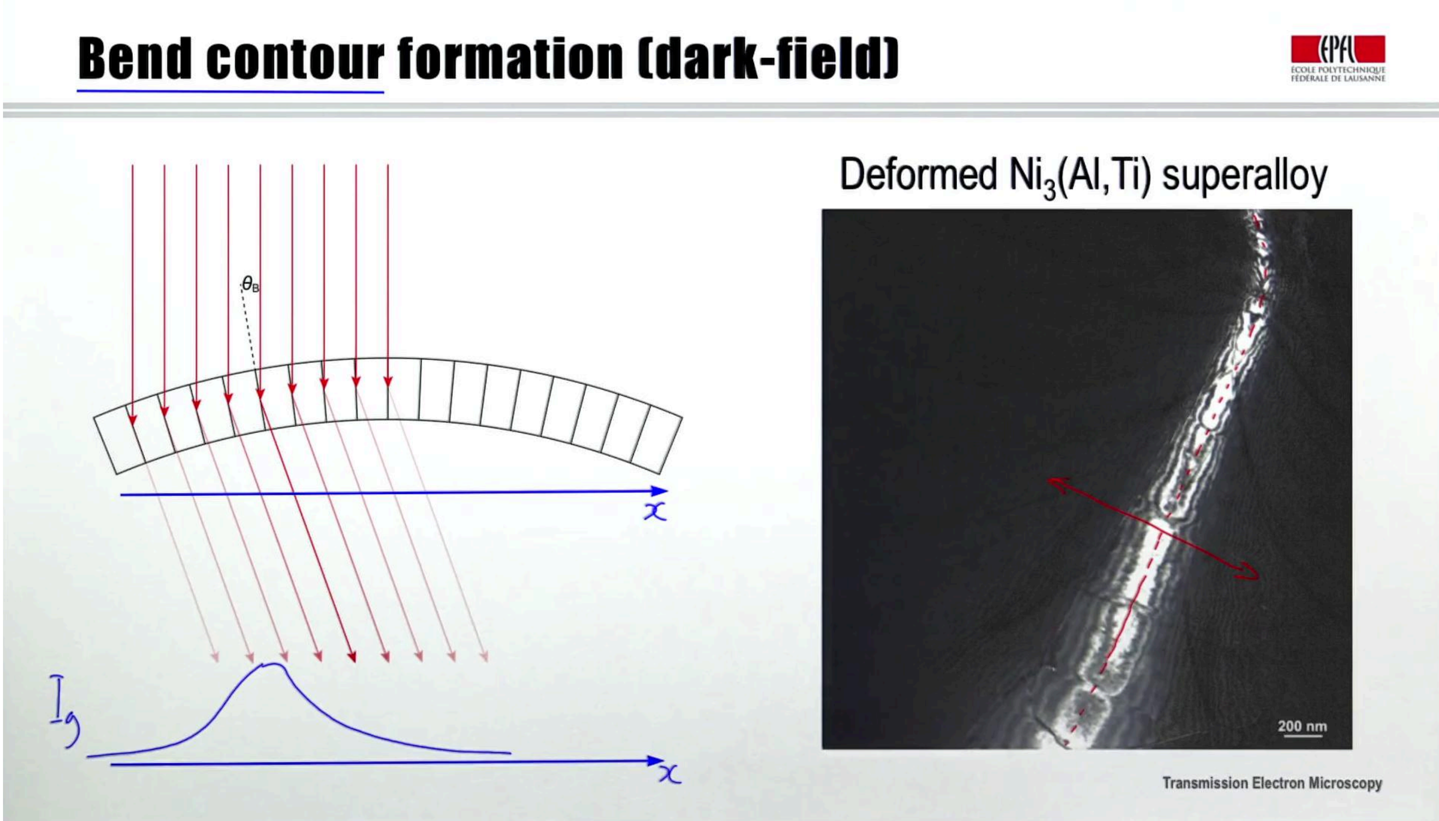


We will first look at the effect of that spatially-dependent diffraction condition on dark-field imaging. To consider this, we will just look this part of the schematic, around the plane which is in the Bragg condition at this place on the sample. Therefore here, there will be strong scattering into a diffracted beam, giving the wave vector k_D. Considering that there will also be transmission of the incident or direct beam, the sample will give a two-beam diffraction pattern with one reflection 0 0 0 – the direct beam – and another for the diffracted beam, g. As we move away from this spatial location, the plane loses that perfect Bragg angle with the incident beam. Thus while it will still scatter with this diffracted wave vector, the intensity in the diffracted beam would decrease. This is illustrated in a simple conceptual way here, where we have strong scattering into the diffracted beam at this location, weakening as we move either side from that location. So, if we plotted the intensity in that diffracted beam relative to some spatial axis x, we would see here there would be a strong intensity in that diffracted beam.

