Electron Microscope

An electron microscope is a microscope that uses a beam of accelerated electrons as a source of illumination. As the wavelength of an electron can be up to 100,000 times shorter than that of visible light photons, electron microscopes have a higher resolving power than light microscopes and can reveal the structure of smaller objects. Few electron microscopes are:

1. **Sanning electron microscope** (**SEM**)
2. **Transmiission electron microscope** (**TEM**)
3. **Scanning Probe Microscopy (SPM)**
4. **Scanning Tunnelling electron microscope** (**STEM**)

Scanning Electron Microscope (SEM)

A **scanning electron microscope** (**SEM**) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about the sample's surface topography and composition. The electron beam is generally scanned in a raster scanpattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer.

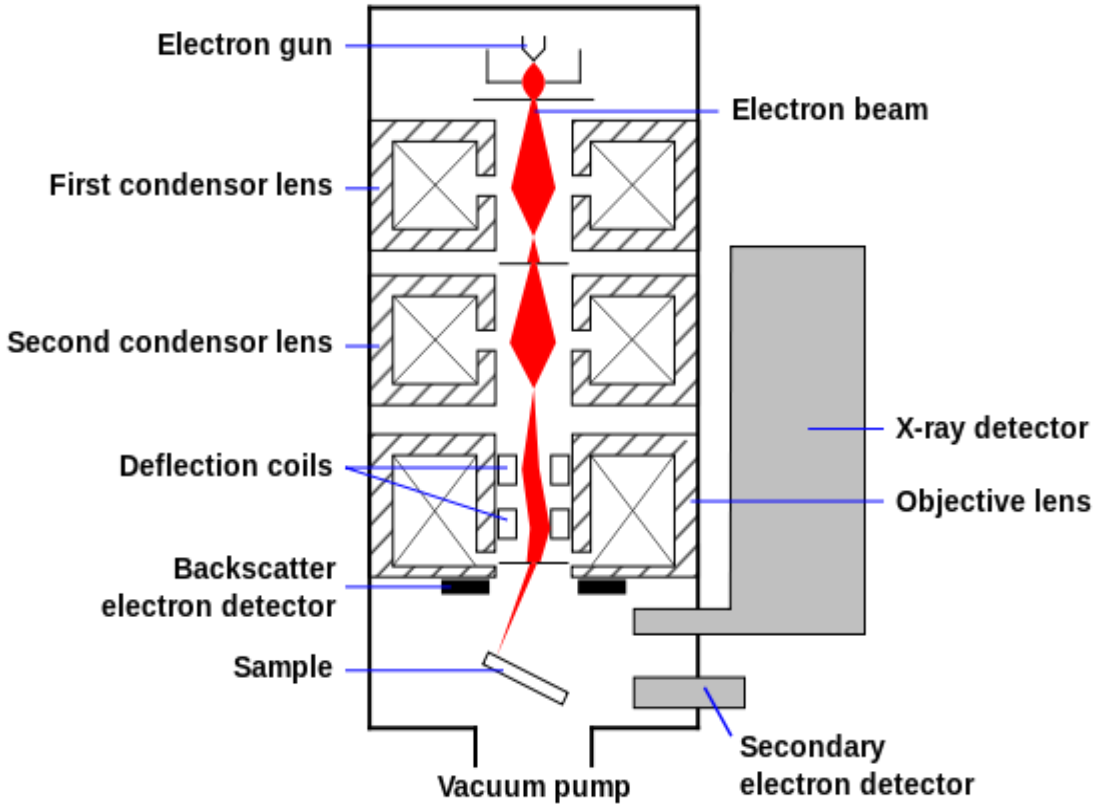


Fig. Schematic Diagram of SEM

In a typical SEM, an electron beam is thermionically emitted from an electron gun fitted with a tungsten filament cathode. Tungsten is normally used in thermionic electron guns because it has the highest melting point and lowest vapour pressure of all metals, thereby allowing it to be heated for electron emission, and because of its low cost. Other types of electron emitters include lanthanum hexaboride (LaB6) cathodes, which can be used in a standard tungsten filament SEM if the vacuum system is upgraded and FEG, which may be of the cold-cathode type using tungsten single crystal emitters or the thermally assisted Schottky type, using emitters of zirconium oxide.

