8. Structure and basic principles of transmission electron microscope

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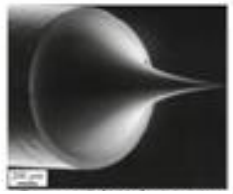
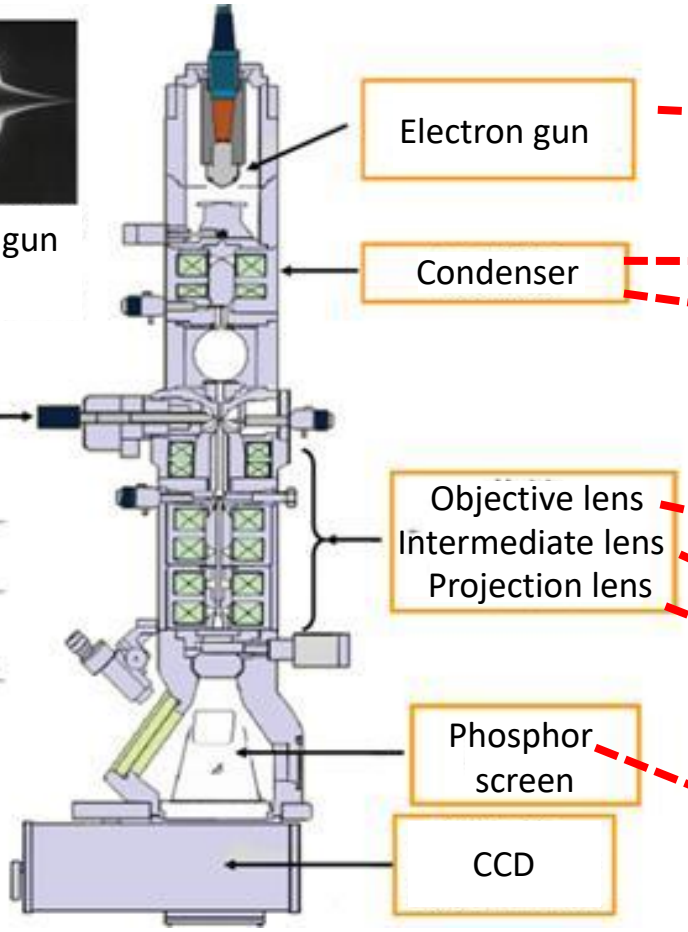
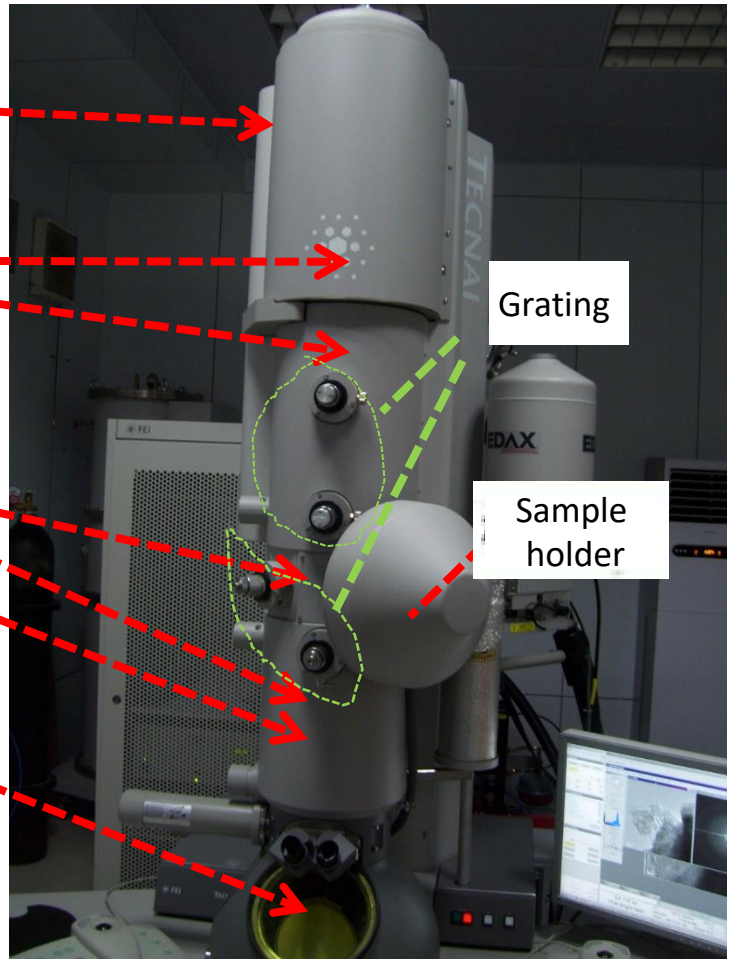


Electron gun

Sample holder



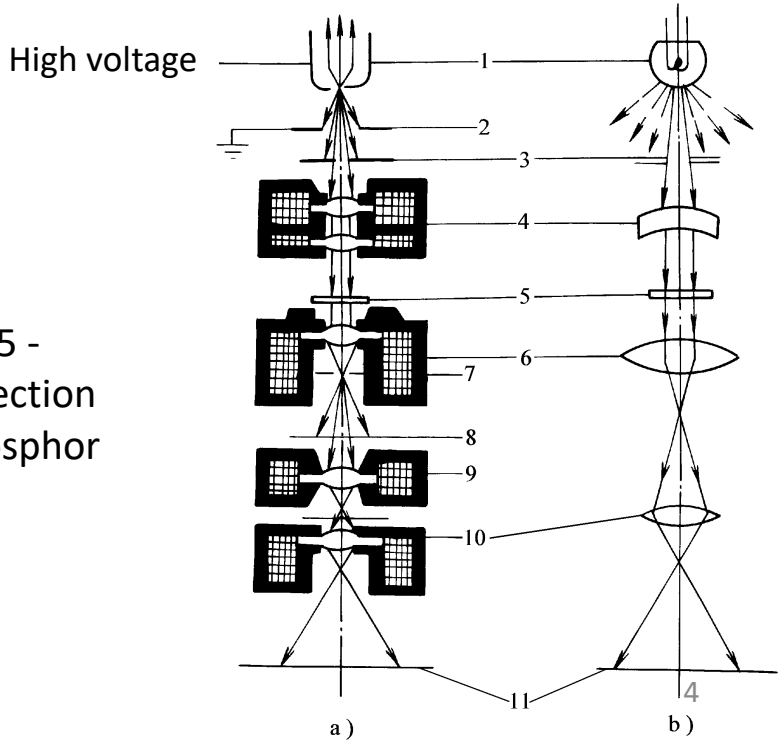


8.1 Structure and imaging principle of transmission electron microscope

The basic components of a transmission electron microscope: electron optical system, power supply and control system, and vacuum system.

The electron optical system is usually called the lens barrel, and its optical path principle is similar to that of a transmission optical microscope.

The components of the electronic optical system: lighting system, imaging system, observation and recording system.

