ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

Introduction: the instrument - I

Transmission Electron Microscopy

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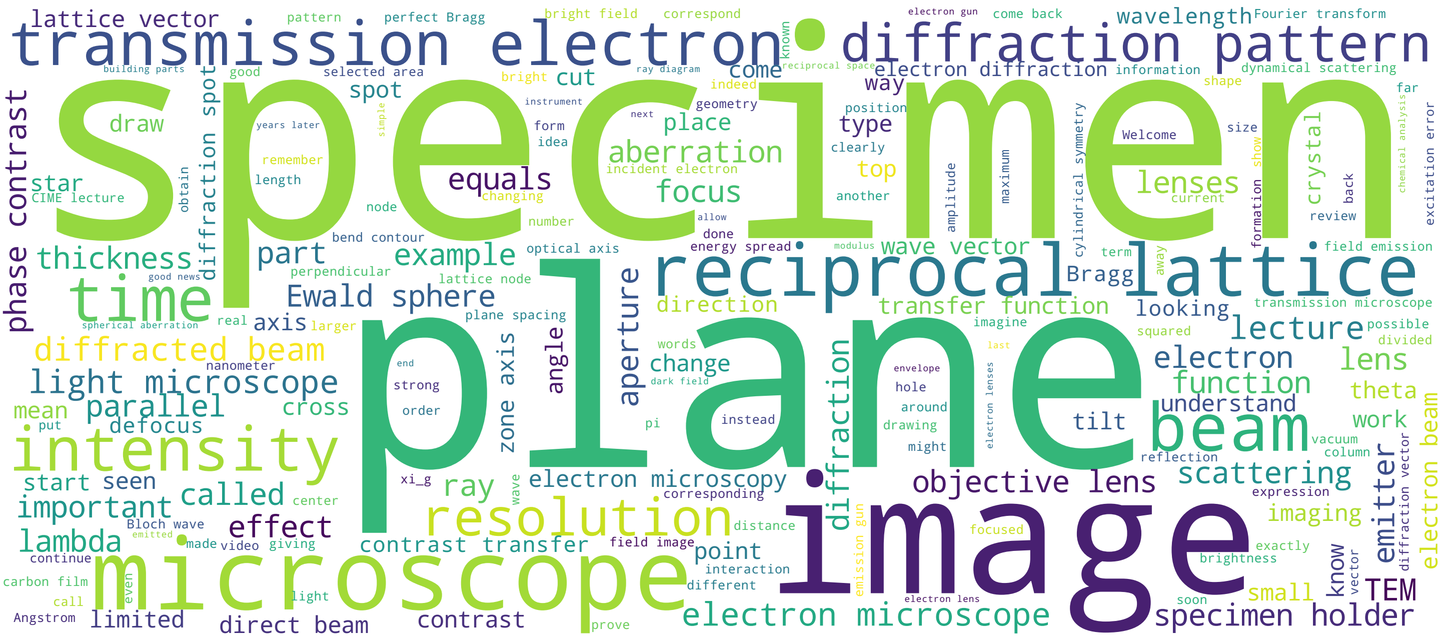

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Video

EPFL

Introduction

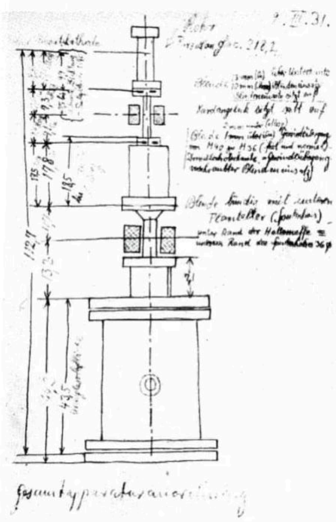
So, welcome to CIME's lecture on Transmission Electron Microscopy for material science. In this video we will have a brief historical introduction about the transmission microscope and then review all the building parts of the instrument with which we will work during this lecture.

Notes

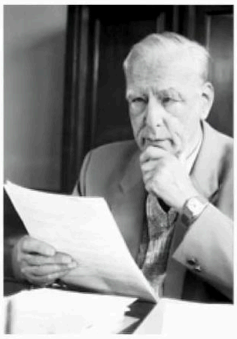
Summary

0m 05s

History of TEM



Sketch of first TEM by Ruska, 1931



E. Ruska

- 1931: Knoll & Ruska build the first prototype
- 1933: The TEM surpasses the light microscope in resolution
- 1939: first commercial instrument

- 1986 E. Ruska is awarded the Nobel prize

From Nobel Lectures, *Physics 1981-1990*, Editor-in-Charge Tore Frängsmyr, Editor Gösta Ekspong, World Scientific Publishing Co., Singapore, 1993