Notes

Summary

2m 01s



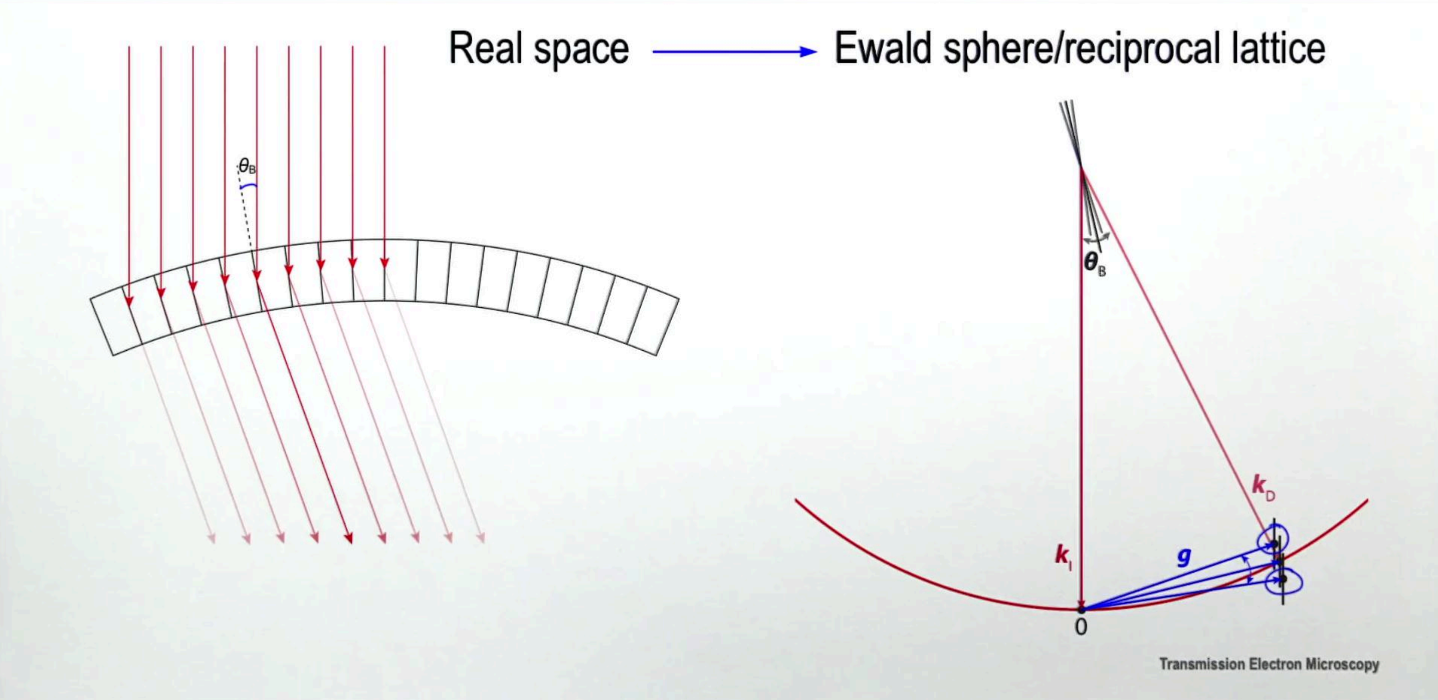
But as we move away in x, that intensity would decrease as we lose that strong diffraction condition, thus giving a profile like this. Looking at a real dark-field image of a deformed crystalline sample, this is indeed what we see. The sample in question is nickel_3 (aluminum, titanium) super alloy. And here we have a bright band. This bright band corresponds to places where the sample is at the exact Bragg condition. As we move away from this band, because of the curvature of the crystal, we lose that strong diffraction condition. Thus the intensity in the dark-field image decreases corresponding to the decrease in I_g, the intensity in the diffracted beam. And it is this bright band which is known as a "bend contour". While, at first, it may look like a simple bright band, if we look closer we can see that there are actually complex patterns of bright and dark fringes associated with this bend contour. These derive from dynamical scattering effects, as now explained.

Notes

Summary

3m 37s

Bend contour formation (dark-field)

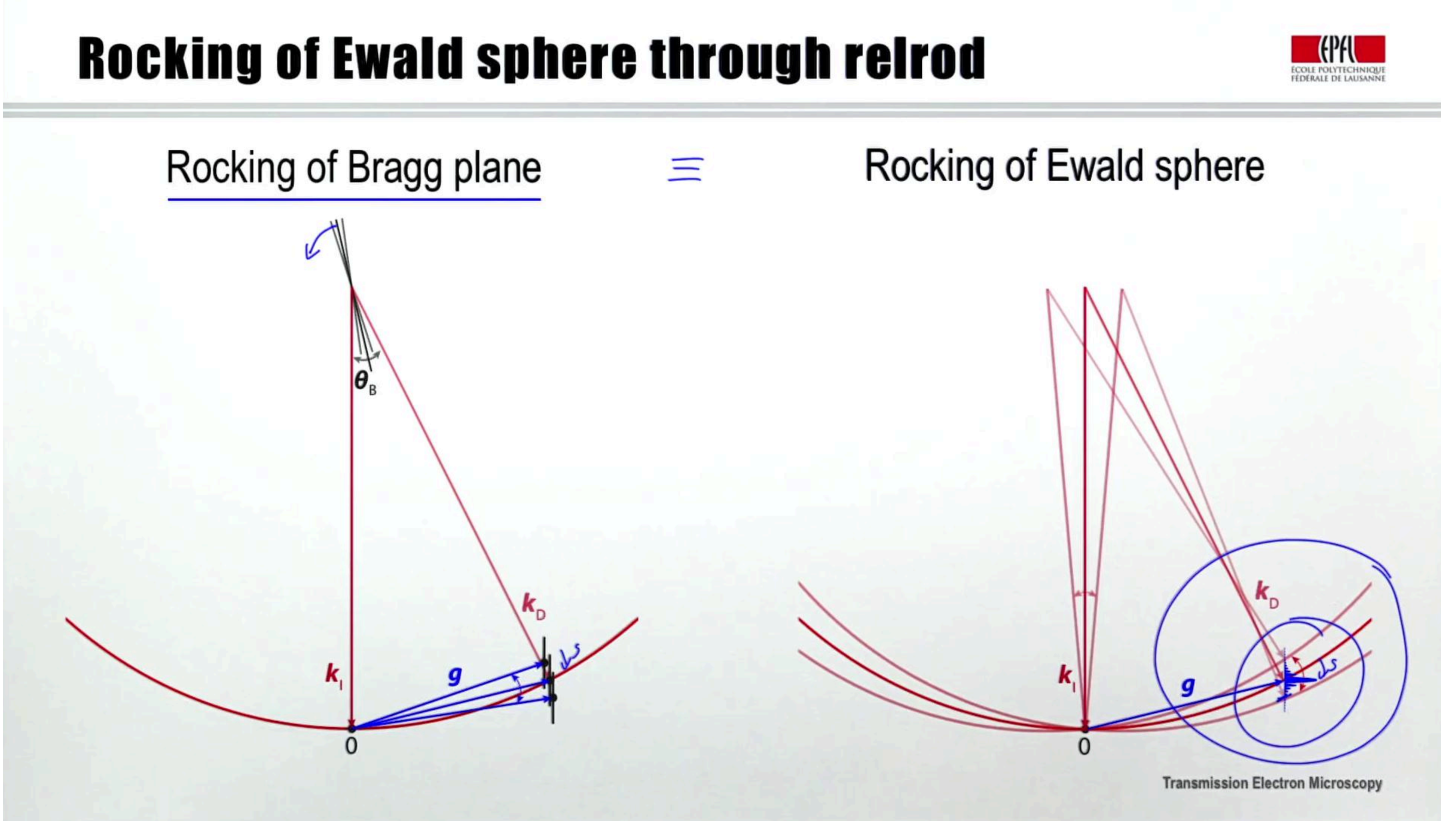


To explain these dynamical effects, we go from this real space illustration to instead considering the reciprocal lattice and the Ewald sphere construction. When the lattice plane is at the perfect Bragg condition, we know that the reciprocal lattice node for this reflection is intersected exactly by the Ewald sphere. However, as this plane tilts either side of this perfect Bragg condition, corresponding to the change in inclination from the bending of the crystal, the reciprocal lattice node moves up or down. So as the plane tilts this way, the node moves up, and as it tilts that way, the node moves down. Here giving a positive excitation error and here a negative one.