The electron beam, which typically has an energy ranging from 0.2 keV to 40 keV, is focused by one or two condenser lenses to a spot about 0.4 nm to 5 nm in diameter. The beam passes through pairs of scanning coils or pairs of deflector plates in the electron column, typically in the final lens, which deflect the beam in the *x* and *y* axes so that it scans in a raster fashion over a rectangular area of the sample surface.

When the primary electron beam interacts with the sample, the electrons lose energy by repeated random scattering and absorption within a teardrop-shaped volume of the specimen known as the interaction volume, which extends from less than 100 nm to approximately 5 µm into the surface. The size of the interaction volume depends on the electron's landing energy, the atomic number of the specimen and the specimen's density. The energy exchange between the electron beam and the sample results in the reflection of high-energy electrons by elastic scattering, emission of secondary electrons by inelastic scattering and the emission of electromagnetic radiation, each of which can be detected by specialized detectors. The beam current absorbed by the specimen can also be detected and used to create images of the distribution of specimen current.Electronic amplifiers of various types are used to amplify the signals, which are displayed as variations in brightness on a computer monitor (or, for vintage models, on a cathode ray tube). Each pixel of computer videomemory is synchronized with the position of the beam on the specimen in the microscope, and the resulting image is therefore a distribution map of the intensity of the signal being emitted from the scanned area of the specimen. In older microscopes image may be captured by photography from a high-resolution cathode ray tube, but in modern machines image is saved to a computer data storage.

Sample-Electron Interaction

The scanning electron microscope (SEM) produces images by scanning the sample with a high-energy beam of electrons. As the electrons interact with the sample, they produce secondary electrons, backscattered electrons, and characteristic X-rays. These signals are collected by one or more detectors to form images which are then displayed on the computer screen. When the electron beam hits the surface of the sample, it penetrates the sample to a depth of a few microns, depending on the accelerating voltage and the density of the sample. Many signals, like secondary electrons and X-rays, are produced as a result of this interaction inside the sample.

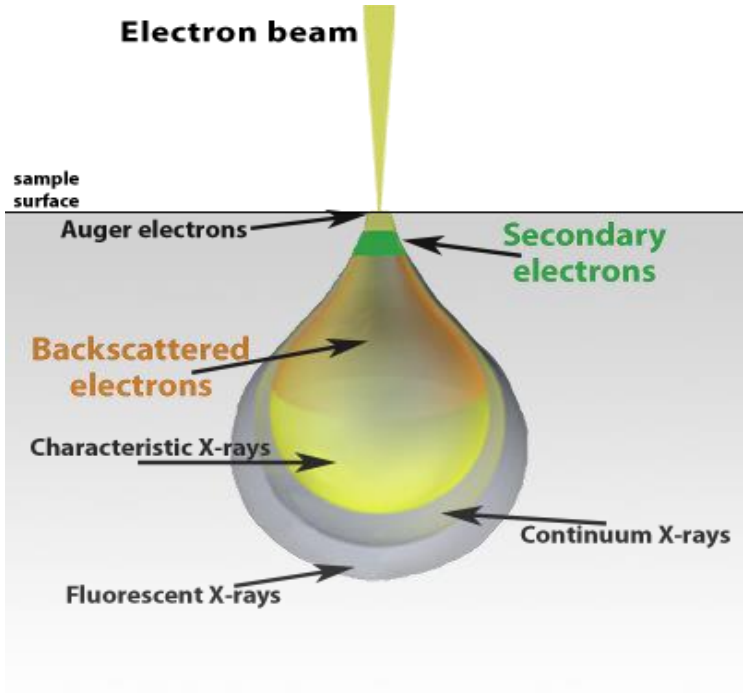


Fig. Schematic of electron beam interaction.

The maximum resolution obtained in an SEM depends on multiple factors, like the electron spot size and interaction volume of the electron beam with the sample. While it cannot provide atomic resolution, some SEMs can achieve resolution below 1 nm. Typically, modern full-sized SEMs provide resolution between 1-20 nm whereas desktop systems can provide a resolution of 20 nm or more.

