## Transmission Electron Microscope (I)

### An extremely brief history

(Transmission) Electron Microscope term was first used in the paper of Knoll and Ruska in 1932. In this paper they developed the idea of electron lens into a practical reality, and demonstrated electron images taken by the instrument they built. This was the most crucial step, for which Ruska received the Nobel prize, some time late, in 1986. Surprisingly, Ruska revealed that he had not heard of de Broglie's ideas about electron waves and thought that the wavelength limit did not apply to electrons. TEMs were developed by commercial companies only four years later (in 1936) in England. Other sources to provide TEMs after that include Simens and Halske, Hitachi, JEOL, Philips and RCA, etc.



A photograph of an actual microscope (JEOL JEM 100B)



**Figure** Different TEMs: (A) a JEOL 1.25-MV high voltage microscope, used for high-resolution imaging; (B) a Hitachi specialized ultrahigh vacuum TEM for high-resolution surface imaging; (c) a Philips 200-KV analytical microscope with an X-ray spectrometer attached to the stage (the liquid-N<sub>2</sub> dewar cools the detector); and (D) a VG dedicated 100-KV ultrahigh vacuum scanning transmission microscope.

#### Limitations of TEM

1. Sampling

Very small sample size. But fortunately we are dealing with nanostructures, as long as we have enough in a small piece, we are fine.

2. Interpreting transmission images:

TEM is presenting 2D images of 3D specimens, viewed in transmission.

3. Electron Beam damage and Safety

With many TEM with the accelerating voltage of 400kV, the beam can destroy any sample if not careful. Safety issue: remember: TEM is a potential dangerous instrument that generates radiation level that is enough to kill not only a fly, but also a human being. So if you are not sure what you are doing, forget it and ask somebody who knows.

4. Specimen preparation:

Your specimens have to be thin, very thin (has to be electron transparent) if you are going to get any information.



Figure Photograph of two thinos taken to that, in projection, they appear as one two-beaded beast. Such projection artifacts in reflectedlight images are easily discernible to the human eye but similar artifacts in TEM images are easily mistaken for "real" features.



Figure Beam damage in quartz after bombaniment with 125-keV electrons. With increasing time, from (A) to (B), the damaged regions increase in size.

#### The instrumentation

We have known that almost all of components in the TEM are the same as that we used and discussed for SEM. Now, we want to see how the guns, lenses, and detectors are combined to form the microscope. We will do the same thing as that we did for SEM, i.e., divide it into three component systems: the illumination system, the objective lens/stage, and the imaging system.

The illumination system comprises the gun and the condenser lens and its role is to take the electrons from the source and transfer them to your specimen. You can operate the illumination system in two principal modes: parallel beam and convergent beam. The first mode is used for TEM imaging and diffraction, while the second is used for scanning (STEM) imaging microanalysis and microdiffraction.

The objective lens/stage system is the heart of the TEM. The critical region usually extended over less than 1 cm along the length of the column. Here is where all the beam-specimen interactions take place and here we create the bright-field, dark-field images, and

selected-area diffraction patterns (SAD) that are the fundamental TEM operations. Likewise, it is here that we manipulate a scanning beam to form STEM images and diffraction patterns.

The imaging system uses several lenses to magnify the image or the diffraction pattern produced by the objective lens and to focus these on the viewing screen. We will refer to the magnifying lenses as intermediate and diffraction lenses and the final lens as the projector lens (it projects an image on the viewing screen). Alternatively, an electron detector coupled to a TV/CRT can be used to display the STEM images.

In many modern TEMs, you will have controls for: the condenser lens current which determines the area of specimen illuminated; the objective lens current which focuses the image; the projector lens current which controls the magnification; and specimen shift controls which enables the operator to look around his specimen.