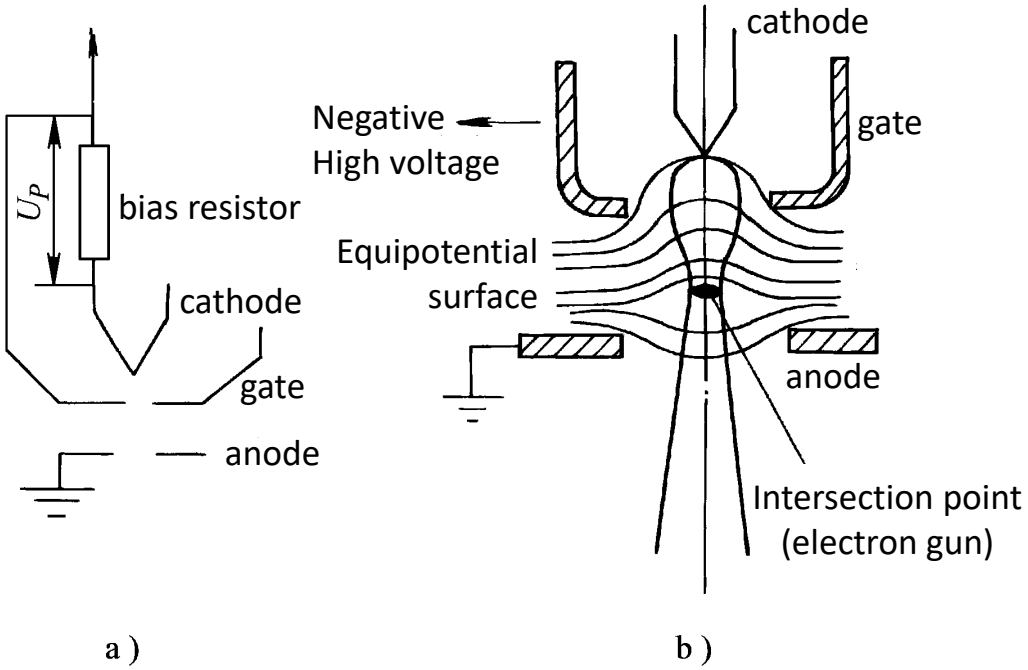


1 - illumination source 2 - anode 3-aperture 4 -condenser 5 - sample 6 - objective lens 7 - objective lens aperture 8 - selection aperture 9 - intermediate lens 10 - projection lens 11 -phosphor screen or film

8.1 Structure and imaging principle of transmission electron microscope

- Lighting system

1. Electron gun



There are two types of electron guns: thermal emission and field emission. The thermal emission electron gun consists of a cathode, a grid and an anode, as shown in the figure.

The negative high voltage of the electron gun is applied to the gate, and there is a potential difference of hundreds of volts between the cathode and the grid to form a self-bias circuit.

The gate can control the effective area of the cathode to emit electrons. The function of the self-bias circuit is to stabilize and adjust the beam field. The launch gun has excellent performance, with small beam spot size, high brightness, and small energy dispersion.

8.1 Structure and imaging principle of transmission electron microscope

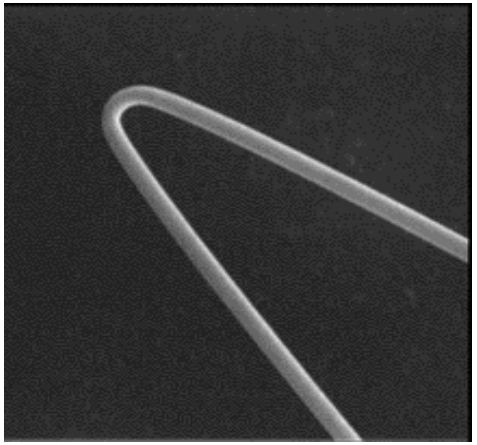
- Lighting system

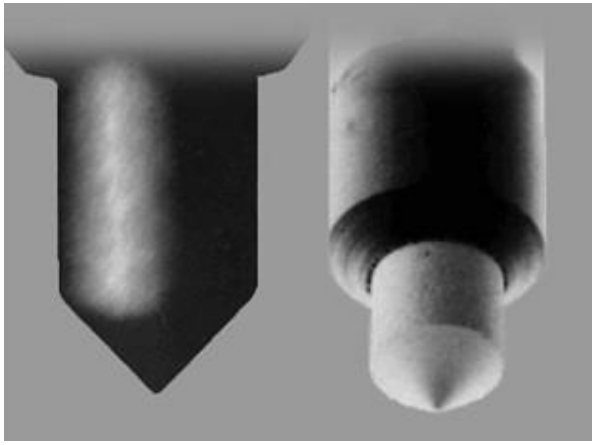
1. Electron gun

In an electron microscope, a heating filament as a cathode is called a direct-heated cathode. The filament is mainly made of metal tungsten wire. It is characterized by low cost, low brightness and short life. The diameter of the filament is about 0.10 ~ 0.12 mm, and its heating current value is continuously adjustable.

In modern electron microscopes, the new material lanthanum hexaboride (LaB_6) is sometimes used to make filaments. It is more expensive, but has high luminous efficiency and brightness (can be improved by an order of magnitude), and its durability is much longer than that of tungsten filaments.