Transmission Electron Microscopy

The Transmission Electron Microscope was first built by Knoll and Ruska in 1931, when they built the first prototype. Actually, when they started working on this device, the idea was to prove the theoretical demonstration that thin electromagnetic coil would have exactly the same effect on electrons than a thin glass lens has on light. So, if you want to prove this, the idea was to say: "OK, let's prove that this lens, electromagnetic lens, can focus electrons and make an image". And very soon they realized that it is possible to get images with better resolution than the light microscope. Only two years later the TEM is surpassing the light microscope in terms of resolution. And then only 6 years later there are the first commercial instruments which is quite fast if you look at the time and history in Germany at that time. And then time passes and it is only in 1986 that Ernst Ruska is awarded the Nobel Prize. About the Nobel lecture I really recommend that you have a read at the Nobel lector by Ernst Ruska because it is both very entertaining and very instructive on how this instrument was imagined, developed, built and all the interaction between engineering techniques and science at that time. I said that in 1933 the TEM surpasses the light microscope in resolution. Why is it so?

Notes

Summary

0m 24s

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- Abbe diffraction limit for the light microscope

$$d = \frac{\lambda}{2n\sin\theta}$$

- Equivalent for the electron microscope

$$d = \frac{1.2\lambda}{\sin\theta}$$

$\lambda = 2{,}5 \cdot 10^{-12}\ m$ @ 200kV

$\theta = 10\ mrad \quad 10^{-2}\ rad$

$d \simeq \frac{1{,}2 \cdot 2{,}5 \cdot 10^{-12}}{10^{-2}} \sim 3 \cdot 10^{-10} \quad 3\,\mathring{A}$

Transmission Electron Microscopy

How can we explain this? For the light microscope you all know the Abbe diffraction limit based on the Rayleigh criterion. It gives you the smallest detail that you can resolve in a microscope as a function of the wavelength, lambda, and the numerical aperture, n sinus theta. A similar formula holds for the electron microscope where lambda is the wavelength of the electrons and theta the maximum angle at which electrons are collected. Let's try to plug in some numbers in there. Lambda, for a typical transmission electron microscope at 200 kilovolt, is about 2.5 picometer. times 10 to the -12 meter at 200 kilovolt. Theta is the angle at which electrons are collected, and it is typically around 10 milliradians, say 10 to 20. So, 10 to the -2 radians. You will come back later to this during the lecture. OK, putting this into the formula, we get d approximatively 1.2 x 2.5 10 to the -12 over 10 to the -2 which is around 3 10 to the -10. It is 3 Angstroms. We are around the atomic distances in material. Atomic resolution is possible and that was already recognized as early as in the 30's.

Notes

Summary

2m 15s

- Abbe diffraction limit for the light microscope

$$d = \frac{\lambda}{2n\sin\theta}$$

- Equivalent for the electron microscope

$$d = \frac{1.2\lambda}{\sin\theta}$$

$$\lambda = 2.5 \cdot 10^{-12}\,\text{m @ 200 kV};$$

$$\theta \approx 10 - 20\ \text{mrad}$$

$$\Rightarrow\ d\ 1.5\ \text{to}\ 3\ \text{Å}$$

Transmission Electron Microscopy

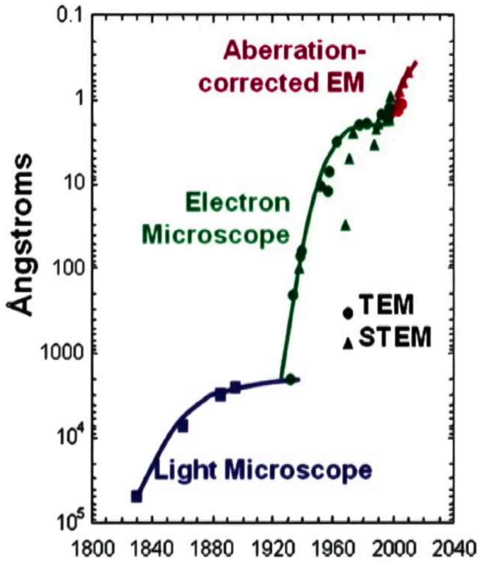
Let's imagine now if we improve things a little bit with lambda, or with theta, then we can get maybe less than 3 Angstroms. If we have 10 to 20 millirads then 20 millirads gives us 1.5 Angstrom, that is really around the interatomic distance in crystals. It is far above the resolution of a light microscope.

Notes

Summary

3m 56s

History of TEM



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S. J. Pennycook & al., in: The Oxford Handbook of Nanoscience and Nanotechnology, ed. A. V. Narlikar and Y. Y. Fu, Oxford University Press, Oxford, United Kingdom, 2010, p. 205.