Transmission Electron Microscope (TEM)

The transmission electron microscope is a very powerful tool for material science. A high energy beam of electrons is shone through a very thin sample, and the interactions between the electrons and the atoms can be used to observe features such as the crystal structure and features in the structure like dislocations and grain boundaries. Chemical analysis can also be performed. TEM can be used to study the growth of layers, their composition and defects in semiconductors. High resolution can be used to analyze the quality, shape, size and density of quantum wells, wires and dots. The TEM operates on the same basic principles as the light microscope but uses electrons instead of light. Because the wavelength of electrons is much smaller than that of light, the optimal resolution attainable for TEM images is many orders of magnitude better than that from a light microscope. Thus, TEMs can reveal the finest details of internal structure - in some cases as small as individual atoms.

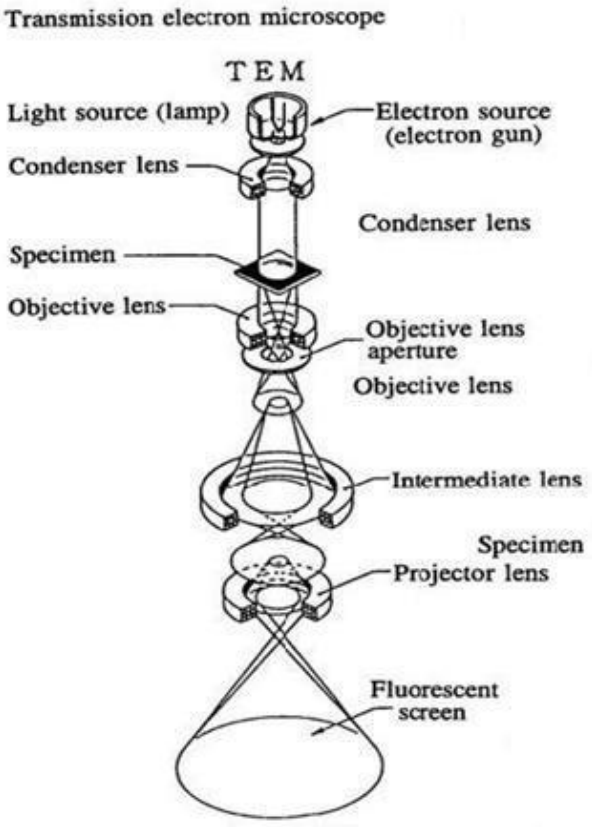


Fig 1

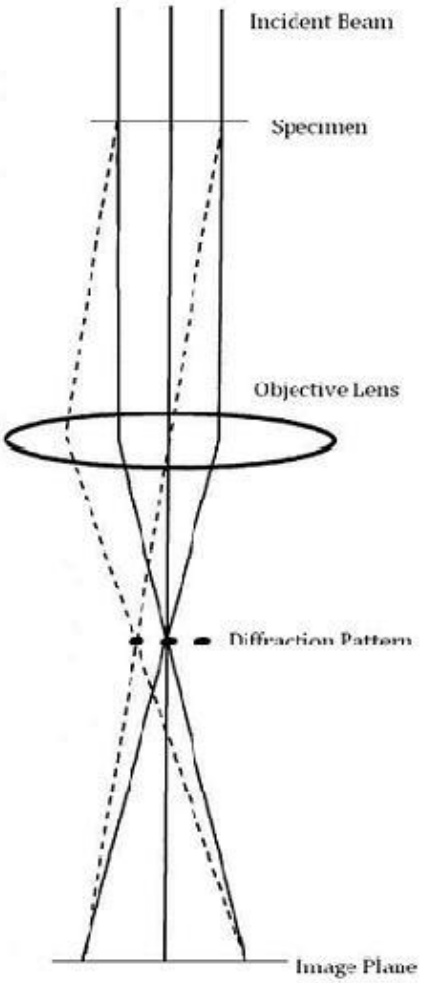


Fig 2

Fig 1 - General layout of a TEM describing the path of electron beam in a TEM

Fig 2 - A ray diagram for the diffraction mechanism in TEM

Imaging

The beam of electrons from the electron gun is focused into a small, thin, coherent beam by the use of the condenser lens. This beam is restricted by the condenser aperture, which excludes high angle electrons. The beam then strikes the specimen and parts of it are transmitted depending upon the thickness and electron transparency of the specimen. This transmitted portion is focused by the objective lens into an image on phosphor screen or charge coupled device (CCD) camera. Optional objective apertures can be used to enhance the contrast by blocking out high-angle diffracted electrons. The image then passed down the column through the intermediate and projector lenses, is enlarged all the way.