Notes

Summary

5m 00s



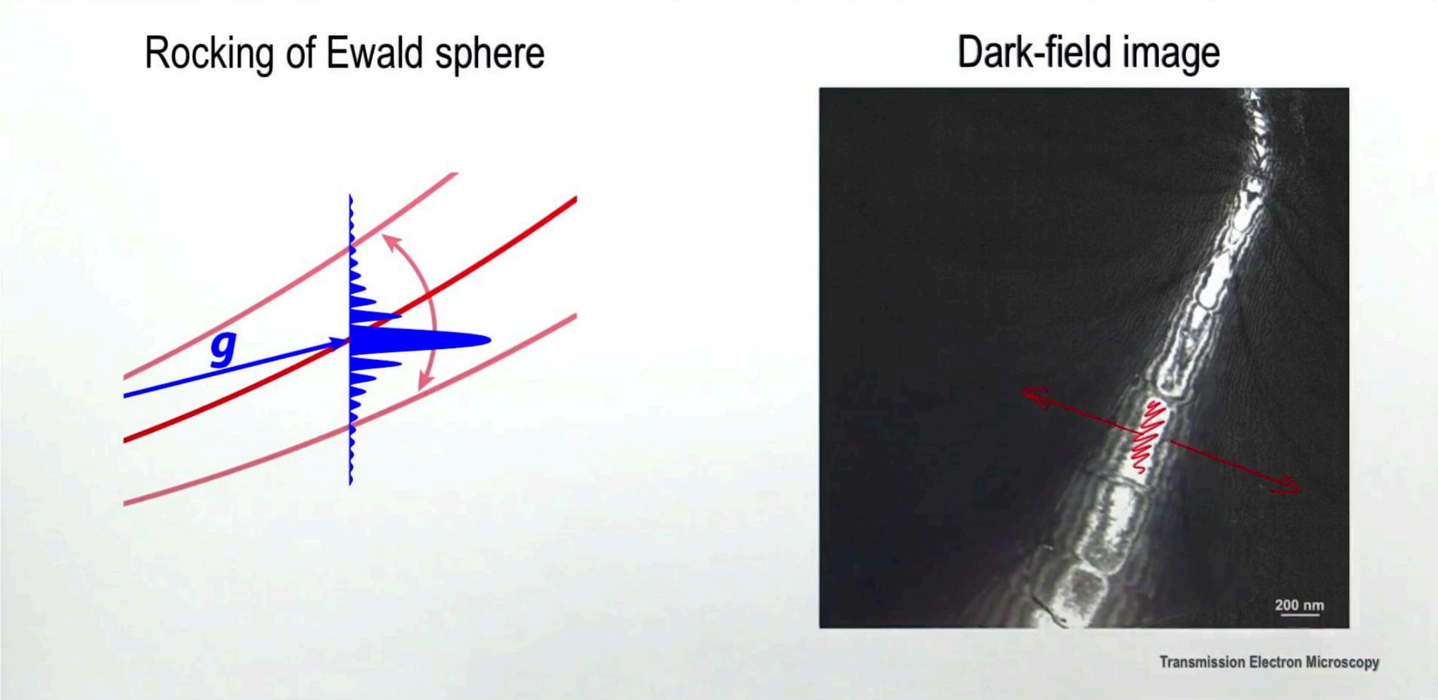
This change in local diffraction condition can be described as a rocking of the Bragg plane. This is equivalent to a rocking of the Ewald sphere, as demonstrated on this diagram here. Here, instead of moving the reciprocal lattice, I have corresponded the tilts of the lattice plane to tilts of the Ewald sphere. For instance a tilt of that lattice plane in this direction leads to a positive excitation error. Equally, if the Ewald sphere is tilted this way, while keeping the reciprocal lattice in one place, we create a positive excitation error; as seen here. In this illustration, I have further replaced that schematic relrod with a little plot of the intensity variations of the diffracted beam against excitation error s, given by the dynamical scattering equations. And by zooming in on this part of the illustration in the next slide, we can start to understand better the pattern of intensity seen in the bend contour on the dark-field image.