Transmission Electron Microscopy

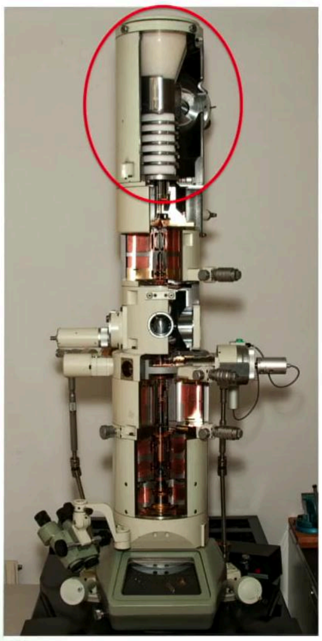
And indeed if we watch the evolution of the resolution of microscope, you see that the resolution of light microscopes was improving at the beginning and then is hitting some maximum, just limited by the Abbe diffraction limit and the wavelength of light and you reach something around 100 to 200 nanometers and as soon as it started being commercialized the electron microscope surpasses the resolution of light microscope. And the resolution increases as progress is made to be able to work with larger theta or smaller lambda. But then, it reaches also a maximum which is just below 1 Angstrom. Why is it so? Can't we improve things by having a very large theta? Well, actually not because we have problems with aberrations. So, for these microscopes the resolution is limited by the aberrations and not as for light microscope by the wavelength. Later on, aberration corrected microscopes were developed and with an aberration corrected transmission electron microscope you can reach even better resolutions. So, I think that is sufficient to be convinced that this is a fantastic instrument that will open an incredible amount of possibilities. But before learning how to work with it and how it works we need to look at its difference building blocks.

Notes

Summary

4m 26s

The constituent of the TEM



A typical TEM: Jeol 200cx

Electron gun :

Transmission Electron Microscopy

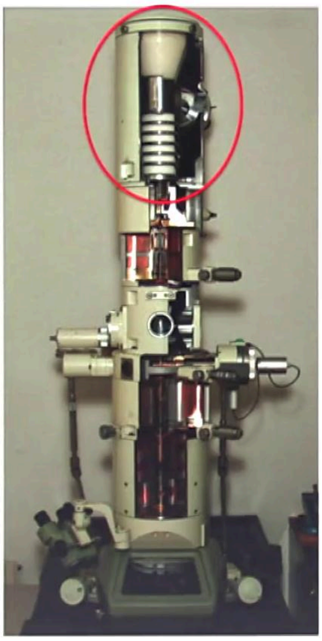
So, we need to look at all of the constituents of the transmission microscope. Let's start with this microscope: it is a Jeol 200cx, an old generation microscope that has been cut open, so you can see all the internal parts because normally they have to be under vacuum. The electrons are generated on the top and come down the electron column, they hit the specimen at the height of the specimen holder and they are focused by all these electromagnetic lenses. So, let's start from the top. We have the electron gun at the top The electron gun will be the part that generates and accelerates the electrons.

Notes

Summary

6m 03s

The constituent of the TEM



A typical TEM: Jeol 200cx

Electron gun :

generates and accelerates the electrons to the desired energy (velocity)

Emitter: emits the electrons

Accelerator stage: accelerate them

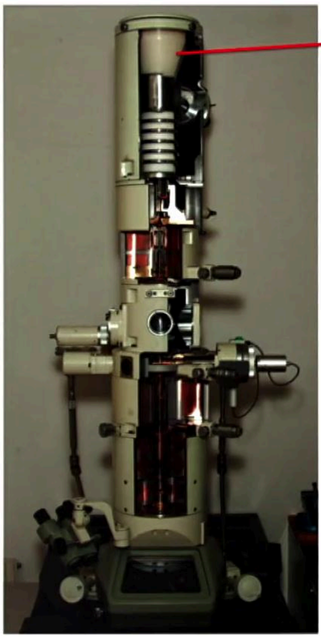
The first part, where the electrons are generated is hidden behind this plastic part and then all these ceramics are separating the different parts of the accelerator. The emitter emits the electrons and the accelerator stage accelerates them to the desired energy.

Notes

Summary

6m 47s

The constituent of the TEM



A typical TEM: Jeol 200cx

The emitter:

thermionic / Shottky FEG

	Unit	W	LaB6	FEG	Cold FEG
Brightness	A/m^2sr	10^{10}	5.10^{11}	5.10^{12}	10^{13}
Cross over size	nm	10^5	10^4	15	3
E-spread	eV	3	1.6	0.7	0.3
vacuum	Pa	10^{-2}	10^{-4}	10^{-6}	10^{-9}
Lifetime	h	100	1000	>5000	>5000