The image strikes the phosphor screen and light is generated, allowing the user to see the image. The darker areas of the image represent those areas of the sample that fewer electrons are transmitted through while the lighter areas of the image represent those areas of the sample that more electrons were transmitted through.

Diffraction

Fig2. shows a simple sketch of the path of a beam of electrons in a TEM from just above the specimen and down the column to the phosphor screen. As the electrons pass through the sample, they are scattered by the electrostatic potential set up by the constituent elements in the specimen. After passing through the specimen they pass through the electromagnetic objective lens which focuses all the electrons scattered from one point of the specimen into one point in the image plane. Also, shown in fig 2 is a dotted line where the electrons scattered in the same direction by the sample are collected into a single point. This is the back focal plane of the objective lens and is where the diffraction pattern is formed.

Specimen Preparation

A TEM specimen must be thin enough to transmit sufficient electrons to form an image with minimum energy loss. Therefore specimen preparation is an important aspect of the TEM analysis. For most electronic materials, a common sequence of preparation techniques is ultrasonic disk cutting, dimpling, and ion-milling. ***Dimpling*** is a preparation technique that produces a specimen with a thinned central area and an outer rim of sufficient thickness to permit ease of handling. ***Ion milling*** is traditionally the final form of specimen preparation. In this process, charged argon ions are accelerated to the specimen surface by the application of high voltage. The ion impingement upon the specimen surface removes material as a result of momentum transfer

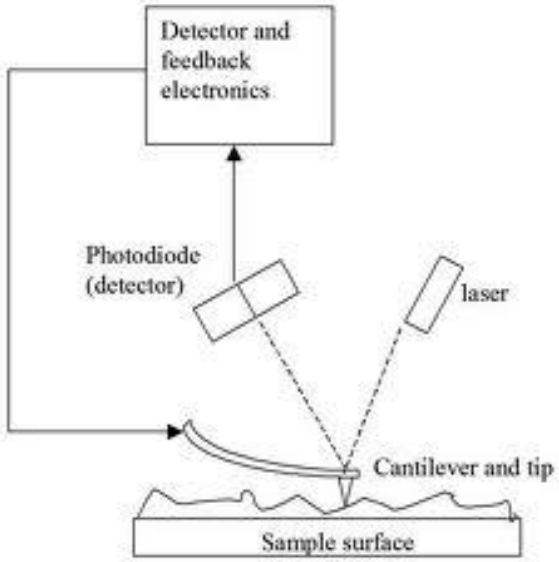
Scanning Probe Microscopy (SPM) is of two types

1. Atomic Force Microscopy (AFM)
2. Scanning tunnelling Microscopy (STM)

Atomic Force Microscopy (AFM)

Atomic Force Microscopy (AFM) is a form of scanning probe microscopy (SPM) where a small probe is scanned across the sample to obtain information about the sample's surface. The information gathered from the probe's interaction with the surface can be as simple as physical topography or as diverse as measurements of the material's physical, magnetic, or chemical properties. These data are collected as the probe is scanned in a raster pattern across the sample to form a map of the measured property relative to the X-Y position. Thus, the AFM microscopic image shows the variation in the measured property, e.g,. height or magnetic domains, over the area imaged.