Notes

Summary

6m 01s

Rocking of Ewald sphere through relrod

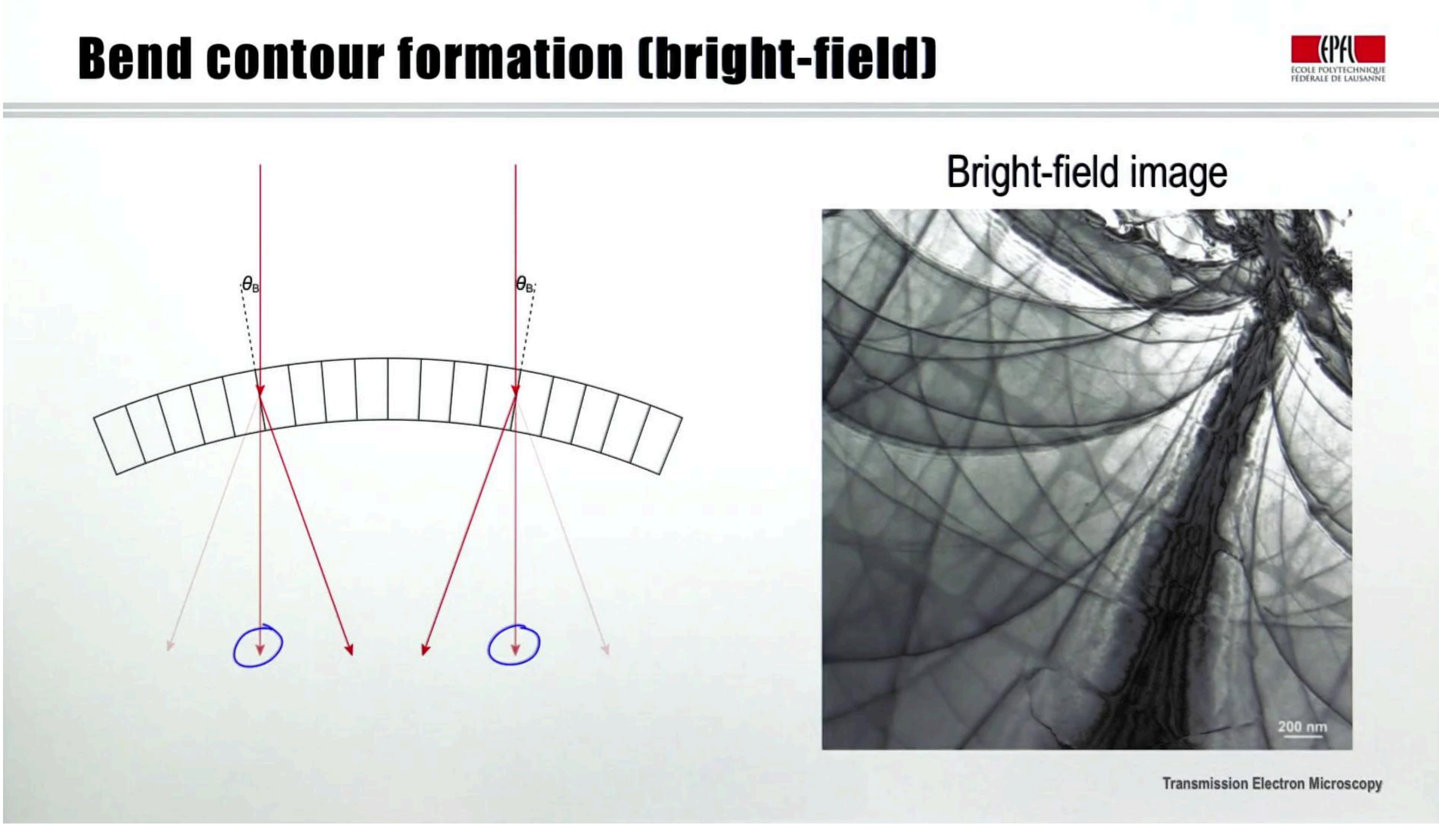


What we see is that, as a Bragg plane rocks, the Ewald sphere will cut through different parts of the intensity modulations along the length of the relrod. Thus in the center, where the crystal plane is at the perfect Bragg condition, we have a broad bright band. Then, after that we have dark and bright fringes corresponding to the modulations on this curve. These oscillations are seen a long way out from this perfect Bragg condition. Clearly, the distance between the modulations will depend sensitively on the local curvature of the crystal lattice. So the more curved, the closer together they would be. Also of course, the intensity modulations will depend strongly on the crystal thickness. For instance, if the crystal has a thickness corresponding to an integer multiple of the extinction distance, then we would have a dark fringe in the center of the bend contour.

Notes

Summary

7m 20s

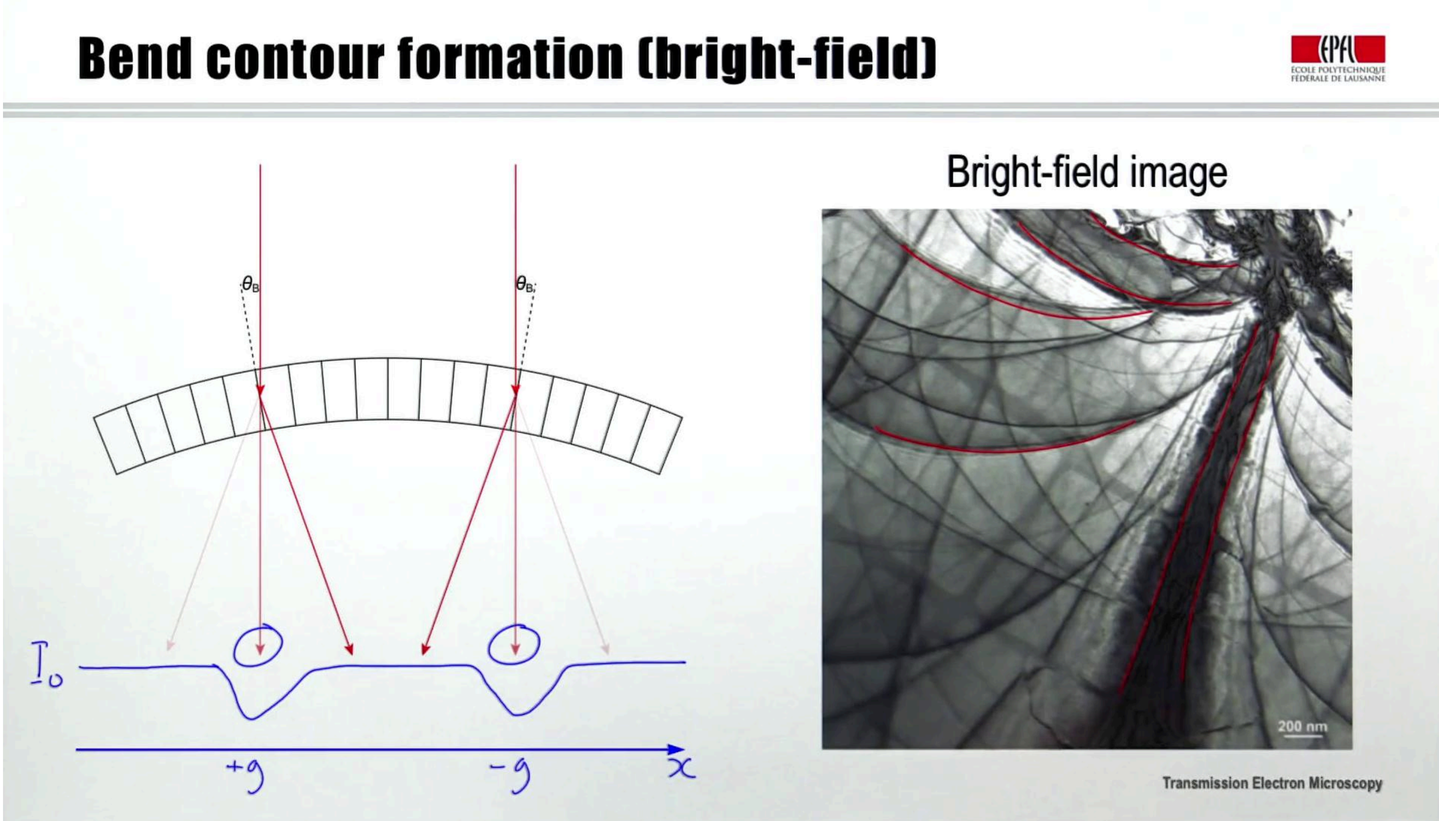


We now instead consider the formation of bend contours in the bright-field image. If we go back to our simple schematic, we can see that there are two locations where the crystal plane achieves the Bragg condition. If we say reflection at this angle corresponds to the plus g diffraction spot, then the reflection at this angle, which gives a ray in the opposite direction, corresponds to the minus g. Thus in our diffraction pattern, we would now consider a central spot with the direct beam, another for g, associated with this diffracted ray, and then a third for minus g, associated with this ray. Thus, we can simplify the schematic to consider only these three rays: the direct beam, g, and minus g, as seen on the next slide. On this simple schematic showing those three rays, we can see that, at the two locations where we have the perfect Bragg condition, there is strong scattering into a diffracted beam. Equally, this will remove electrons from the direct beam: the beam that interests us for bright-field image formation.