Transmission Electron Microscopy

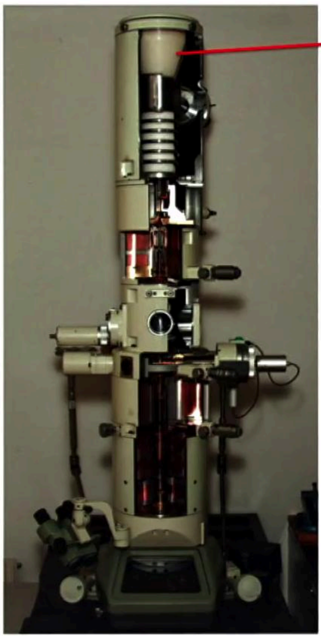
There are 4 types of emitter listed in the next table. Tungsten and lanthanum hexaborides are so-called thermionic emitters. For those 2 types, the emitter is heated and electrons are emitted because of this heating. The cold field emission gun is a real field emission emitter - the electrons are emitted thanks to a high electric field applied to the emitter. And in the middle we have the so-called shotkey FEG which is a compromise between thermionic emitter and cold FEG where the electrons are emitted by field emmissions assisted by a heating of the filament. A key quantity to characterize the emitter is the brightness. The brightness gives the intensity emitted per square metre and steradian. So, if you take an area of your emitter and you look at the current, the number of electrons by unit of time emitted for this given area and in a solid angle omega, you get the brightness. There is a huge difference in brightness between the different type of emitters. And the high brightness is linked to a high coherency because the electrons come from a small part of the emitter. This is important for phase contrast. A second characteristic is the size of the cross over after the emitter. It is much larger for tungsten, than for the cold FEG.

Notes

Summary

7m 10s

The constituent of the TEM



A typical TEM: Jeol 200cx

The emitter:

thermionic — Shottky FEG

	Unit	W	LaB6	FEG	Cold FEG
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vacuum	Pa	10^{-2}	10^{-4}	10^{-6}	10^{-9}
Lifetime	h	100	1000	>5000	>5000

Transmission Electron Microscopy

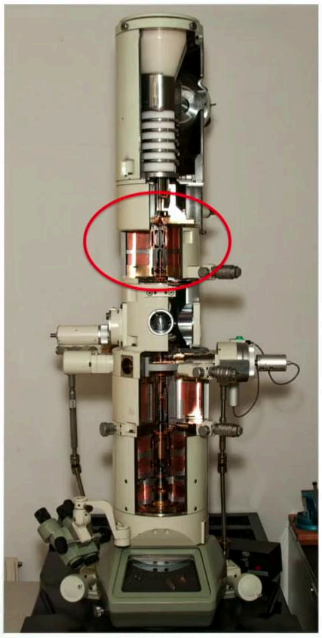
With a small size of the cross over after the gun, you will be able to focus your beam more precisely on the specimen which will be good for chemical analysis, for example, or for working in scanning transmission electron microscopy. The third characteristic is the energy spread. How well are the electrons defined in energy? Field emission guns have a much narrower energy spread. This is both important for chemical analysis, but also a smaller energy spread gives a better temporal coherency important for phase contrast as well. But those better characteristic come at a price. We need a higher vacuum for those emitters, which means that they are much more expensive in operation, but fortunately they also have a longer lifetime.

Notes

Summary

9m 09s

The constituent of the TEM



A typical TEM: Jeol 200cx

Electron lenses are electromagnetic coils:

Transmission Electron Microscopy

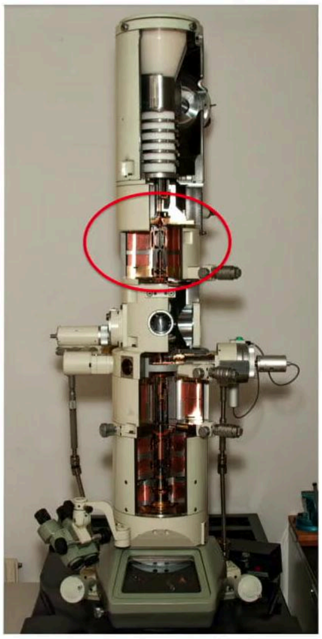
Let's continue down the column to the next part. One of the most important one: it is the electron lens. The electron lenses, you see some there, but also some down the column. They are electromagnetic coils.

Notes

Summary

10m 09s

The constituent of the TEM



A typical TEM: Jeol 200cx

Electron lenses are electromagnetic coils:

•Generate a field with cylindrical symmetry

•Fixed in place. Current influences focal length

•“bad” lenses, compared to glass lenses in light microscopy: resolution of the TEM limited by aberrations