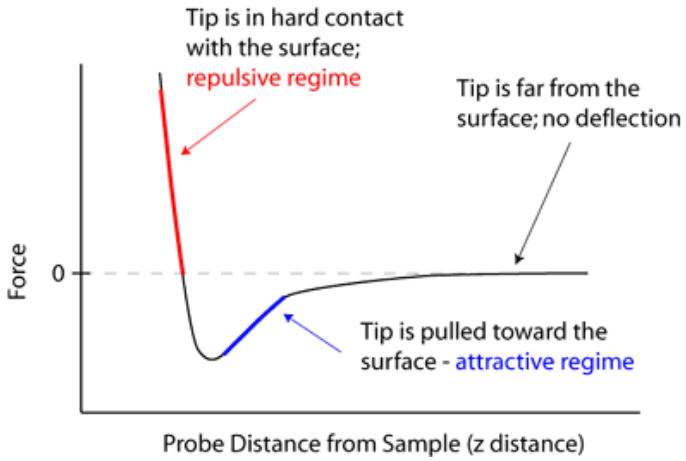
The AFM probe has a very sharp tip, often less than 100Å diameter, at the end of a small cantilever beam. The probe is attached to a piezoelectric scanner tube, which scans the probe across a selected area of the sample surface. Inter atomic forces between the probe tip and the sample surface cause the cantilever to deflect as the sample's surface topography (or other properties) changes. A laser light reflected from the back of the cantilever measures the deflection of the cantilever. This information is fed back to a computer, which generates a map of topography and/or other properties of interest. Areas as large as about 100 μm square to less than 100 nm square can be imaged.



The Atomic Force Microscope was developed to overcome a basic drawback with STM - that it can only image conducting or semiconducting surfaces. The AFM, however, has the advantage of imaging almost any type of surface, including polymers, ceramics, composites, glass, and biological samples.

Measuring forces

Because the atomic force microscope relies on the forces between the tip and sample, knowing these forces is important for proper imaging. The force is not measured directly, but calculated by measuring the deflection of the lever, and knowing the stiffness of the cantilever. Hook's law gives F = -kz, where F is the force, k is the stiffness of the lever, and z is the distance the lever is bent.



AFM Modes of operation

Because of AFM's versatility, it has been applied to a large number of research topics. The Atomic Force Microscope has also gone through many modifications for specific application requirements.

Contact Mode - The first and foremost mode of operation, contact mode is widely used. In this mode the AFM probe is scanned at a constant force between the probe and the sample surface to obtain a 3D topographical map. As the tip is raster-scanned across the surface, it is deflected as it moves over the surface corrugation. In constant force mode, the tip is constantly adjusted to maintain a constant deflection, and therefore constant height above the surface. It is this adjustment that is displayed as data. However, the ability to track the surface in this manner is limited by the feedback circuit. Sometimes the tip is allowed to scan without this adjustment, and one measures only the deflection. This is useful for small, high-speed atomic resolution scans, and is known as variable-deflection mode. Lateral resolution of <1 nm and height resolution of <1 Å can be obtained.

Because the tip is in hard contact with the surface, the stiffness of the lever needs to be less that the effective spring constant holding atoms together, which is on the order of 1 - 10 nN/nm. Most contact mode levers have a spring constant of < 1N/m.

Noncontact mode - Noncontact mode belongs to a family of AC modes, which refers to the use of an oscillating cantilever. A stiff cantilever is oscillated in the attractive regime, meaning that the tip is quite close to the sample, but not touching it (hence, "noncontact"). The forces between the tip and sample are quite low, on the order of pN (10^{-12} N). The detection scheme is based on measuring changes to the resonant frequency or amplitude of the cantilever.

Intermittent Contact (Tapping Mode) AFM - In this mode, the probe cantilever is oscillated at or near its resonant frequency. The oscillating probe tip is then scanned at a height where it barely touches or "taps" the sample surface. The system monitors the probe position and vibrational amplitude to obtain topographical and other property information. Accurate topographical information can be obtained even for very fragile surfaces. Optimum resolution is about 50 Å lateral and <1 Å height. Images for phase detection mode, magnetic domains, and local electric fields are also obtained in this mode. The advantage of tapping the surface is improved lateral resolution on soft samples. Lateral forces such as drag, common in contact mode, are virtually eliminated. For poorly adsorbed specimens on a substrate surface the advantage is clearly seen.

Lateral Force Microscopy - This mode measures the lateral deflection of the probe cantilever as the tip is scanned across the sample in contact mode. Changes in lateral deflection represent relative frictional forces between the probe tip and the sample surface.