Notes

Summary

8m 29s

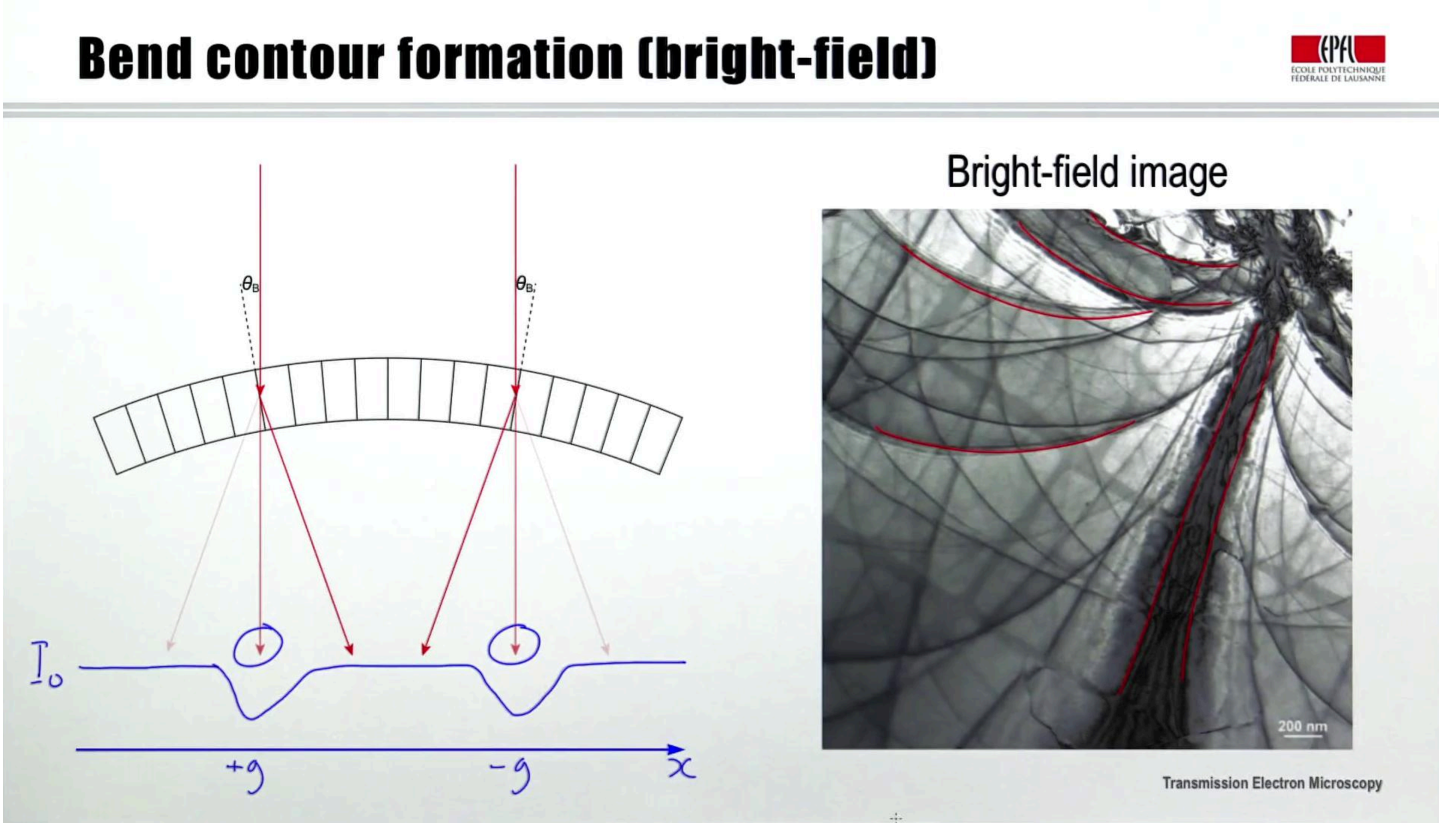


Now, if we make a plot of the intensity in that direct beam, with respect to spatial location, when we are far from a strong diffraction condition, the intensity will be high, and then, as we reach this strong diffraction condition, which gives more intensity in the diffracted beam, the intensity in the direct beam will decrease. It will then increase again. But, as we reach the minus g orientation, it decreases again. Thus, associated with this plane, we should see two dark bend contours in the bright field image: one for the plus g diffraction condition; and one for the minus g diffraction condition. And if we look at this bright-field image, that is exactly what we see. Here, we have the bend contour for the plane discussed in the dark-field image, and here we have one dark line for the plus g, and here we have another dark line for the minus g. We can also see that we have many other bend contours for other diffracting planes. We have a plus g minus g pair here, another pair here, and so on. Because of dynamical scattering effects, we can see that there are also complex intensity modulations; modulations that could, for instance, be modelled by simulations using Bloch wave theory.

Notes

Summary

9m 55s



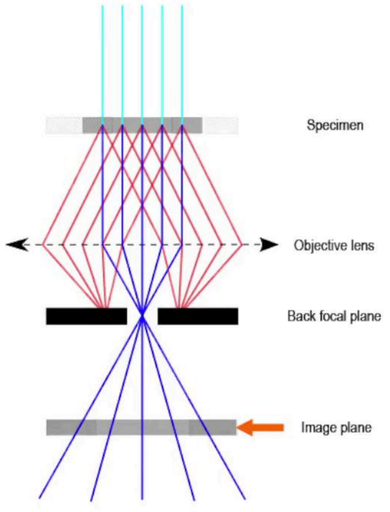
The higher the index of the scattering plane, the weaker the probability of scattering. Because of this, high index planes have less dynamical scattering effects and give much sharper bend contours. So on these higher index planes, we just see sharp, dark lines, where the plane meets the exact Bragg condition. If we tilt the crystal by tilting the sample, this will change the diffraction condition locally. Thus, the bend contours will move across the image, as the places in the sample which are at the perfect Bragg condition change.

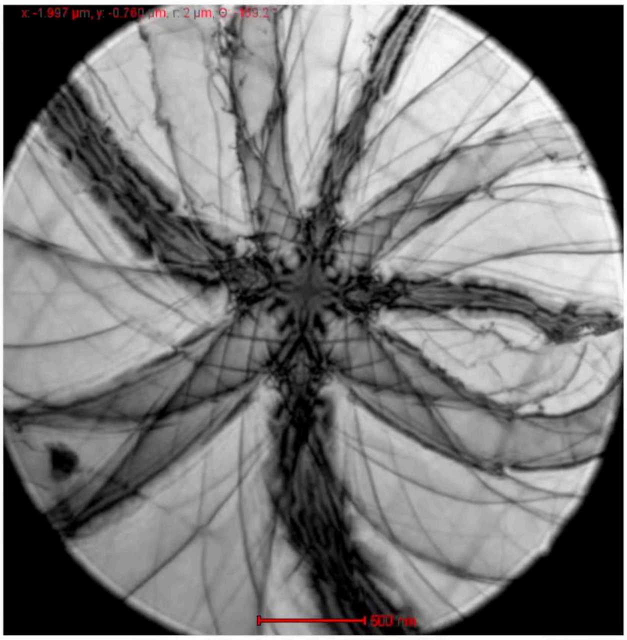
Notes

Summary

11m 27s

Bend contour demo (bright-field)