•Only convergent lenses

•One can use ray diagrams, as in light microscopy

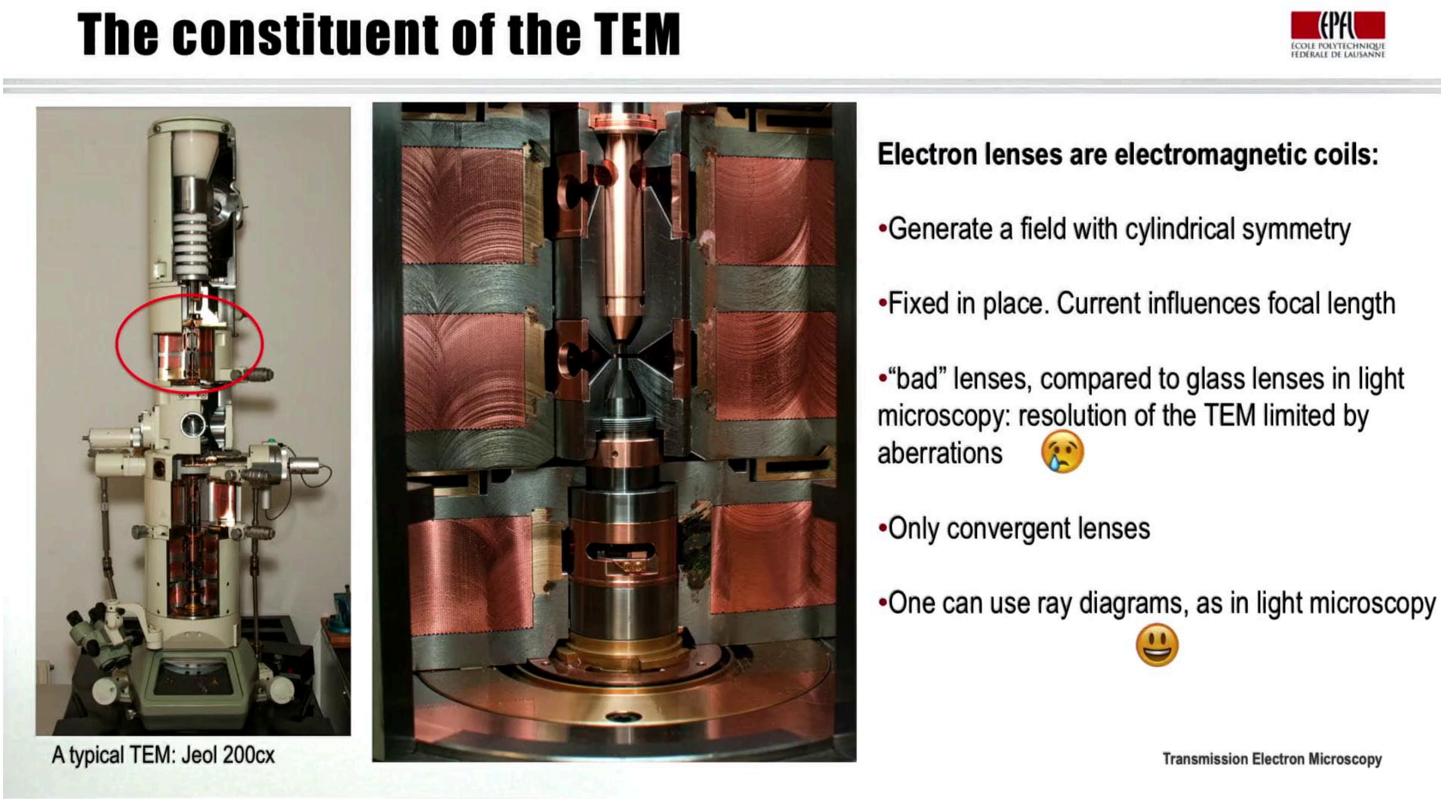
Transmission Electron Microscopy

If you look at an enlarged picture, where it is cut, you can clearly see how the wires... so it just copper wires winded around the optical axes of the microscope - and when they are cut, you see all these wires there. So, with this geometry, they will definitively generate a field with a cylindrical symmetry. Also, with this type of lenses, there is a big difference compared to light microscopy. Here we can easily change continuously the focal length of the lens by just changing the current in the coil and that is what we do all the time when we operate the microscope. For example, when we change magnification, the focus of the image, change from imaging to diffraction, etc. This is much more versatile than for a light microscope. We had a little talk about this. They are "bad" lenses. They have a lot of aberrations, and that will be the limiting factor to the resolution of microscope. So the TEM is limited by the aberration of this kind of lenses. That is why we will review the aberrations in the next lesson as they are very important to understand the imaging properties of the microscope. Also, if you stick to lenses with cylindrical symmetry, as is usually the case in the transmission electron microscope, then you only get convergent lenses.

Notes

Summary

10m 26s



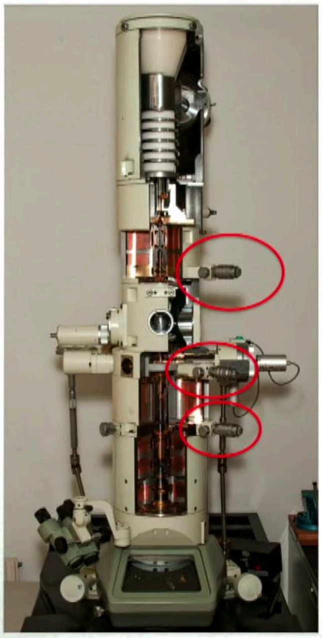
And last, because the electron lens has the same focusing property on electrons than a glass lens has on light, you can make use of ray diagrams to understand your microscope as you do in light microscopy. So, basically, I have two news there. One bad news: the electron microscope is limited by the resolution of those lenses. One good news: you will be able to use ray diagrams and you will make use of them all the time during this lecture. They are very important to understand the working property of the transmission electron microscope. OK. it is a good news provided you remember how to use them.