Magnetic Force Microscopy - This mode images local variations in the magnetic forces at the sample's surface. The probe tip is coated with a thin film of ferromagnetic material that will react to the magnetic domains on the sample surface. The magnetic forces between the tip and the sample are measured by monitoring cantilever deflection while the probe is scanned at a constant height above the surface. A map of the forces shows the sample's natural or applied magnetic domain structure.

Image Analysis - Since the images are collected in digital format, a wide variety of image manipulations are available for AFM data. Quantitative topographical information, such as lateral spacing, step height, and surface roughness are readily obtained. Images can be presented as two-dimensional or three-dimensional representations in hard copy or as digital image files for electronic transfer and publication.

Nanoindentation - A specialized probe tip is forced into the sample surface to obtain a measure of the material's mechanical properties in regions as small as a few nanometers.

TYPICAL APPLICATIONS

3-dimensional topography of IC device
Roughness measurements for chemical mechanical polishing
Analysis of microscopic phase distribution in polymers
Mechanical and physical property measurements for thin films
Imaging magnetic domains on digital storage media
Imaging of submicron phases in metals
Defect imaging in IC failure analysis
Microscopic imaging of fragile biological samples
Metrology for compact disk stampers

Sample Requirements

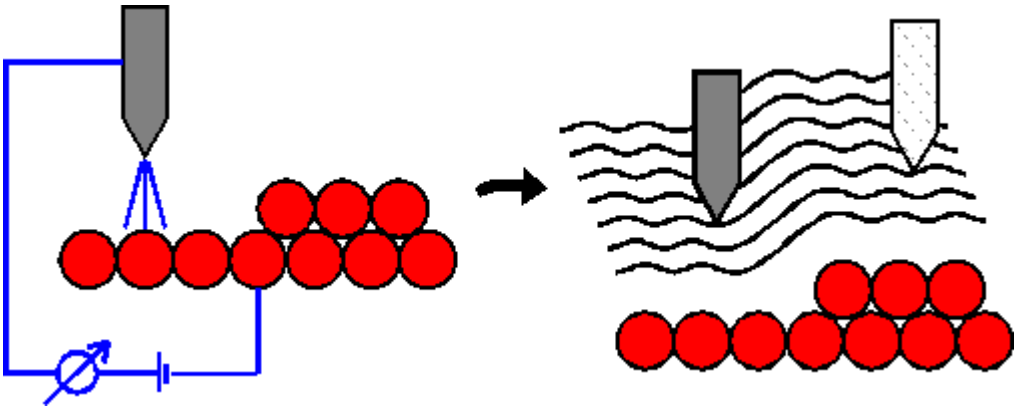
No sample preparation is typically required. Samples can be imaged in air or liquid. Sample height is limited to about 1.5 inches. Areas up to 8 inches in diameter can be fully traversed without repositioning. Larger samples can be fixtured for imaging within a limited area. Total surface roughness in the image area should not exceed about 6 μm.

SCANNING TUNNELING MICROSCOPE (STM)

1. Concept

The Scanning Tunneling Microscope (STM) was introduced by G. Binnig and W. Rohrer at the IBM Research Laboratory in 1982 which was honoured by the Noble Prize in 1986. It has become widely used as an important instrument for real space analysis in surface science.

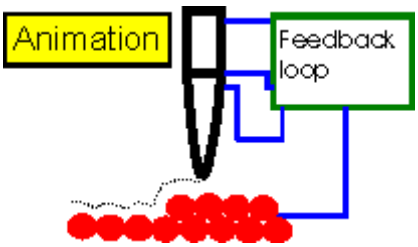
The basic idea is to bring a fine metallic tip in close proximity (a few Å) to a conductive sample. By applying a voltage ($U \lesssim 4V$) between the tip and the sample a small electric current (0.01nA-50nA) can flow from the sample to the tip or reverse, although the tip is not in physical contact with the sample. This phenomenon is called electron tunneling . The exponential dependence of the tunneling current on the tip to sample distance results in a high vertical resolution. By scanning the tip across the surface and detecting the current (one can also use the current as a vertical positioning signal for the tip - see Modes of Operation) a map of the surface can be generated with a resolution in the order of atomic distances. It has to be mentioned that the image cannot just be interpreted as a topographic map as the tunneling current is influenced by the lateral and vertical variation of the electronic state density at the surface. The lateral resolution is about 1Å whereas a vertical resolution up to 0.01Å can be achieved. The STM can be used in ultra high vacuum, air or other environments.