Tungsten filament



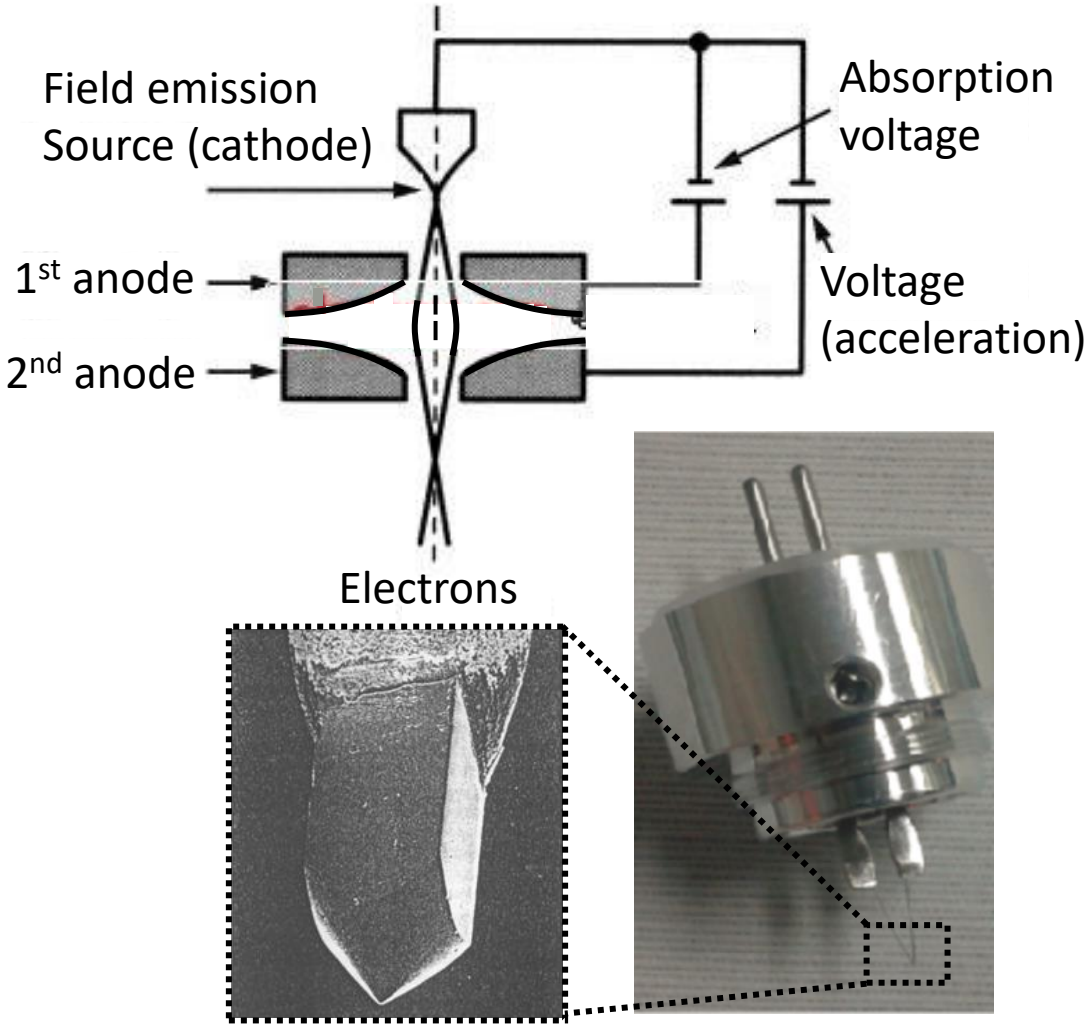


Lanthanum hexaboride

8.1 Structure and imaging principle of transmission electron microscope

- Lighting system

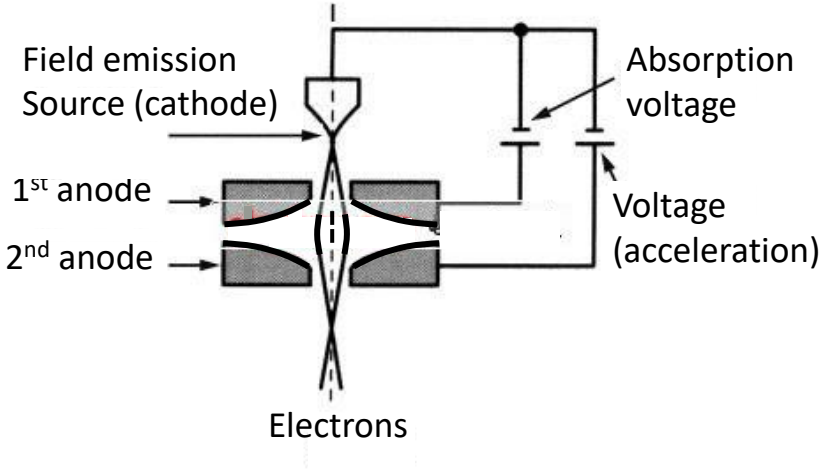
1. Electron gun



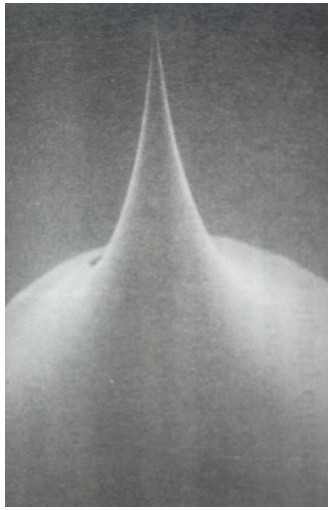
It consists of a cathode and two anodes. A slightly lower (relative to the second anode) adsorption voltage is applied to the first anode to attract the free electrons on the cathode, while the extremely high voltage on the second anode. The free electrons are accelerated to a very high speed and emit an electron beam. This requires ultra-high voltage and ultra-high vacuum as working conditions.

8.1 Structure and imaging principle of transmission electron microscope

- Lighting system



There is also a new type of electron gun called a field emission electron gun. Field emission refers to the phenomenon of electrons being released from the cathode surface under the action of a strong electric field.

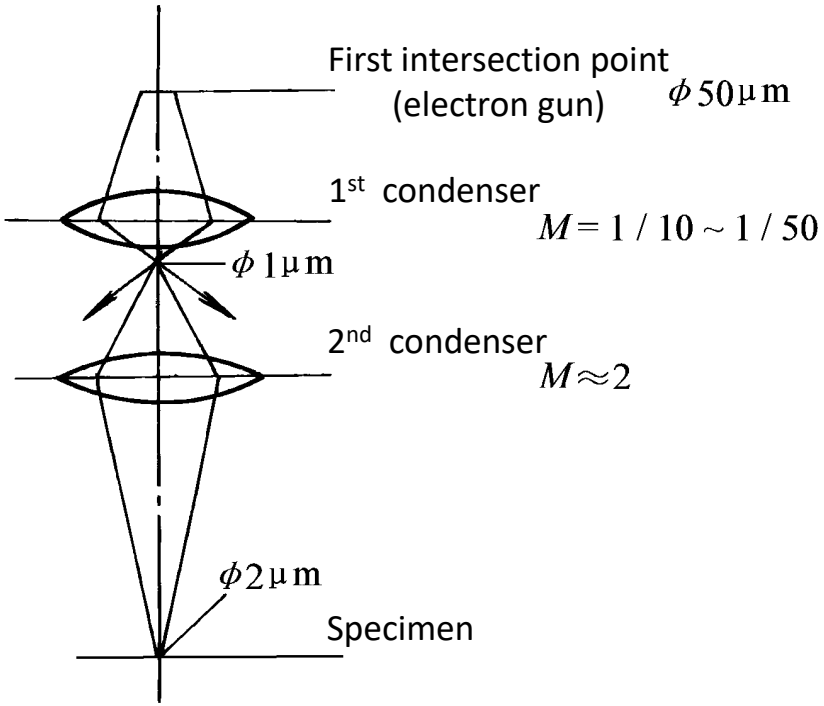


The field emission electron gun consists of a cathode and two anodes. A slightly lower adsorption voltage (relative to the second anode) is applied to the first anode. The first anode is called the extraction electrode, which is several thousand volts larger than the cathode. The free electrons are attracted out, and the extremely high voltage on the second anode can reach 100 kV and above, which is used to accelerate the free electrons to a very high speed and emit an electron beam.

8.1 Structure and imaging principle of transmission electron microscope

- Lighting system

2. Condensers



High-performance transmission electron microscopy uses a dual condenser system. The first condenser is a strong excitation lens, which reduces or adjusts the beam spot size and reduces the electron gun cross spot by 10 to 50 times.

The second condenser is a weak excitation lens, which adjusts the illumination intensity. The function of the condenser is to reduce and adjust the beam spot size, adjust the illumination intensity and the half angle of the illumination aperture with minimal loss.

8.1 Structure and imaging principle of transmission electron microscope

- Lighting system

3. Objective lens

The objective lens is the lens used to form the first image, so the resolution of the transmission electron microscope mainly depends on the objective lens, which is the core component. The objective lens is a lens with strong excitation and short focal length (f =1 ~ 3 mm). The resolution of a high-quality objective lens is about 0.1 nm, and the magnification is generally 100 ~ 300 times.

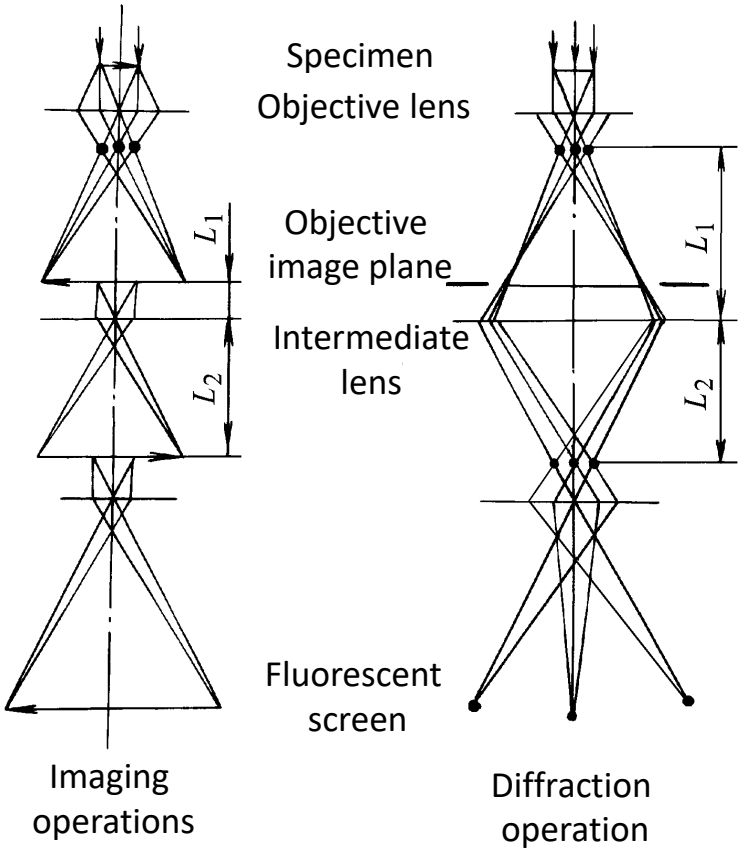
The incident electron beam passes through the sample and is focused by the objective lens to form a diffraction pattern on the back focal plane of the objective lens, forming a microscopic image on the image plane.

The resolution of the objective lens mainly depends on the shape and processing accuracy of the pole piece. The smaller the distance between the inner hole of the pole piece and the upper and lower pole pieces, the higher the resolution of the objective lens.

8.1 Structure and imaging principle of transmission electron microscope

- Lighting system

3. Intermediate lens



The intermediate lens is a variable magnification lens with weak excitation and long focal length. One of its functions is to use its variable magnification to control the total magnification of the electron microscope; the other is to realize the conversion between the transmission electron microscope imaging operation and the diffraction operation.