Notes

Summary

11m 56s

The constituent of the TEM



A typical TEM: Jeol 200cx

Apertures

Transmission Electron Microscopy

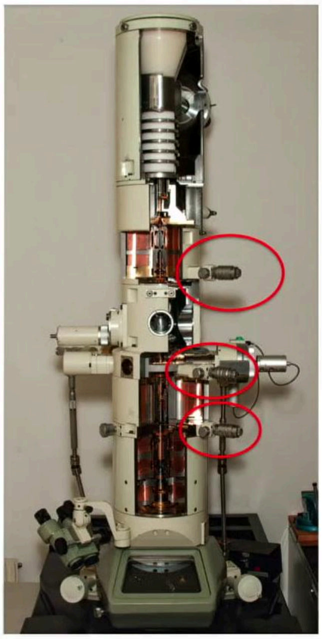
So, do you remember how to draw the image produced by the thin convergent lens? If not, it will be explained in the next video. But let's continue on the building parts of the microscope now. The next one, that we will look at is the apertures. The apertures are holes of defined size placed on the path of the beam. They are supported by a thin plate mounted on a rod.

Notes

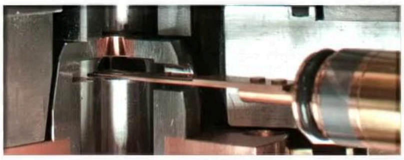
Summary

12m 39s

The constituent of the TEM



A typical TEM: Jeol 200cx



Apertures

- Small (10-200 micrometers) holes limiting the extension of the beam
- Generally 4 to 8 sizes to chose from.
- At “strategic” places in the microscope, they will define some properties of the beam, or select some part of it. Crucial in contrast formation, imaging etc.

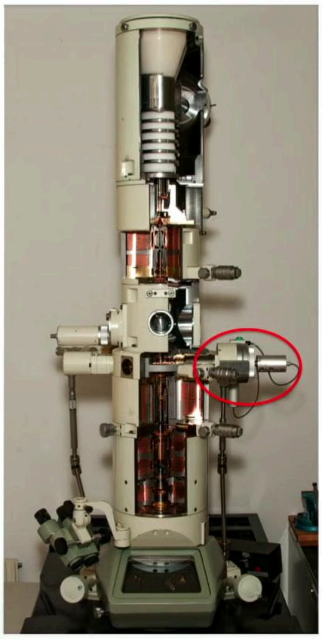
Transmission Electron Microscopy

You can see here some apertures in the lower part of the image also. These holes are small 10 to 200 micrometers and they will limit the extension of the beam. Generally, you have something like four to eight sizes you can chose from, like here, for example you recognize maybe three or four sizes, and they are at strategic places in the microscope. They will define some properties of the beam, like the one which is just after the beam-forming coils that we will review next time or the ones which are at strategic places in the imaging parts of the microscope.