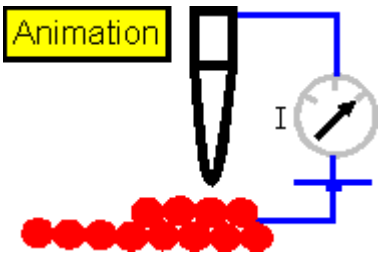
2. Modes of Operation

2.1. Constant Current Mode

By using a feedback loop the tip is vertically adjusted in such a way that the current always stays constant. As the current is proportional to the local density of states, the tip follows a contour of a constant density of states during scanning. A kind of a topographic image of the surface is generated by recording the vertical position of the tip.



2.2. Constant Height Mode



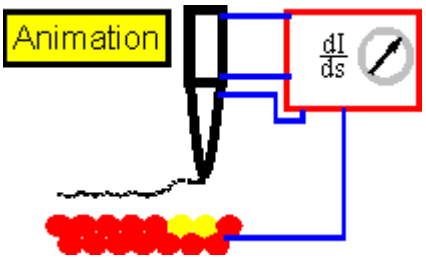
In this mode the vertical position of the tip is not changed, equivalent to a slow or disabled feedback. The current as a function of lateral position represents the surface image. This mode is only appropriate for atomically flat surfaces as otherwise a tip crash would be inevitable. One of its advantages is that it can be used at high scanning frequencies (up to 10 kHz). In comparison, the scanning frequency in the constant current mode is about 1 image per second or even per several minutes.

2.3. Barrier Height Imaging

Up to now homogoneous surfaces were considered. If there is an inhomogeneous compound in the surface the work function will be inhomogeneous as well. This alters the local barrier height. By using the two modes described above we would get a virtual hole or adatom. But this can further be explored:
Differentiation of (*) yields

$$\frac{d(\ln I)}{ds} \propto \sqrt{\Phi}$$

.



Thus the work function can directly be measured by variing the tip-sample distance, which can be done by modulating the current with the feedback turned on.

3.4. Scanning Tunneling Spectroscopy

If the matrix element and the density of states of the tip is nearly constant, the tunneling current (**) can be estimated to

$$I \propto \int_0^{eV} \rho_{sa}\left(E_F - eV + \varepsilon\right) d\varepsilon$$

Differentiation yields the density of states

$$\frac{dI}{dV} \propto \rho_{sa}(E_F - eV)$$

The density of states can be deduced by

- Modulation of the bias voltage
- Current-Imaging Tunnelling Spectroscopy (CITS): The tip is scanned in the constant current mode to give a constant distance to the sample. At each point the feedback loop is disabled and a current-voltage curve (I+V curve) is recorded.

3. Technical Aspects

Demands

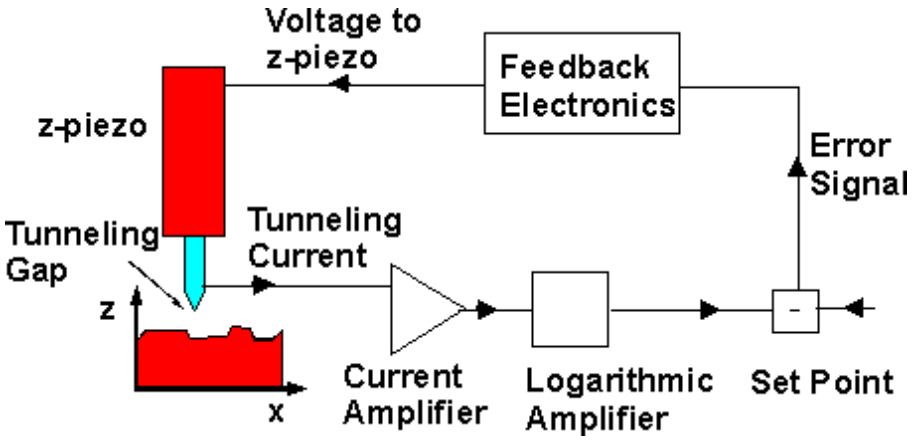
- Controlling the tip-sample distance from a few mm down to 0.01Å
- Exact lateral positioning
- Stabilized tip-sample distance
- Sharp tip
- Measuring a current in the range of 0.01nA-50nA