If the object plane of the intermediate mirror coincides with the image plane of the objective lens, an image is obtained on the fluorescent screen, which is called imaging operation; if the object plane coincides with the back focal plane of the objective lens, an electron diffraction pattern is obtained on the fluorescent screen, which is called diffraction operation.

8.1 Structure and imaging principle of transmission electron microscope

- Lighting system

4. Projection lens

The projection lens is a strong excitation lens with short focal length.

The aperture angle at which imaging electrons enter the projection lens is very small (about 10^{-5}rad), so its depth of field and focal length are both very large. The large depth of field and long focal length of the projection mirror allow the positions of the object plane and image plane to move within a certain range, which is beneficial to the adjustment of the total magnification and facilitates observation and recording.

The function of the projection lens is to further magnify the image of the intermediate lens and project it onto a fluorescent screen or photographic substrate for observation or recording.

8.1 Structure and imaging principle of transmission electron microscope

- Imaging system

Figure 1 shows the image of the transmission electron microscope; Figure 2 shows the structure and vacuum system of the transmission electron microscope. High-performance transmission electron microscopes mostly use level 5 (or above) magnification for imaging.

Figure 1

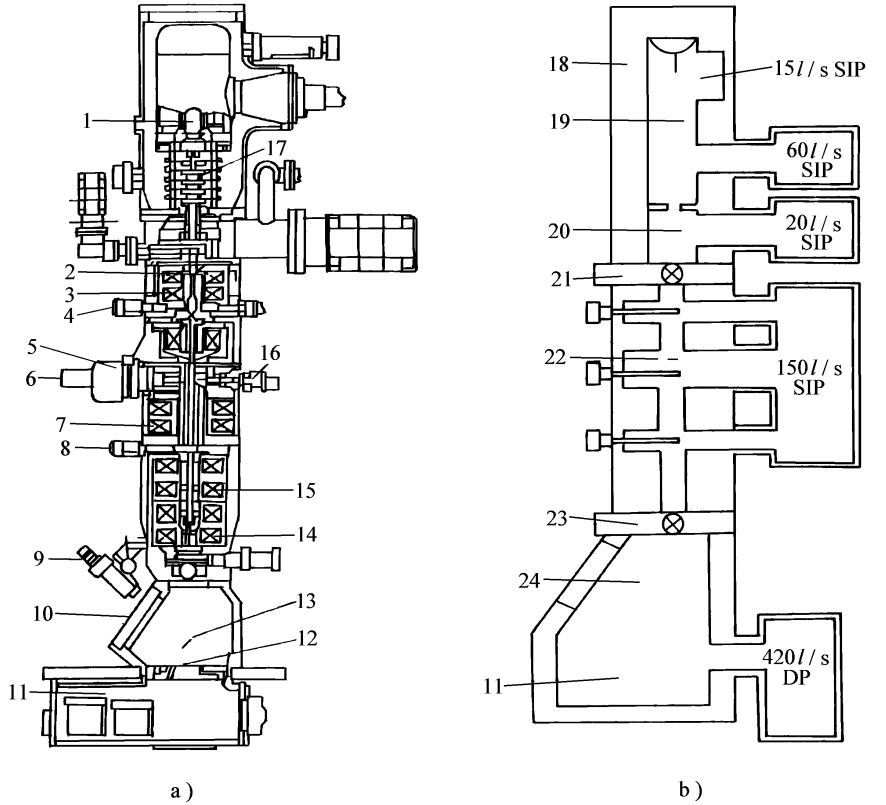


Figure 2. a) Lens barrel cross-section b) vacuum system

8.1 Structure and imaging principle of transmission electron microscope

- Observation recording system

The early transmission electron microscope observation and recording system consisted of a fluorescent screen and a camera structure. The observation was made of a fluorescent plate coated with a green-emitting fluorescent substance that is more sensitive to the human eye under dark room conditions.

It is recorded using electronic photosensitive film that is sensitive to electron beam exposure and has a very small grain size. The film exposure time adopts three methods: automatic, manual setting, or timing.

Most recent transmission electron microscopes are equipped with CCD imaging systems, which can input images to a computer monitor for observation; images can be stored and output in a variety of file formats, making image observation and recording very convenient.

8.2 Structure and working principle of main components

- Sample stage

Figure 1 shows the support film and copper mesh used to support the powder sample. For crystal samples, the sample stage should have translation in three directions and tilt around at least one axis in order to select the observation point and adjust the crystal orientation.

Transmission electron microscopes are usually equipped with very precise side-insert sample translation and tilting devices, called double-tilt stages (see Figure 2). The translation value along the mutually perpendicular OX and OY directions is ± 1mm; it can tilt about ± 40° around the OX axis and OY axis.

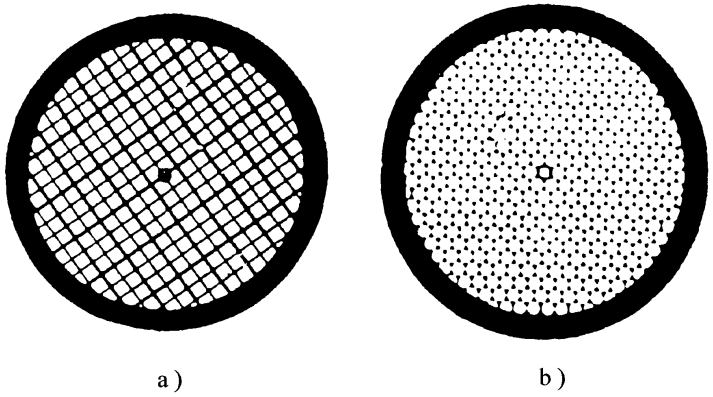


Figure 1

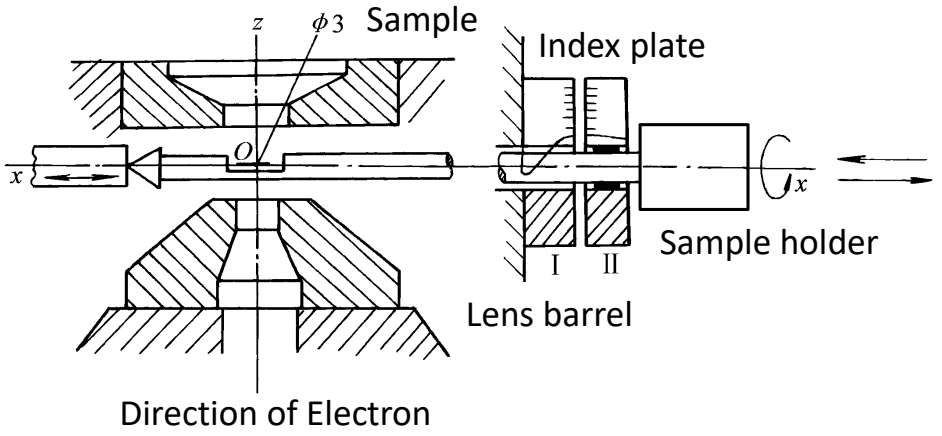
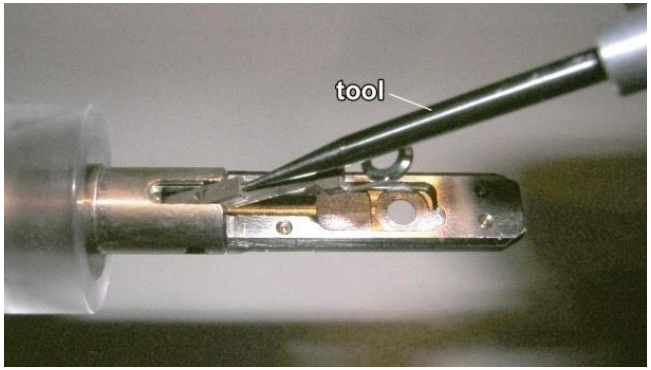
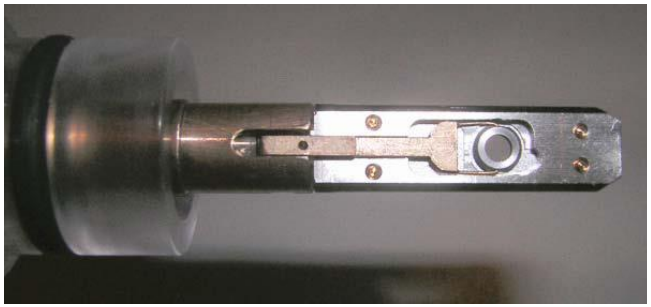