Fig. A schematic of a modern six-lens transmission electron microscope

We understand that the TEM is basically analogous to transmission optical microscope. Nevertheless it does not mean that we have already understood everything of the image formed by TEM. In fact, the principle of the TEM image formation is a little bit more fancy due to one of the facts of <u>electron diffraction</u>. So the first thing we want to investigate is the electron-specimen interaction. We tried to minimize our discussion in SEM lectures on the transmitted electrons. Now we have to look a little bit more at this process since it will help us to explain what is going on in TEM imaging and diffraction patterns.



(a) A two-dimensional array of atoms with two sets of atomic planes, of spacing  $d_1$  and  $d_2$ , indicated. (b) The scattering of an incident beam of electrons (I) by a crystalline specimen. Intense beams of electrons may emerge from the other side of the specimen undeviated (T) or having been diffracted from the atomic planes of spacing d(D). In other directions (e.g. N) no intense beams will be formed.

Bragg's law:

The diffraction of electrons only occurs when the atoms of the specimen are arranged in some regular ways and the elastically scattered electrons within a specimen then travel predominantly in certain directions instead of being randomly scattered in all directions. Look at the previous figure: The incident beam is coherent, in other words, all individual electrons are in phase. Any scattered electron waves, which are also in phase with one another will reinforce and lead to a strong beam of electrons, whereas any scattered waves, which are out of phase will not reinforce. Look at the scattered waves in the D direction: if

 $CB+BE=n\lambda$ , n is an integer, then these waves are in phase. Because  $CB=BE=dsin\theta$ , therefore  $2dsin\theta=n\lambda$  is the condition for reinforcement. In practice, beams with n=0 and n=1 are much strong than n>1 therefore

 $\lambda$ =2dsin $\theta$ ,  $\theta$  is small ( $\lambda$  small, d small). So  $\lambda$ =2d $\theta$ 

<u>Camera length</u>: L from specimen plane to diffraction pattern plane. r: distance from undiffracted beam (n=0) and one first order beam (n=1) at an angle  $2\theta$ . So:



The effect of projector lenses on the geometry of diffraction patterns. The action of the two lenses means that the actual distance between the specimen and the diffraction pattern, X, is not the same as the effective ' camera length ' L.

Note: 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 diffraction screen, but is a notional distance, which may be adjusted by the microscopist.





Fig.

The types of diffraction pattern which arise from different specimen microstructures. (a) A single perfect crystal; (b) a small number of grains—notice that even with only three grains the spots begin to appear to form circles; (c) a large number of randomly oriented grains—the spots have now merged into rings; (d) an amorphous specimen merely gives rise to a diffuse halo, indicating that on average the atoms are similar distances apart.



Single crystal aluminum

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Polycrystalline copper



For the same crystal, the diffraction pattern could be different

Two of the commonest crystal structures of metals and the diffraction patterns which can arise from them. (a) A body-centred cubic (b.c.c.) arrangement of atoms with the electron beam incident on it parallel to the cube edges. (b) A hexagonal close-packed (h.c.p.) arrangement of atoms with the electron beam incident parallel to the prism edges.



Fig.

The same two arrangements of atoms with the electron beam incident along a different direction in each. The diffraction patterns are very different from those formed in the diffraction Note that pattern (b) shows no sign of the hexagonal nature of the crystal structure which gave rise to it.





# Selected Area Diffraction (SAD) patterns



It is a basic principle of TEM operation that when you want to look at the diffraction pattern (i.e, the back focal plane of the objective lens), you put an SAD aperture into the image plane of the objective lens.

In fact, to see the diffraction pattern you have to adjust the imaging system lenses so that the back focal plane of the objective lens acts as the object plane for the intermediate lens. Then the diffraction pattern is projected onto the viewing screen.

If you want to look at image instead, you readjust the intermediate lens so that its object plane is the image plane of the objective lens. Then an image is projected onto the viewing screen.



Figure The two basic operations of the TEM imaging system involve (A) projecting the diffraction pattern on the viewing screen and (B) projecting the image onto the screen. In each case the intermediate lens selects either the back focal plane or the image plane of the objective lens as its object.