3.1. Positioning

The large distance range the tip has to be controlled on makes it necessary to use two positioners: a coarse and a fine positioner. The fine positioner is also used as a scanner. Every fine positioner/scanner is made out of a piezocrystal or piezoceramic material.

3.2. Electronic Circuit

In case of measuring in the constant current mode a feedback circuit has to be build up to control the z-piezo.



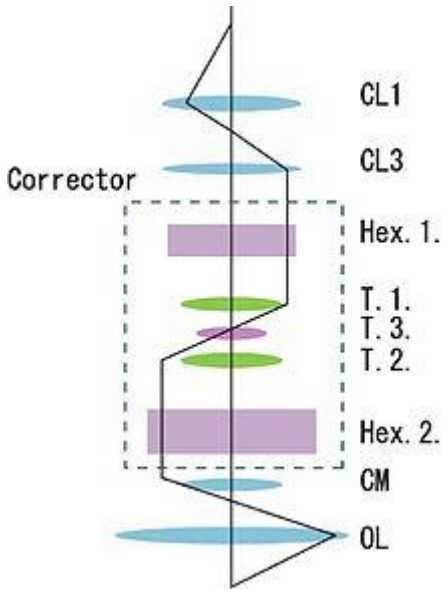
The tunnelling current (0.01mA-50mA) is converted into a voltage by a current amplifier. To get a linear response with respect to the tunnelling gap (the current is exponentially dependant on the

tip-sample distance) the signal is processed by a logarithmic amplifier. The output of the logarithmic amplifier is compared with a predetermined voltage which is used as a reference current. The error signal is passed to feedback electronics, which applies a voltage to the z piezo to keep the difference between the current set point and the tunnelling current small. Care has to be taken to keep the noise signal ratio on a low level. Also the response time of the feedback has to be minimized without losing accuracy.

Scanning transmission electron microscopy (STEM)

Scanning transmission electron microscopy (STEM) combines the principles of transmission electron microscopy and scanning electron microscopy and can be performed on either type of instrument. Like TEM, STEM requires very thin samples and looks primarily at beam electrons transmitted by the sample. One of its principal advantages over TEM is in enabling the use of other of signals that cannot be spatially correlated in TEM, including secondary electrons, scattered beam electrons, characteristic X-rays, and electron energy loss.

Like SEM, the STEM technique scans a very finely focused beam of electrons across the sample in a raster pattern. Interactions between the beam electrons and sample atoms generate a serial signal stream, which is correlated with beam position to build a virtual image in which the signal level at any location in the sample is represented by the gray level at the corresponding location in the image. Its primary advantage over conventional SEM imaging is the improvement in spatial resolution.



Scattered beam electrons. Beam electrons may be elastically scattered by the nuclei of sample atoms. In a bulk specimen in a SEM, elastically scattered beam electrons that have been directed back out of the sample constitute the backscattered electron (BSE) signal. In STEM, transmitted

beam electrons that have been scattered through a relatively large angle are detected using a high angle annular dark field (HAADF) detector.

X-ray microanalysis. Electrons bombarding the specimen cause it to emit X-rays whose energy is characteristic of the elemental composition of the sample. X-ray microanalysis uses an energy dispersive X-ray (EDX) spectrometer to count and sort characteristic X-rays according to their energy.

Wavelength dispersive X-ray (WDX) spectrometry measures and counts X-rays by their wavelength (a correlate of energy). A wavelength spectrometer uses a crystal or grating with known spacing to diffract characteristic X-rays.

Electron energy loss spectrometry (EELS) analyzes transmitted electrons to determine the amount of energy they have lost in interactions with the sample. It provides information about the interacting atoms, including elemental identity, chemical bonding, valence and conduction band electronic properties, surface properties, and element-specific pair distance distribution functions.