Imaging Methods: Scanning Force Microscopy (SFM / AFM)

The atomic force microscope (AFM) probes the surface of a sample with a sharp tip, a couple of microns long and often less than 100 Å in diameter. The tip is located at the free end of a cantilever that is 100 to 200 μm long. Forces ($10^{-8} - 10^{-6}$ N) between the tip and the sample surface cause the cantilever to bend, or deflect. A detector measures the cantilever deflection as the tip is scanned over the sample, or the sample is scanned under the tip. The measured cantilever deflections allow a computer to generate a map of surface topography. AFMs can be used to study insulators and semiconductors as well as electrical conductors.

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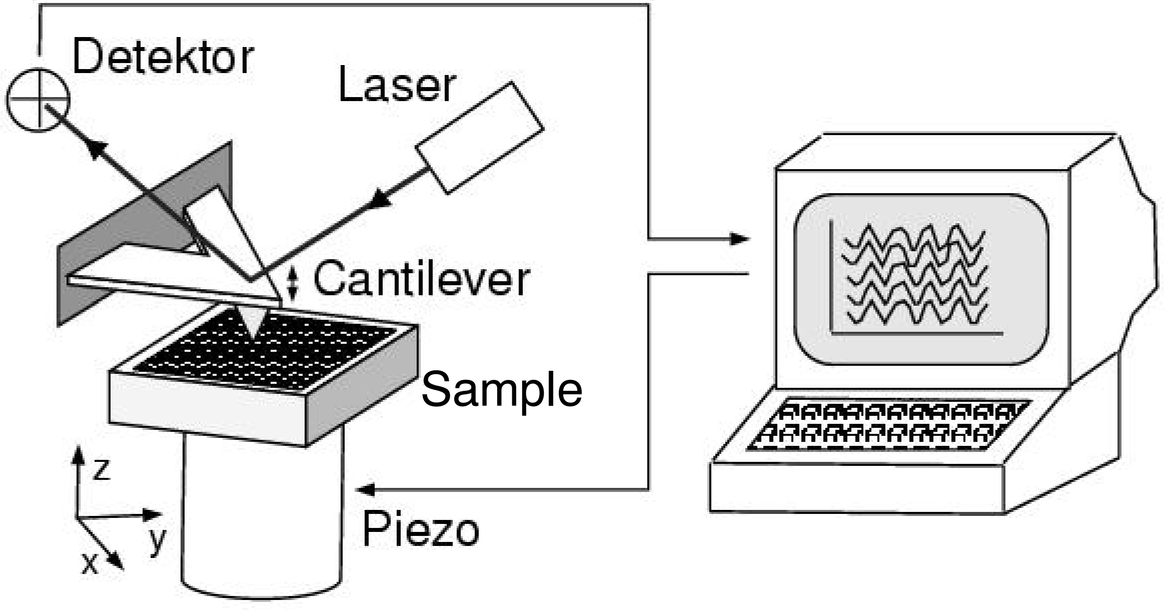
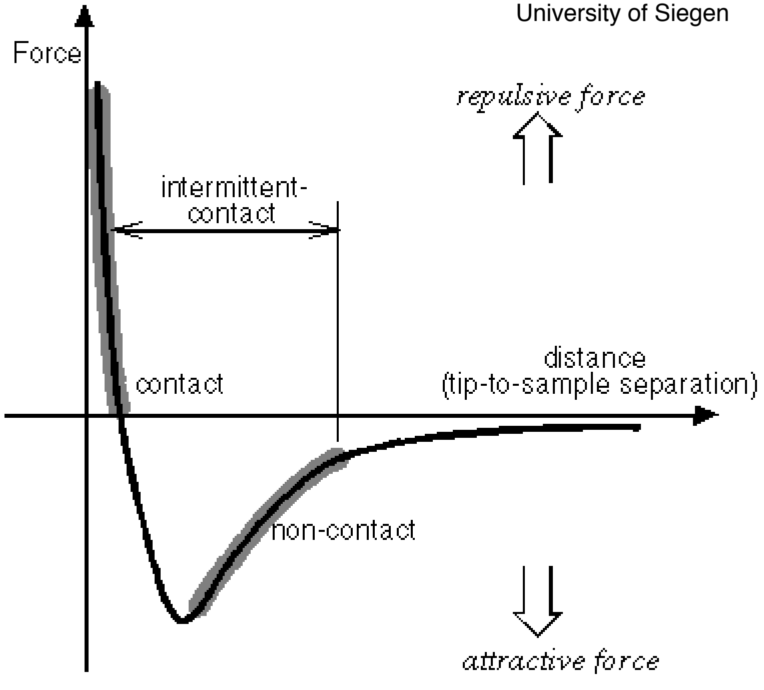
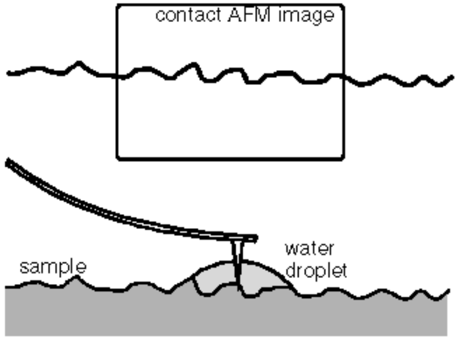
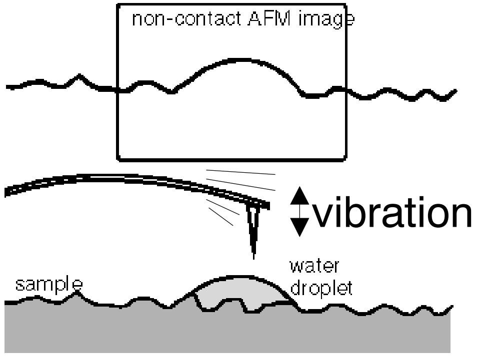