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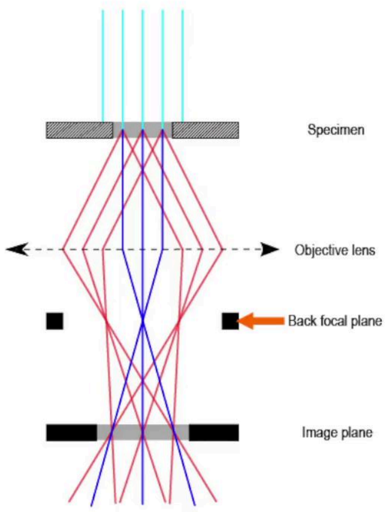
This I demonstrate with this movie. While imaging in bright-field mode, I now start tilting the sample. As it tilts, we can see the bend contours moving, as the local diffraction conditions change. I now stop tilting and, instead, translate the sample over to this region here, where we see many different bend contours coming together. With a bit of extra tilt and translation, I center that region and then insert a selected area aperture.

Notes

Summary

12m 08s

Bend contour demo (bright-field)



Transmission Electron Microscopy

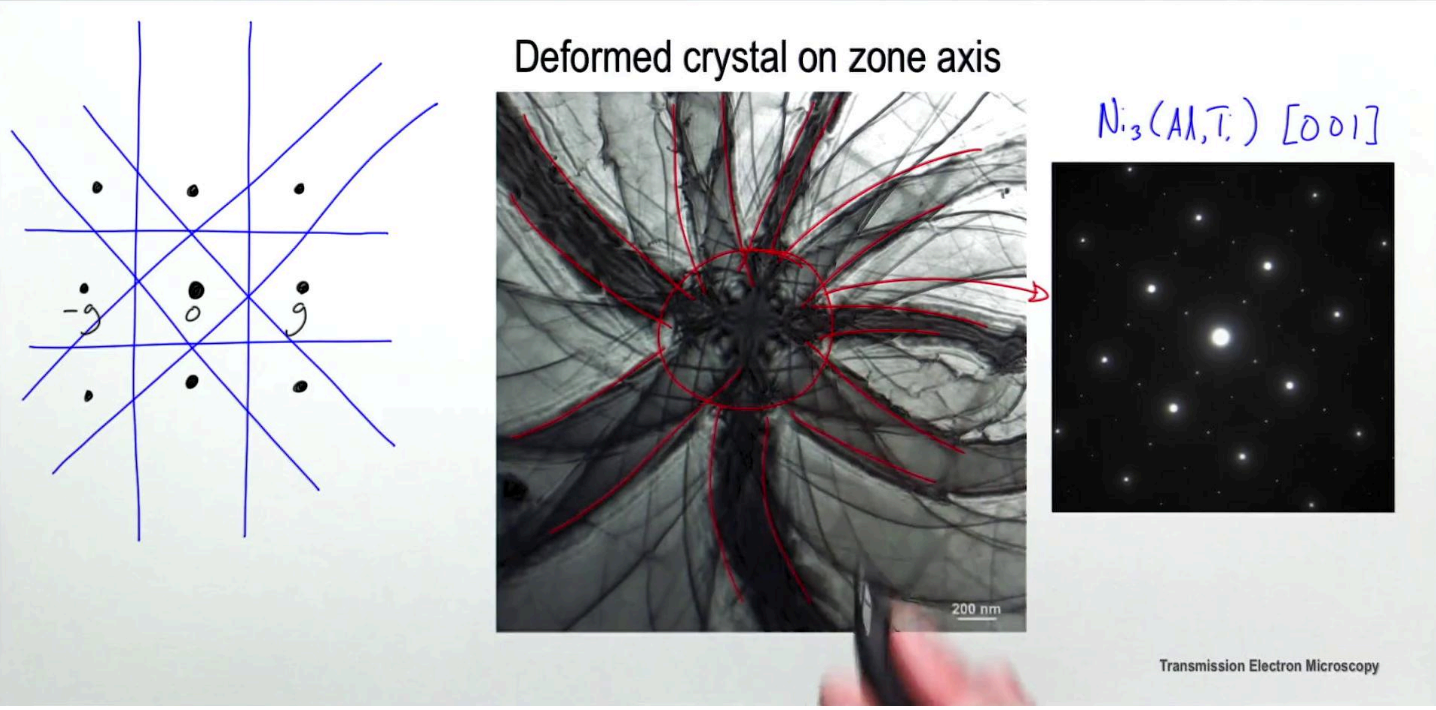
Now, going to diffraction mode, I remove the objective aperture, which had been forming the bright-field image, and we see we have a perfect zone axis diffraction pattern. The reason for this is now explained.