Figure 2

8.2 Structure and working principle of main components

- Sample stage



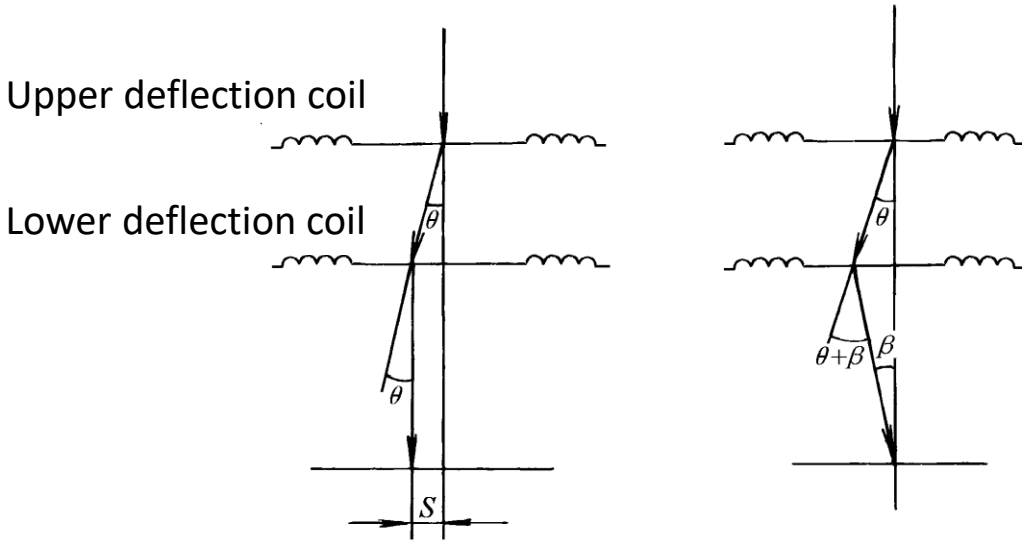


8.2 Structure and working principle of main components

- Electron beam translation and tilt device

As shown in the figure, the electron beam can be translated and tilted using an electromagnetic deflector. The principle is to use the upper and lower deflection coils to linkage.

- If the upper and lower deflection coils deflect the electron beam at equal angles but in opposite directions, the electron beam can be translated;
- If the upper coil deflects the electron beam by an angle θ and the lower coil deflects the electron beam in the opposite direction by an angle ($\theta + \beta$), then the electron beam is tilted by an angle β relative to the original direction.

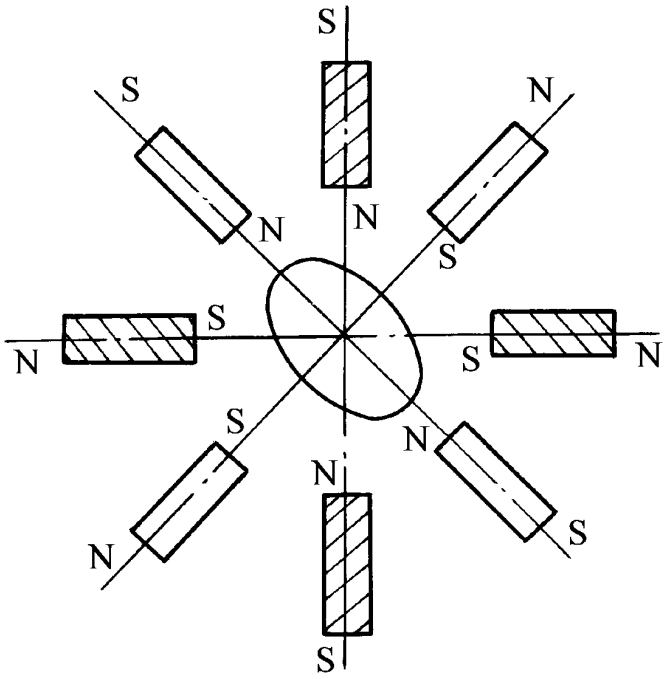


a) Translation b) Title

8.2 Structure and working principle of main components

- Astigmatism absorbers

Astigmatism absorbers can be divided into two types: mechanical and electromagnetic. New transmission electron microscopes are often equipped with electromagnetic astigmatism absorbers, as shown in the figure. It consists of two groups of four pairs of electromagnets arranged around the lens magnetic field, and each pair of electromagnets is placed with the same pole facing each other.



If the magnetic field of the lens appears non-rotationally symmetrical, the elliptical magnetic field is corrected by changing the excitation intensity and direction of the two sets of electromagnets, thereby eliminating astigmatism. The astigmatism eliminater is generally installed between the upper and lower pole pieces of the lens.

8.2 Structure and working principle of main components

- Aperture

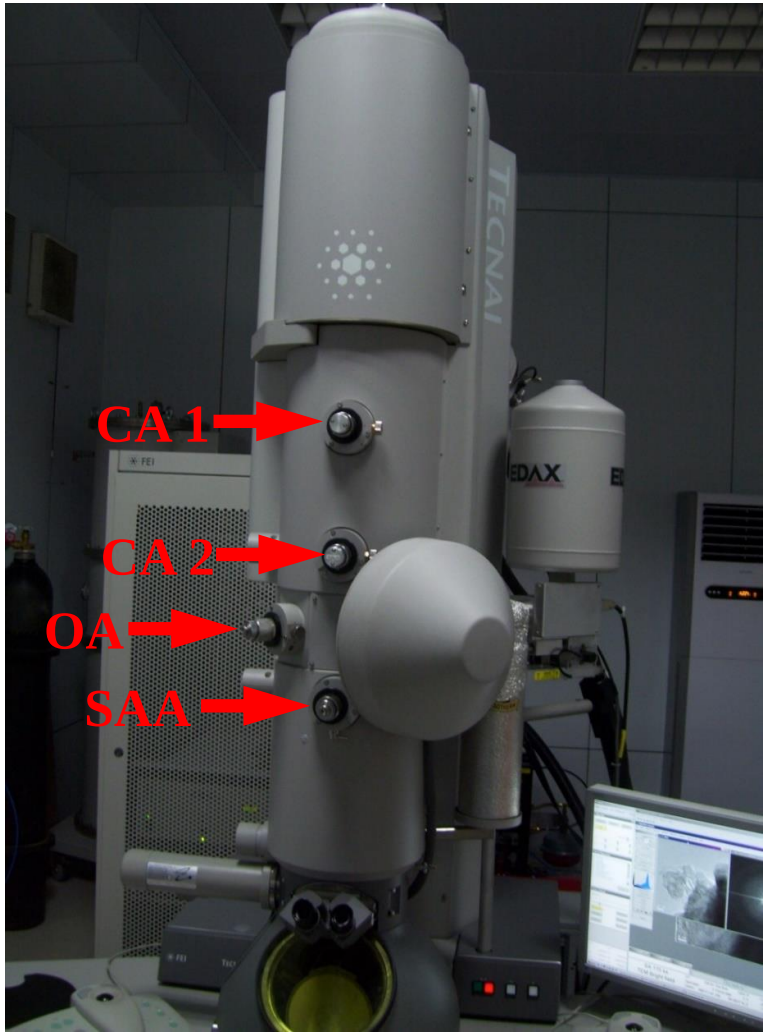
1. Condenser aperture: used to limit and change the half angle of the illumination aperture and the illumination intensity; a dual condenser system is installed below the second condenser, and the aperture is 20 ~ 400 **μm.**

2. Objective aperture: It is used to reduce the spherical aberration of the objective lens and select the imaging electron beam to obtain a bright field or dark field image. In addition, it can improve the image contrast, so it is also called the contrast aperture. The objective aperture is installed on the back focal plane of the objective lens, with an aperture of 20 ~ 120 **μm**.