SFM / AFM: Contact, Non− / Intermittent Contact, Friction



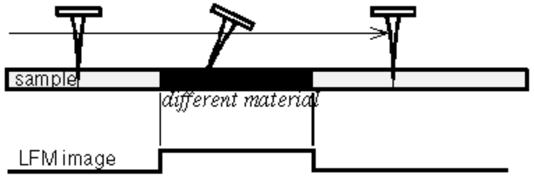
contact (repulsive) **mode**: tip makes soft "physical contact" with the sample, the tip is attached to the end of a cantilever with a low spring constant (lower than the effective spring constant holding the atoms of the sample together), the contact force causes the cantilever to bend to accommodate changes in topography

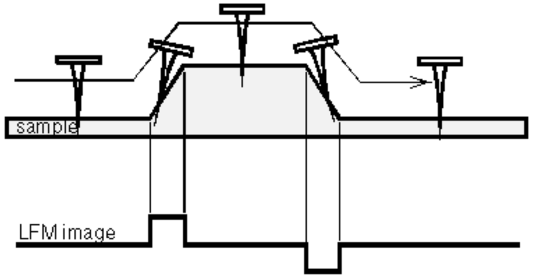


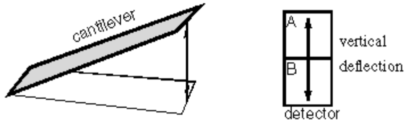
non−contact / **intermittent contact**: AFM cantilever is vibrated near the surface of a sample with spacing on the order of tens to hundreds of angstroms for non−contact or touching of the surface at lowest deflection for intermittent contact ("tapping mode")

phase mode: compare phase of driving signal and cantilever response (information on elastic modulus of surface material

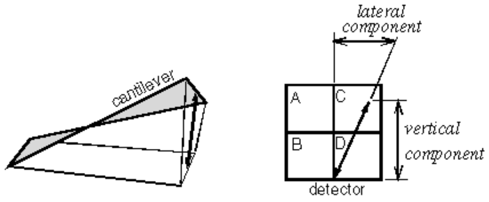
lateral force / **friction mode**: AFM cantilever in contact mode is laterally deflected in the sample plane due to scanning motion perpendicular to cantilever axes, lateral deflection is measured and gives information on surface material apart from topography