Notes

Summary

12m 42s

Zone axis condition

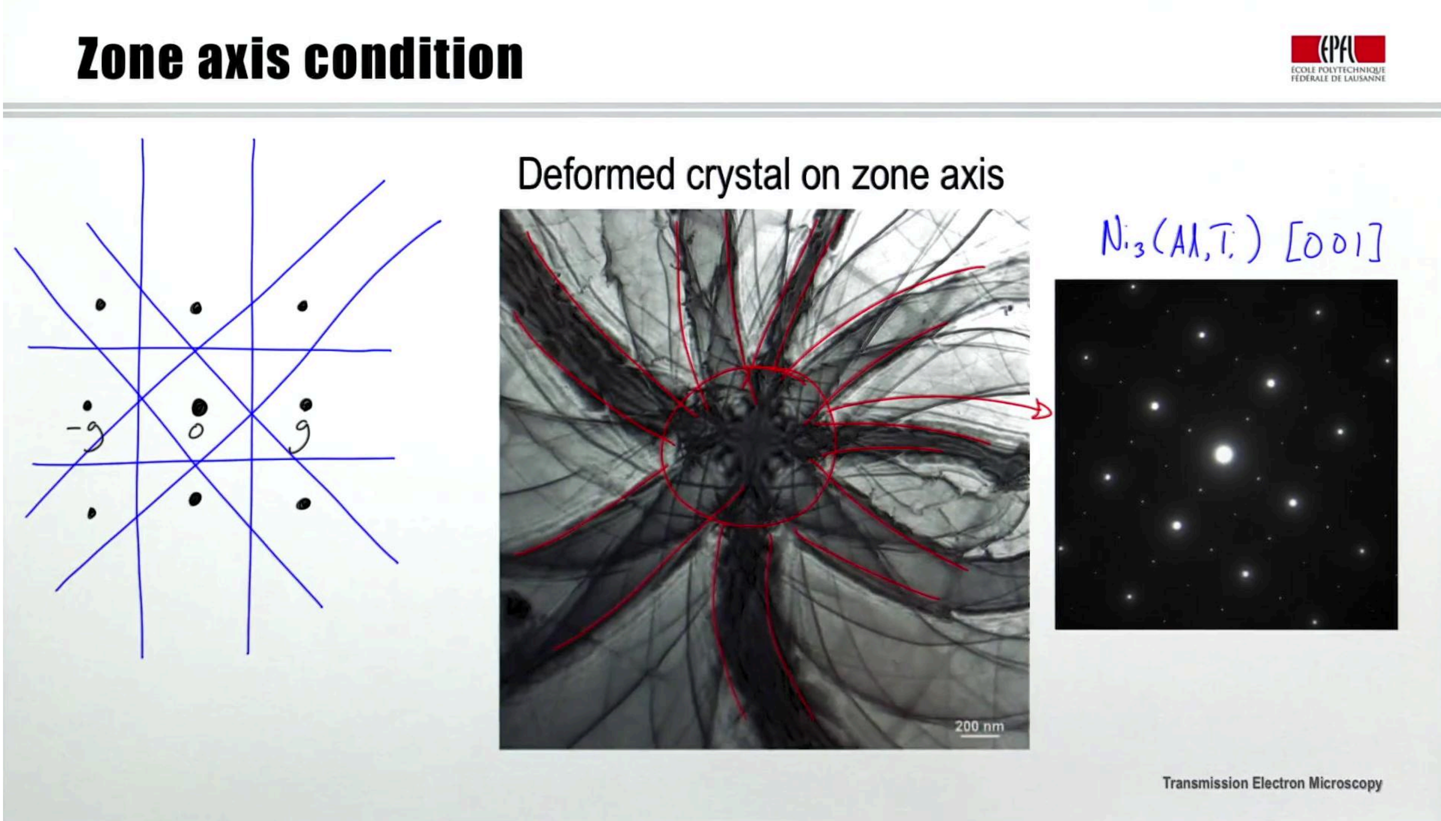


We have seen already that for one plane with a plus g and a minus g, in the bright-field image, we obtain bend contours perpendicular to their corresponding diffraction spots. If we now consider a low index zone axis, there will be diffraction from many other planes. So this next plus g and minus g pair of spots will, in turn, have corresponding bend contours like this, and like this, in the bright-field image. And we can go on considering diffraction from other planes, marking in their respective diffraction spots and their schematic bend contours. Where in the diffraction pattern, we have scattering from many different planes at the same time, in the bright-field image, there will be a convergence of bend contours associated with these different planes. And that is exactly what we see on this bright-field image of bend contours, with different plus g and minus g pairs converging on this zone axis. Confirming this, when we take the selected area diffraction pattern from this region of interest in the center, we obtain a perfect zone axis diffraction pattern, here of the nickel_3 (aluminum, titanium) superalloy, on the [0 0 1] zone axis. Looking closer, in the center, we see very complicated patterns of fringes.

Notes

Summary

12m 56s



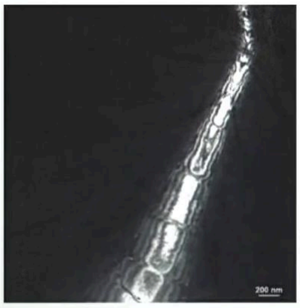
These are from strong dynamical scattering. Because of the complexity of the dynamical interactions between all these different planes, it would need simulations made, for instance, using block wave theory, to model this dynamical scattering pattern.

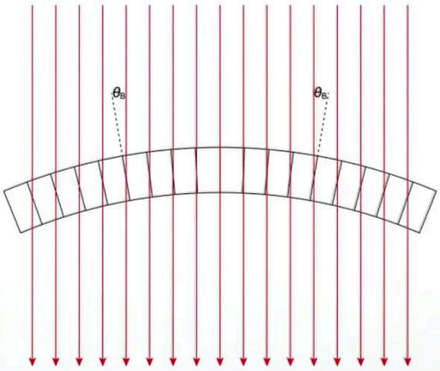
Notes

Summary

14m 38s

Dynamical scattering – bend contours summary





- Deformation of crystals produces locally changing diffraction condition
- Dark/bright bend contours when lattice planes meet Bragg condition
- Bend contours may show strong dynamical effects
- Impede imaging at precise diffraction conditions