3. Selection aperture: also called field aperture. During diffraction analysis, it limits and selects the sample analysis area to achieve selected area electron diffraction. The selective aperture is placed on the image plane of the objective lens, and the aperture is 100 ~ 400 **μm**. If the objective lens is magnified 100 times, the corresponding sample area is 1 ~ 4 **μm**.

8.2 Structure and working principle of main components

- Aperture

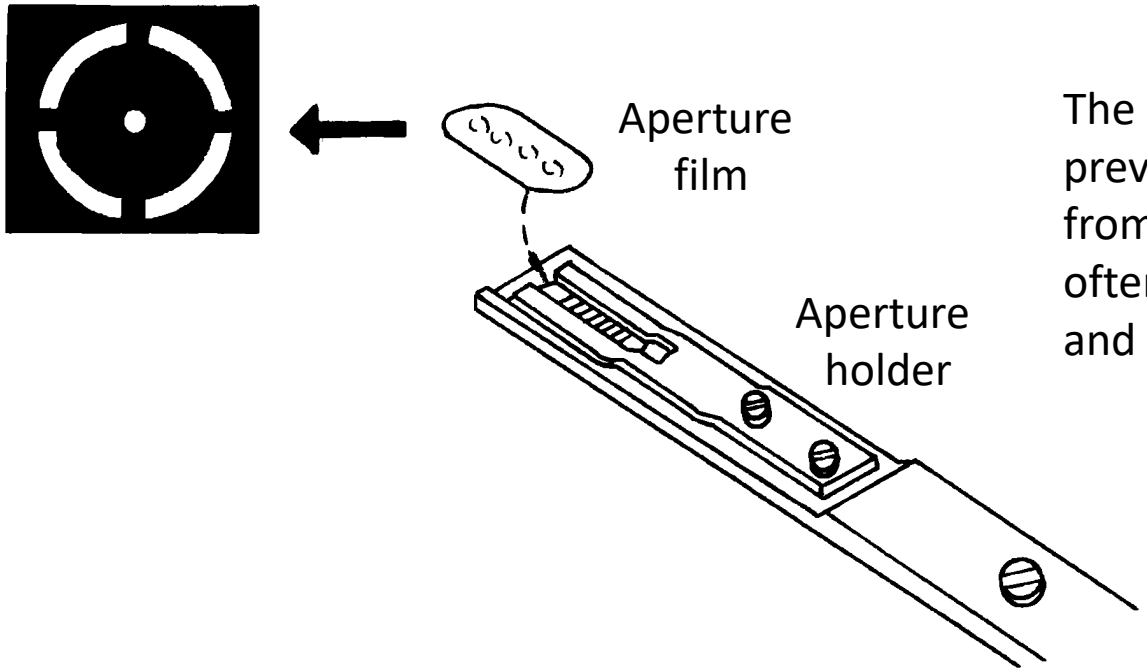


CA 1:Condenser aperture 1
CA 2:Condenser aperture 2
OA: Objective aperture
SAA: Selection aperture

8.2 Structure and working principle of main components

- Aperture

Apertures are usually made of non-magnetic metals (platinum, molybdenum, etc.). Since the aperture of the diaphragm is small and easy to be contaminated, high-performance electron microscopes often use anti-pollution diaphragms (also called self-cleaning diaphragms). The structure is shown in the figure.



The gaps around the aperture hole prevent the heat of the aperture from dissipating easily, and it is often in a high temperature state and is not easily contaminated.

8.2 Structure and working principle of main components

- Spherical aberration corrector

$$\Delta r_s = \frac{1}{4} C_s \alpha^3 \qquad \Delta r_0 = \frac{0.61\lambda}{N \sin \alpha}$$

As mentioned earlier, the electron beam wavelength and spherical aberration are one of the main factors limiting the resolution of the electromagnetic lens. Increasing the acceleration voltage and reducing the spherical aberration coefficient ***Cs*** are the main approaches to improve the electromagnetic resolution. Increasing the accelerating voltage can reduce the wavelength of electron waves, thereby improving electromagnetic resolution.

However, excessively high accelerating voltage limits the types of samples to be analyzed and at the same time, severely damages the structure of the sample. In addition, such equipment is expensive and has high maintenance costs, which has many disadvantages. Therefore, improving the resolution of electromagnetic lenses by reducing the spherical aberration coefficient ***Cs*** has become the current research direction in the development of high-resolution transmission electron microscopes.

8.2 Structure and working principle of main components

- The role of the spherical aberration corrector

Spherical aberration is spherical aberration, which is one of the lens aberrations. The lens system, whether it is an optical lens or an electromagnetic lens, cannot be absolutely perfect.

For convex lenses, the convergence ability of the edge of the lens is stronger than that of the center of the lens, causing all light rays to fail to converge to a focus, thus affecting imaging capabilities.

In optical lenses, the combination of convex lenses and concave lenses can effectively reduce spherical aberration. However, electromagnetic lenses only have "convex lenses" and no "concave lenses". Therefore, spherical aberration has become the most important and most difficult factor to correct for the resolution of electron microscopes.

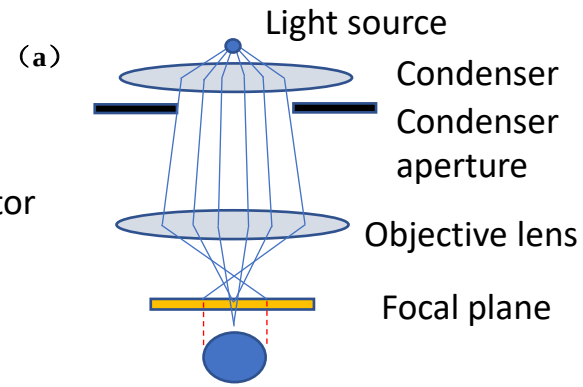
8.2 Structure and working principle of main components

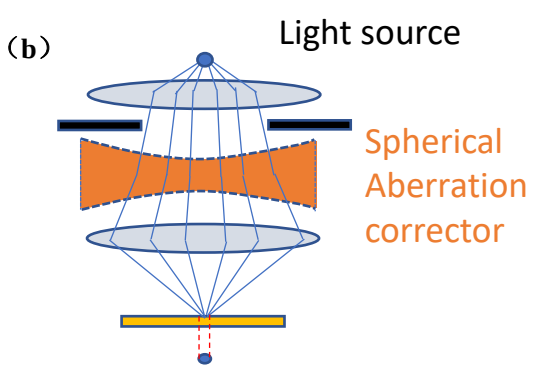
- The role of the spherical aberration corrector

Figure (**a)** shows a schematic optical path diagram without a spherical aberration corrector. The light source emits an electron beam. After passing through the condenser, condenser aperture, and objective lens, due to the inevitable spherical aberration in the objective lens of the transmission electron microscope, the points in the sample are During the imaging process, it spreads into a disk.

The function of the spherical aberration corrector can be compared to a concave lens, which diverges the electron beam after passing through the condenser, so that the electron beams at different angles can be re-converged to a point after passing through the objective lens, thus eliminating the influence of spherical aberration of the objective lens and improving the resolution of transmission electron microscopy (Figure (**b**)).

(a). Optical path without spherical aberration corrector





(b). Optical path when including spherical aberration corrector

8.2 Structure and working principle of main components

- Structural design of spherical aberration corrector

Since the invention of the transmission electron microscope, scientists have been working to improve its resolution.

Scherzer proved that a circularly symmetrical electromagnetic prism cannot achieve divergence of electron beams, so the realization of a spherical aberration corrector must rely on the redesign of the electromagnetic prism.