Notes

Summary

13m 08s



A typical TEM: Jeol 200cx

Goniometer and specimen

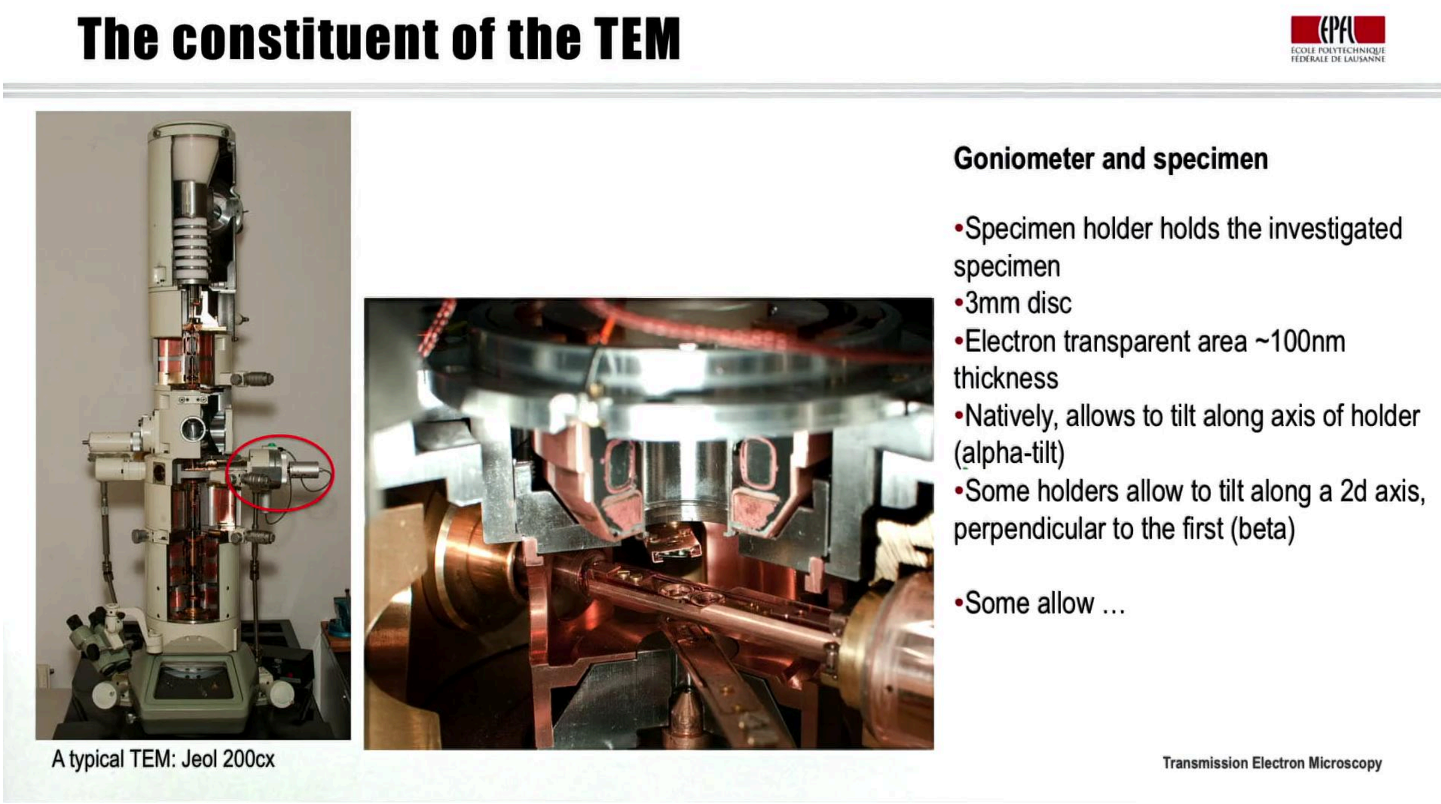
Transmission Electron Microscopy

And, finally, we need to look at something, right? We want a specimen, we want the specimen hold at some place. It is held in the goniometer and in a thing called the specimen holder.

Notes

Summary

13m 50s



The specimen we look at in TEM is small and you will see that it is the specimen holder that enters the beam, the optical axis is down there and at that point you have the place for the specimen. So, the specimen holder will hold the investigated specimen. Very commonly it is a 3 millimetre disc and you need an area that will be transparent to your electrons. The order of magnitude should be around 100 nanometres or less. If it is thinner, it is much better for some imaging. Natively, you will be able to tilt your specimen along the axis of the specimen holder. That is what we call the alpha-tilt. Some holders will also allow you to tilt the specimen along a second axis which is perpendicular to it. And then some holders will allow incredible things. For example, you will be able to heat your specimen or cool your specimen to liquid nitrogen temperature or even to liquid helium temperature. You might be able to have electrical fields or electrical currents. You might be able to have some straining experiments or some indentation experiments. There is no limit to the imagination. That will not be too much part of the lecture but if you look into what is done in transmission electron microscopy, there is a huge variety of in situ experiments allowed by these specimen holders.

Notes

Summary

14m 06s

Specimen holders and specimen

And, finally, to come back to the real world, well I have brought you a specimen holder. It is there in its protective cover and if I remove it, you see this very thin long rod.

Notes

Summary

15m 44s

The constituent of the TEM

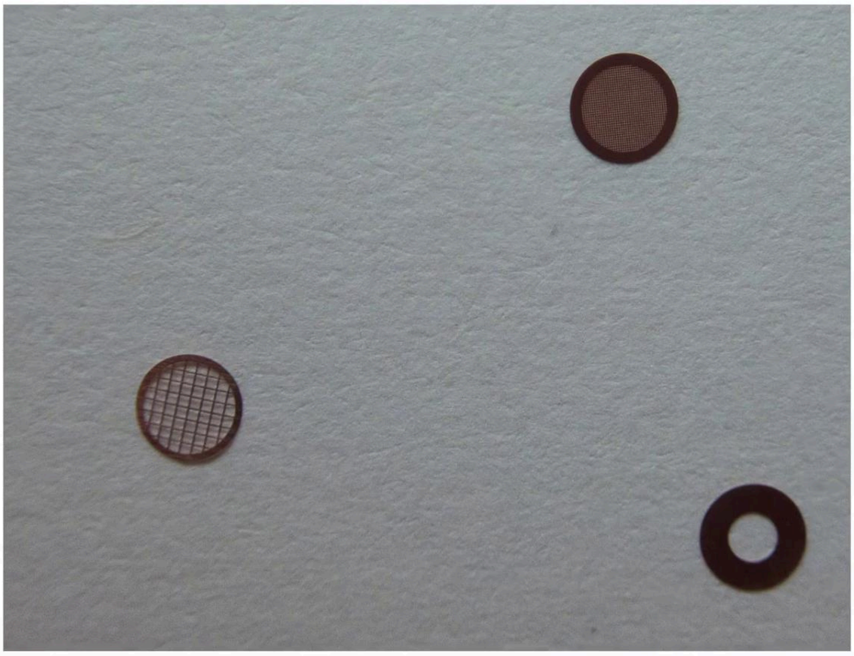
Specimen holders and specimen

That is an O ring that will allow to keep the vacuum in the microscope and there you have the place where to put the specimen. So, it is a small, thin disc and some specimen can be, for example, nanoparticles or whatever that are deposited on a copper grid covered with a thin carbon film.