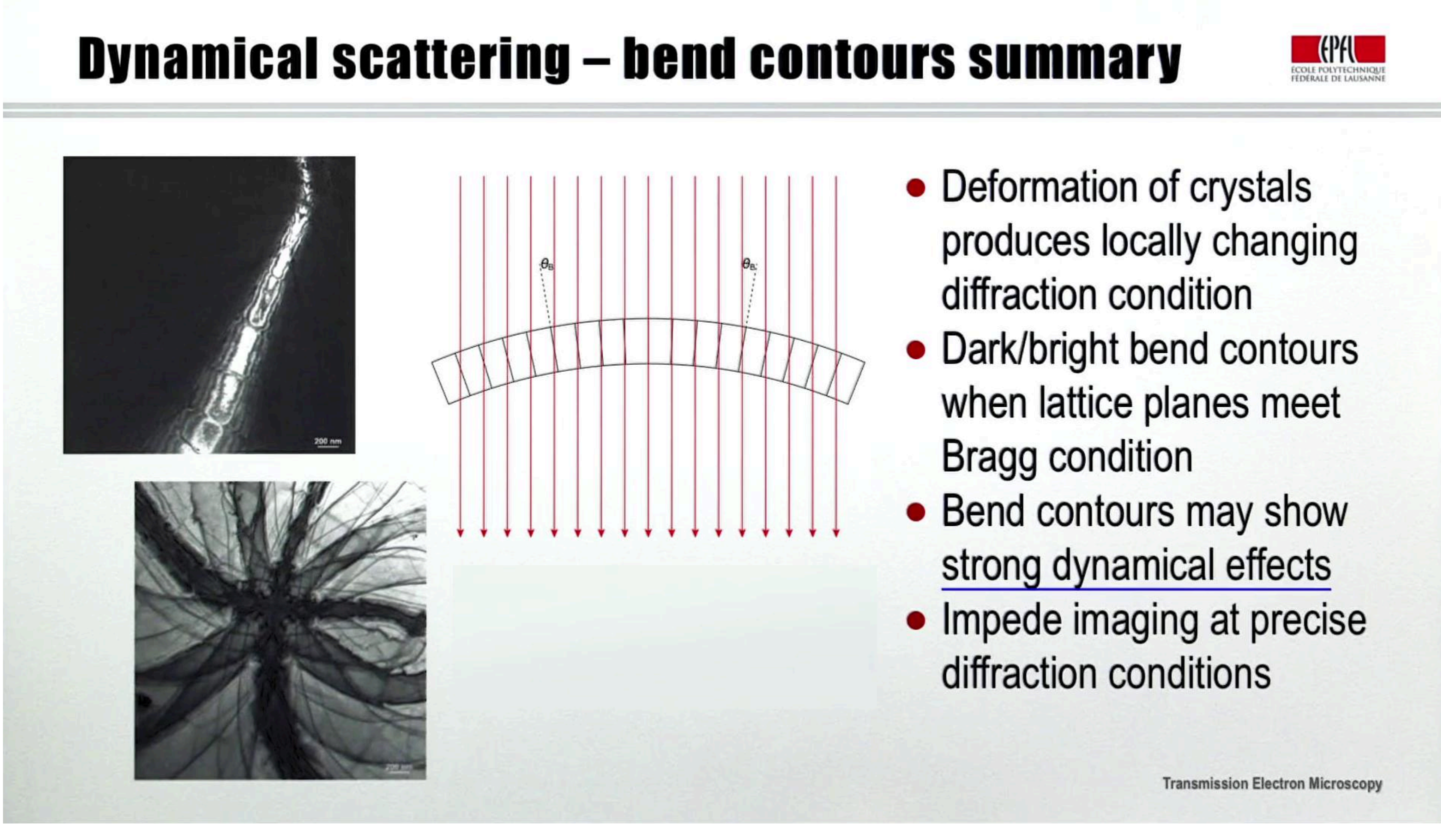
Transmission Electron Microscopy

To summarize, when a crystalline TEM sample is deformed or bent, the orientation of a crystal plane, relative to the incident electron beam, changes locally. This results in the formation of bend contours, as the sample comes into and leaves strong diffraction conditions. In dark-field images, bright bend contours are seen when a plane matches the exact Bragg condition. Conversely, dark bend contours are seen in bright-field images. In the bright-field image, each plane essentially has two dark lines, one for the plus g diffraction condition, and another for the minus g condition. Plus g minus g pairs of lines from different planes converge on low index zone axes. These bend contours may show strong dynamical scattering effects, which can be associated with dynamical scattering theory. Because any bent crystalline sample may show these strong contrast effects, they can have important consequences for standard TEM imaging, since samples thinned to electron transparency are often deformed in this way. Finally, while bend contours can be useful for helping orientate our sample, in general, the crystal bending that creates them impedes our ability to take TEM images at precise diffraction conditions.

Notes

Summary

14m 57s

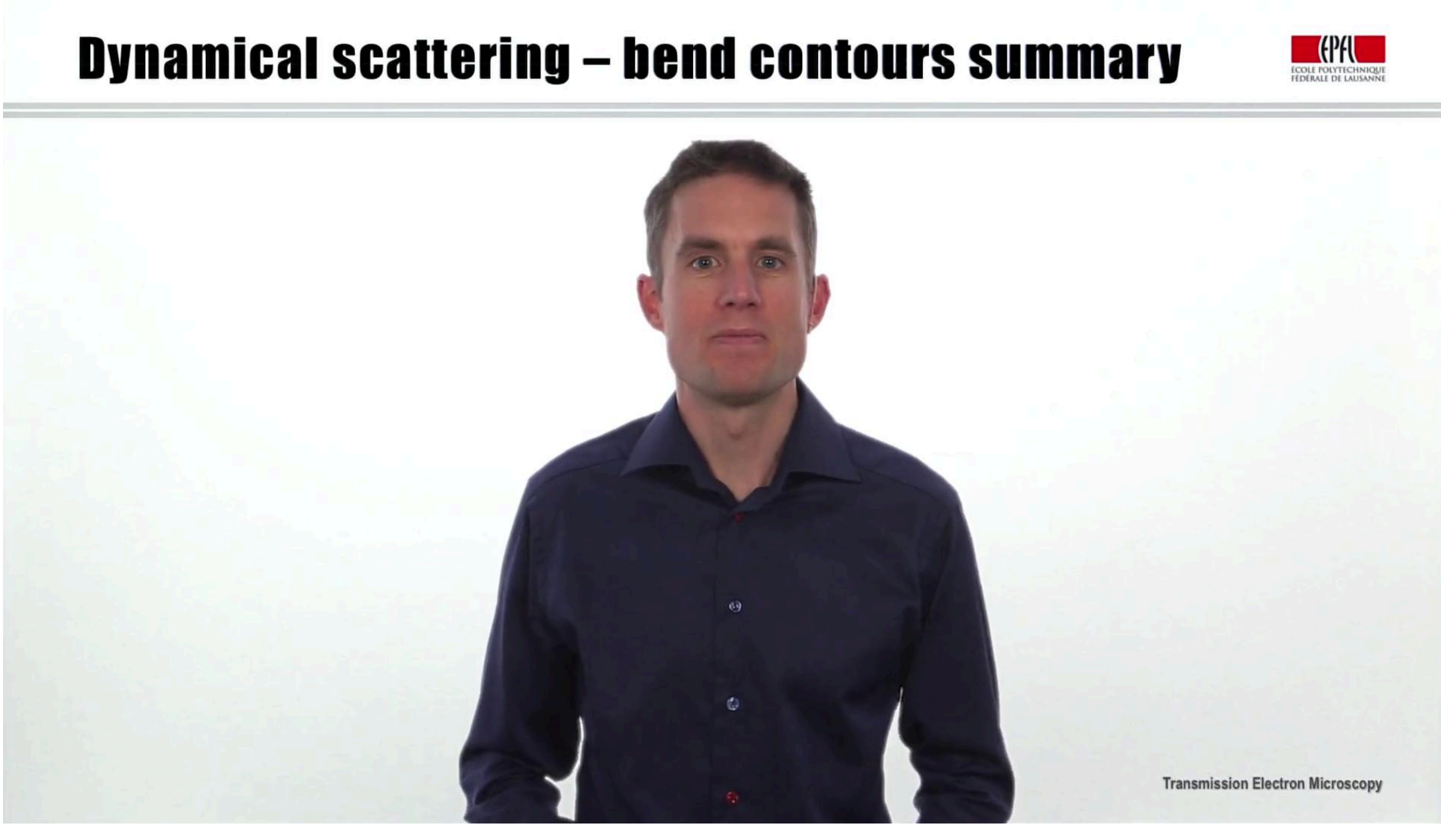


For instance, when a dark-field image only has strong bright contrast in the narrow zone of the bend contour, it cannot be used to make observations across a large region of interest, as for example would be needed to use the image to study dislocations in a sample.

Notes

Summary

16m 30s



Having looked at two effects of dynamical scattering on TEM imaging, first with thickness fringes and then with bend contours, in the next lecture, we will look at an effect on diffraction patterns with what is known as double scattering, in which dynamical scattering leads to the observation of diffraction spots for planes which are kinematically forbidden, and so should be systematically absent.

Notes

Summary

16m 50s