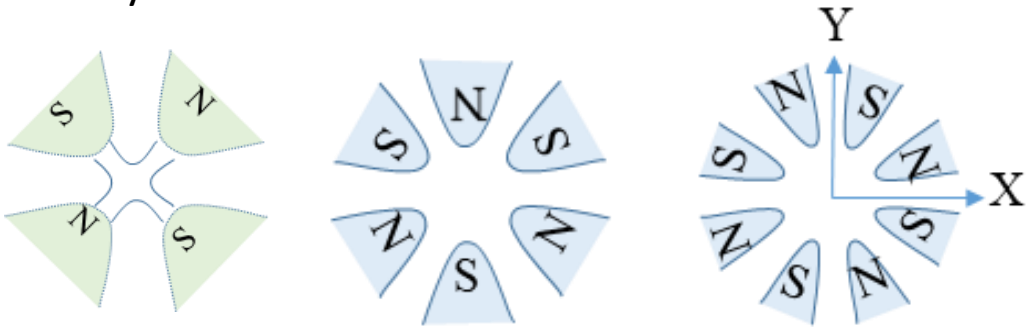
In 1992, three German scientists, Harald Rose, Knut Urban, and Maximilian Haider, developed the use of a multipole correction device to adjust and control the focusing center of an electromagnetic lens to correct spherical aberration, ultimately achieving sub-angstrom resolution.

8.2 Structure and working principle of main components

- Structural design of spherical aberration corrector

The multipole correction device gradually adjusts the spherical aberration of the transmission electron microscope through multiple sets of magnetic mirror groups with adjustable magnetic fields that act on the Lorentz force of the electron beam (such as quadrupole, hexapole or octupole magnetic field, as shown in Figure), thereby achieving sub-angstrom resolution.

In 1990, Rose theoretically proved the feasibility of a dual six-pole spherical aberration corrector. In 1998, it successfully developed the world's first TEM spherical aberration corrector. This prototype spherical aberration corrector is installed on Philips CM200 and increases its point resolution from 0.24 nm to 0.13 nm, officially bringing the transmission electron microscope industry into a new era of atomic-level resolution.



quadrupole hexapole octupole

8.2 Structure and working principle of main components

- Types of spherical aberration correctors

A transmission electron microscope contains multiple magnetic lenses: condenser lens, objective lens, intermediate lens, projection lens, etc. Spherical aberration is caused by the imperfect structure of the magnetic mirror, so these magnetic mirror groups will produce spherical aberration. When correcting different magnetic lenses there are different types of spherical aberration corrected transmission electron microscopes.

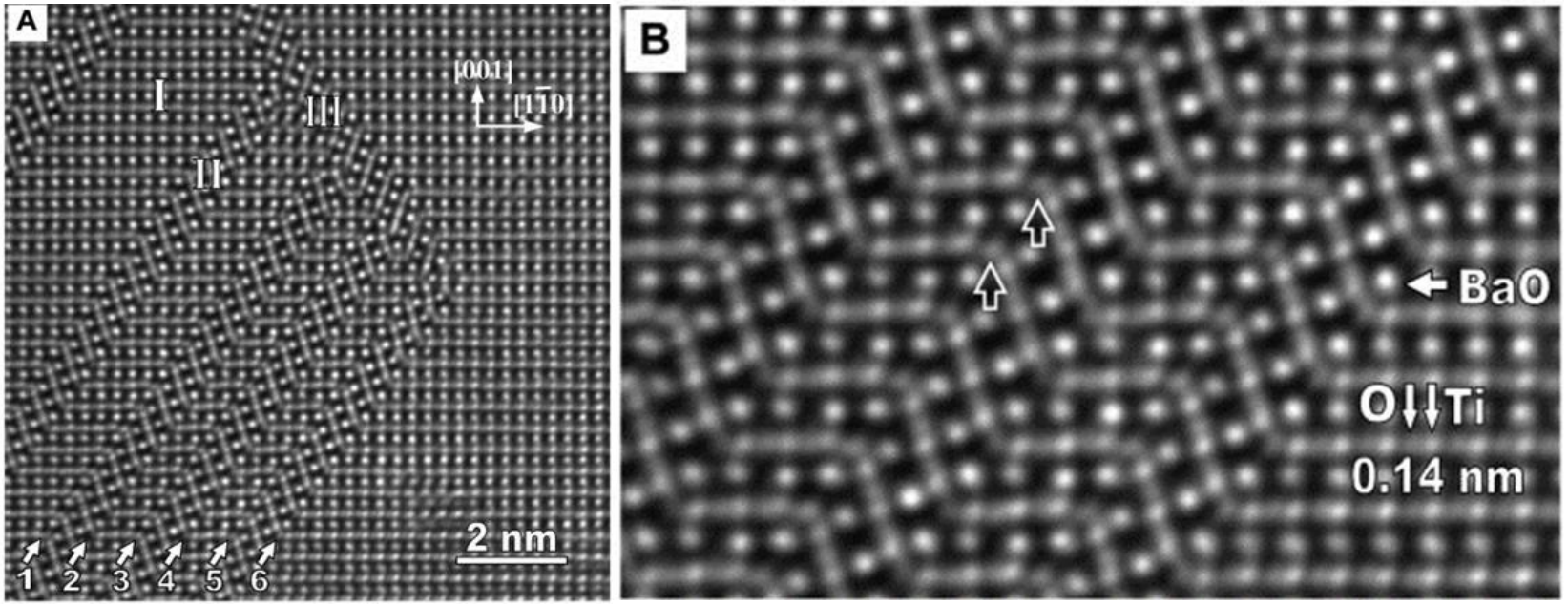
When using the scanning transmission mode (STEM), the condenser condenses the electron beam to scan the sample for imaging. At this time, the condenser spherical aberration is the main reason that affects the resolution. Therefore, for transmission electron microscopes mainly used for STEM, the spherical aberration correction device will be installed at the condenser position, which is a spherical aberration correction STEM electron microscope.

When using the ordinary image mode, the main factor that affects the imaging resolution is the spherical aberration of the objective lens. This kind of corrector installed at the position of the objective lens is a spherical aberration-corrected TEM electron microscope.

Of course, two correctors are also installed on one transmission electron microscope, this is so-called double spherical aberration correction TEM. In addition, since the corrector has a voltage limit, different models of spherical aberration correction transmission electron microscopes have their corresponding accelerating voltages.

8.2 Structure and working principle of main components

- Applications of spherical aberration transmission electron microscopy



(A) I represents the matrix, II and III represent two twins, with O atoms on the twin boundaries, as indicated by the numbers 1-6 in the figure;

(B) An enlarged photo of the twin region in the above figure. The black arrow indicates the oxygen atoms located on the twin boundary. The minimum distance between Ti and O atoms is 0.14 nm in the lower right corner of the figure.

8.2 Structure and working principle of main components

- Digital imaging system

The transmission electron microscope digital imaging device is an indispensable key component of modern transmission electron microscopes. This device plays a role in image imaging, such as the charge couple device (CCD) camera, which is a typical example. Today's new CCD products mainly include bottom-mounted and side-mounted.

The CCD camera has a powerful self-scanning function, good image clarity, and can capture images at any time. It supports multiple merged pixel modes. The innovative readout technology can entirely reduce noise and achieve a higher sensitivity and conversion effect, making the image extremely high-quality. Signal-to-noise ratio. Compared with traditional cameras, CCD cameras have the advantages of small size, high reliability, high sensitivity, resistance to intense light, resistance to vibration, resistance to magnetic fields, small distortion, long life, clear images, and easy operation.

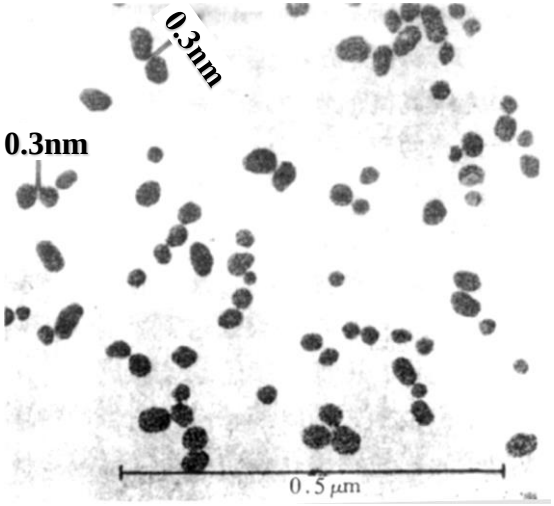
The CCD camera has a stable, independent cooling system that is isolated from the vacuum system of the transmission electron microscope. In addition, the CCD camera has powerful video image recorder software functions and a working language interface.