Notes

Summary

16m 02s

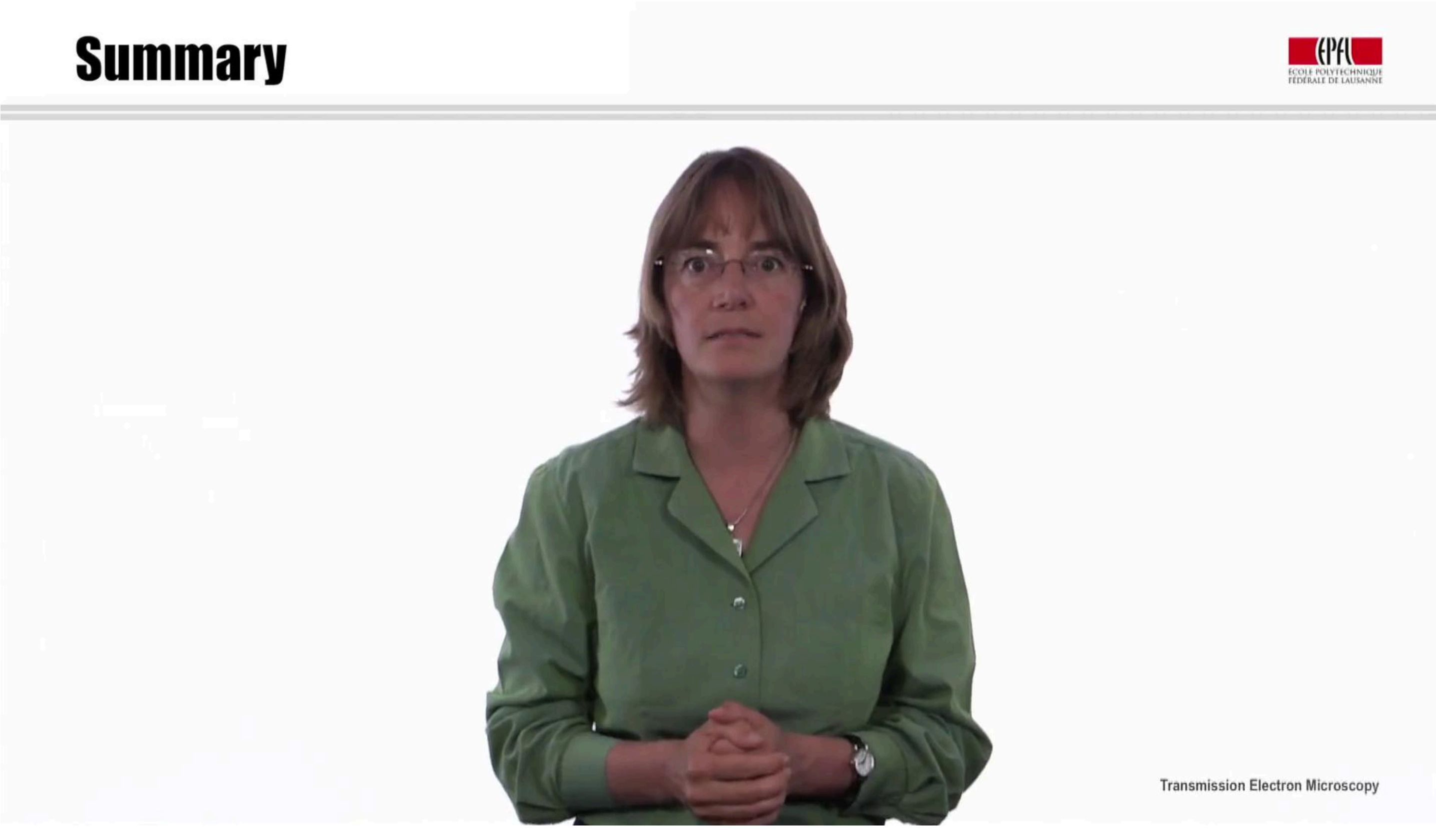


How does the copper grid looks like? Well, it is this very thin, tiny 3 millimetre disc that you have here. So, manipulating them you need a lot of care.

Notes

Summary

16m 25s



So, in this module you have seen the main constituents of the transmission microscope. One of them is the lens. In the next module we will review all the aberrations that are relevant to electron lenses.

Notes

Summary

16m 39s