8.3 Determination of resolution and magnification of transmission electron microscope

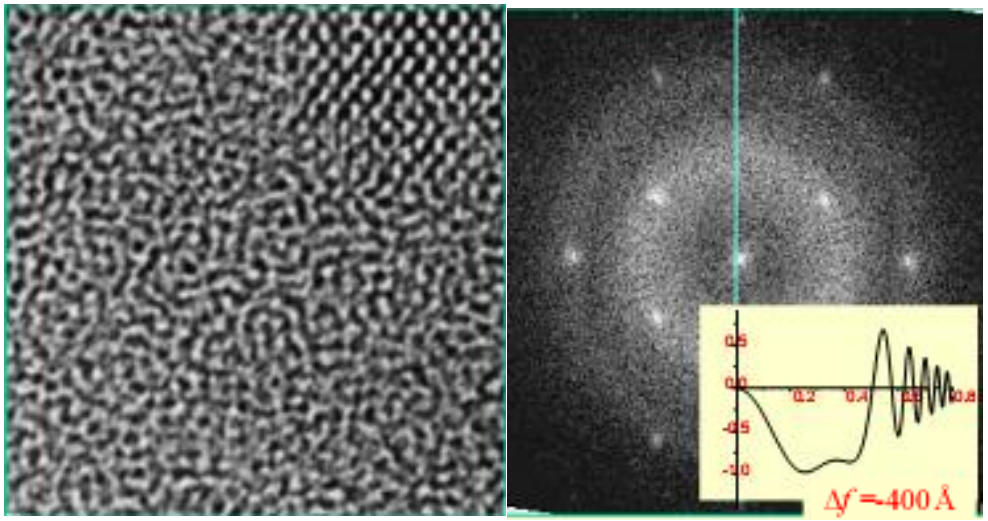
- Resolution

1. Point resolution: An early method of measuring point resolution is to use vacuum evaporation of platinum, gold and other particles on a carbon support film. Find the minimum distance between particles in a high-magnification photo and divide it by the magnification to get the point resolution, as shown in the figure.

At present, the high-resolution image of the amorphous carbon film is used to perform Fourier transform to obtain the diffraction pattern. The reciprocal of the radius of the first dark ring is the point resolution, as shown in the figure.



Determination of point resolution

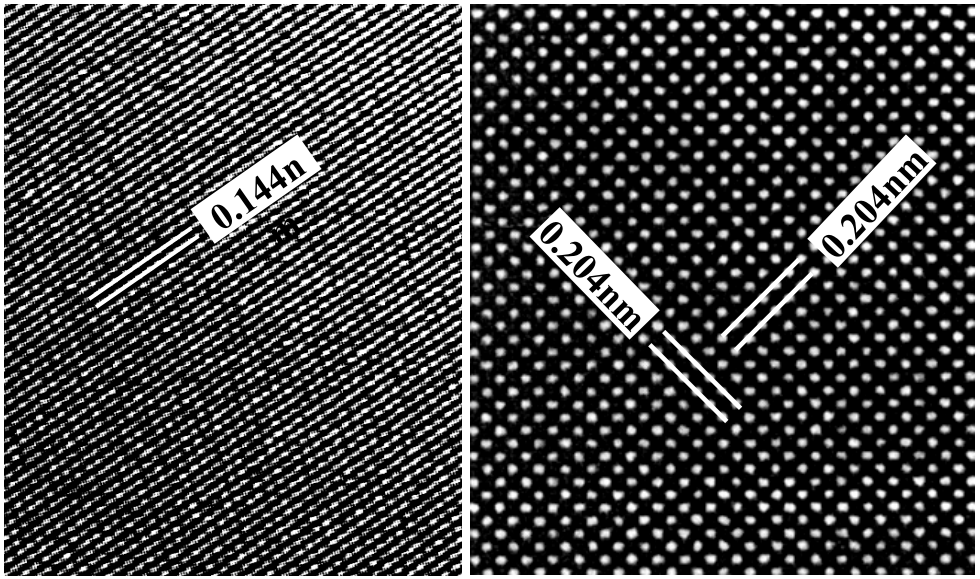


Determination of point resolution

8.3 Determination of resolution and magnification of transmission electron microscope

- Resolution

2. Line resolution: Line resolution is also called lattice resolution. The method of measuring line resolution is to use a single crystal film with known orientation as a standard sample, take a picture of the lattice, and determine the line resolution of the instrument based on the lattice stripes with known spacing. The data of commonly used crystals for measuring line resolution are shown in Table as shown in Figure.



Lattice image measurement line resolution of gold (220) and (200) planes.

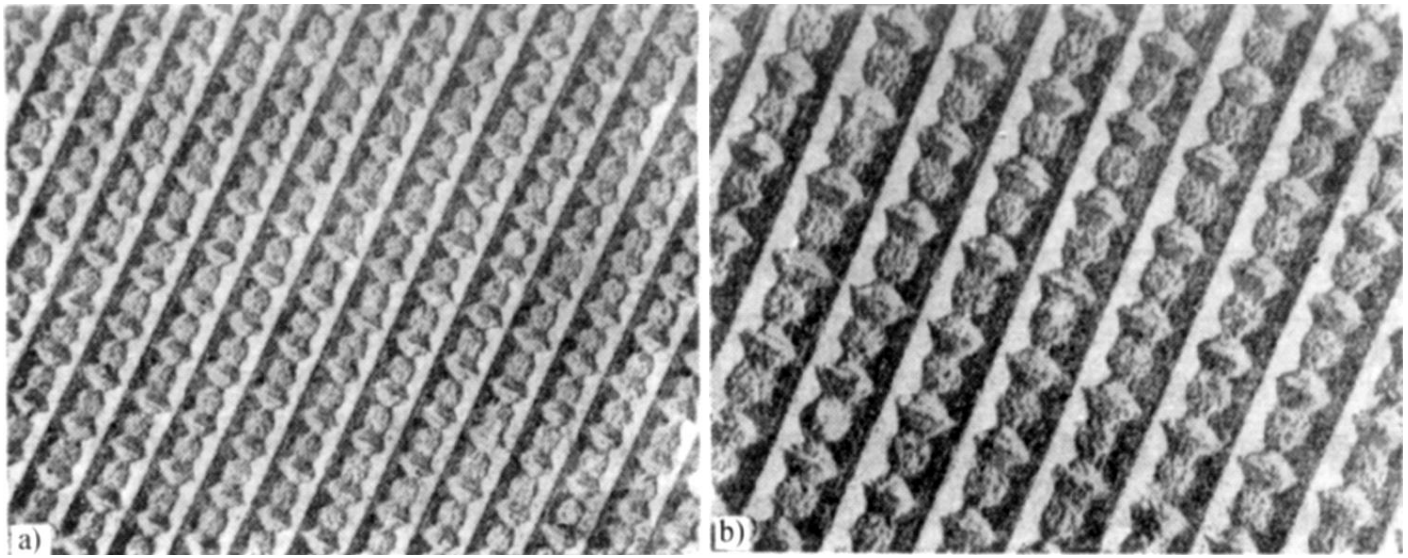
Crystal	*H K L*	*d* nm
Gold	(200)	0.204
	(220)	0.144
Palladium	(111)	0.224
	(200)	0.194
	(400)	0.097

Commonly used crystals for measuring resolution.

8.3 Determination of resolution and magnification of transmission electron microscope

- Magnification

Magnification changes with sample height, acceleration voltage, and lens current. In order to ensure the accuracy of the magnification, regular calibration is required, and the normal allowable error is ± 5%. A replica of a diffraction grating is often used as a standard sample. The average spacing of the grating stripes is measured on the film. Dividing the actual spacing is the magnification factor under this condition. High magnification can also be measured using lattice images.



1152 lines/mm grating strips replica pattern **a**) 5700 times **b**) 8750 times