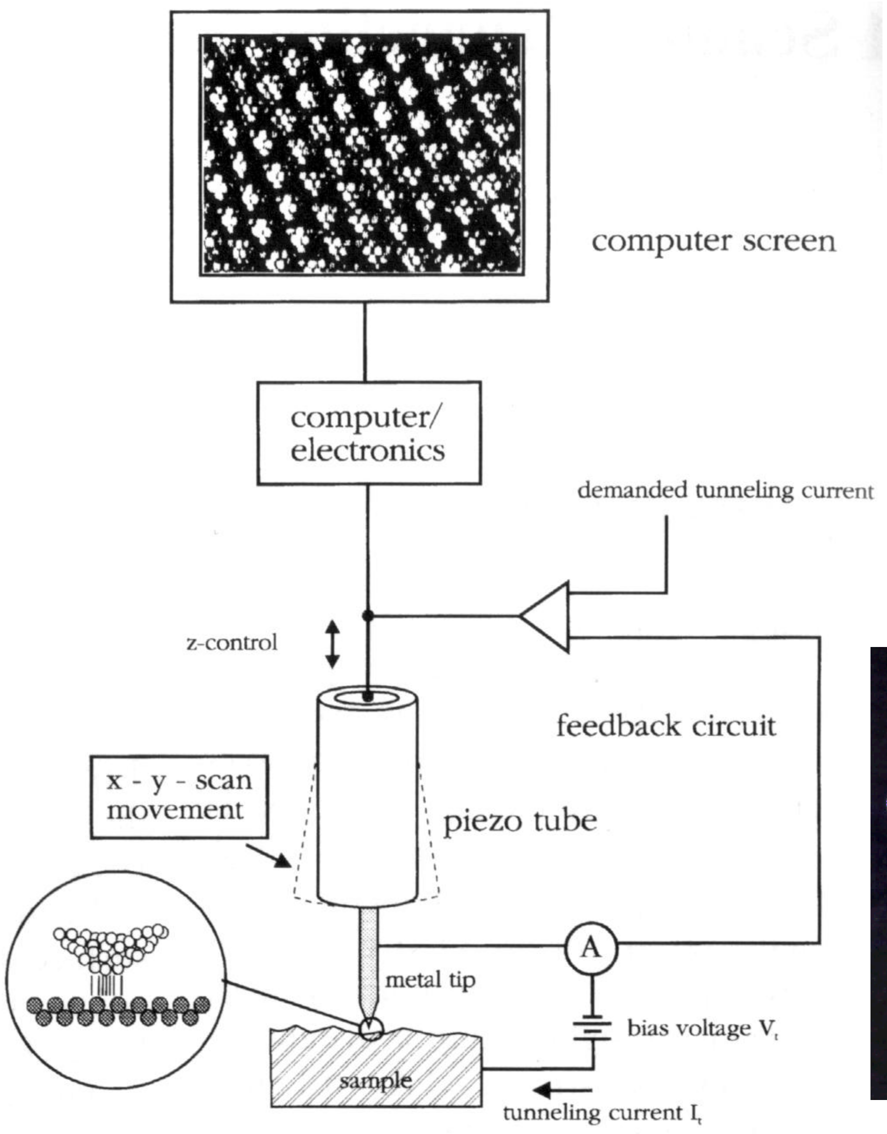
vertical deflection: topography

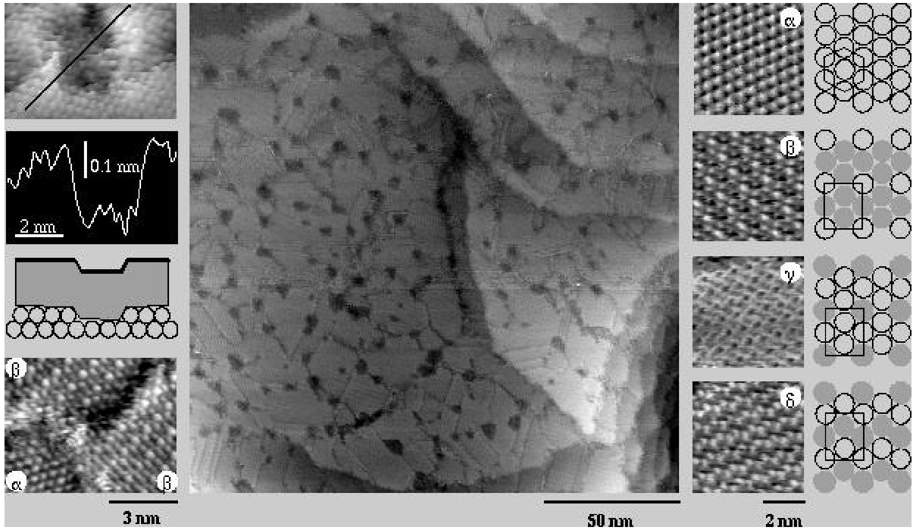


horizontal deflection: friction

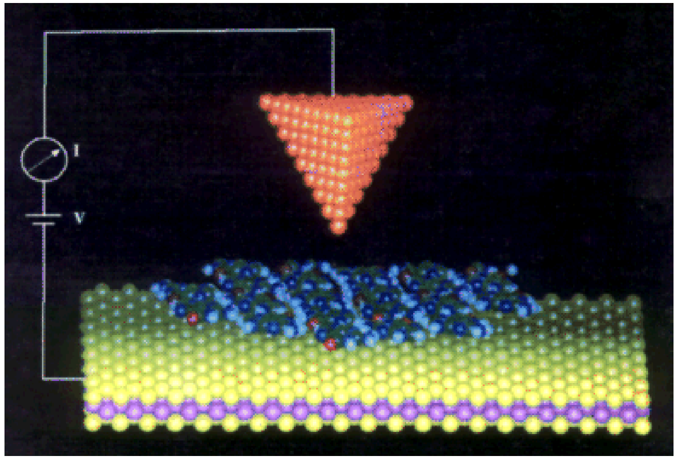
Imaging Methods: Scanning Tunneling Microscopy (STM)

sharp conducting tip is scanned over conducting surface and electrons tunneling between tip and surface (depends on bias voltage) at a separation below ~10 angstroms are measured with respect to tip position





monolayers of dodecanethiol $C_{12}H_{25}SH$ on gold (111)



tunneling principle of STM:

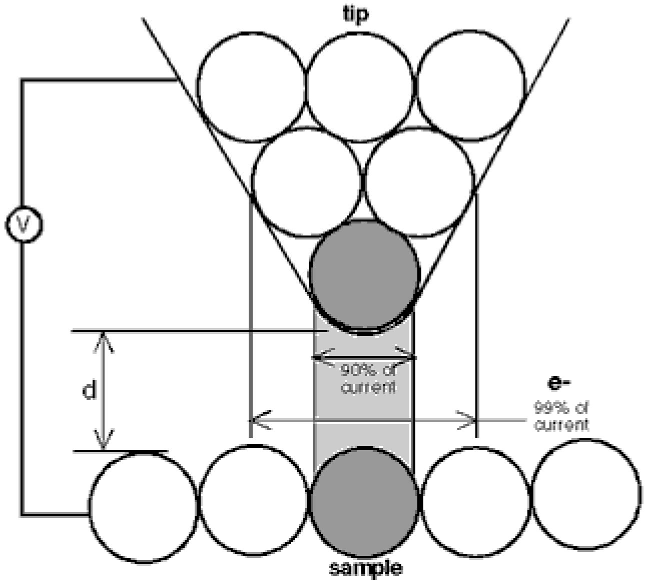
$$I_{tunnel} \rightarrow U \frac{k}{s} e^{(-2ks)}$$

I: tunnel current
U tunneling voltage
1/k: range of WF over surf.
s: distance tip–surface

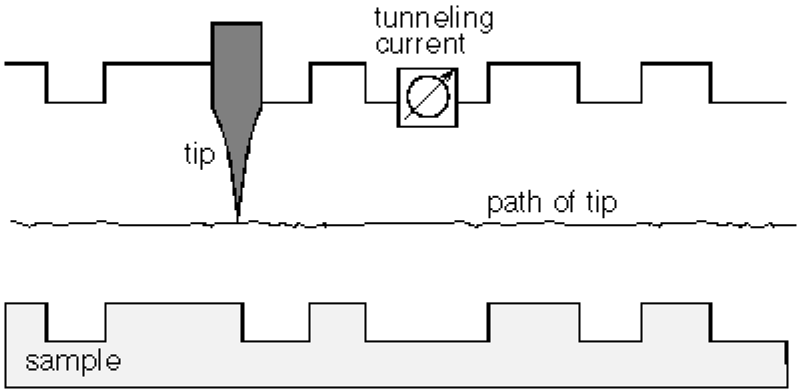
STM: Constant–Height versus Constant–Current Mode

constant height mode: the tip is scanned over the surface keeping the vertical tip position constant, topography / conductivity differences are mapped by recording variations in tunnel current with respect to x–y–position of tip

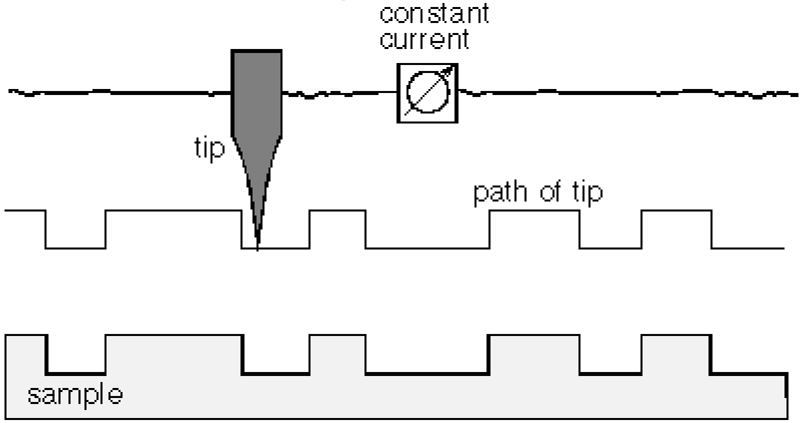
constant current mode: the vertical tip position is adjusted during scanning to keep tunnel current constant, topography / conductivity map is constructed from vertical tip position with respect to x–y–position



high resolution possible since most of the tunnel current (~90 %) flows within the shortest tip–surface separation (exponential distance dependence of tunnel current)



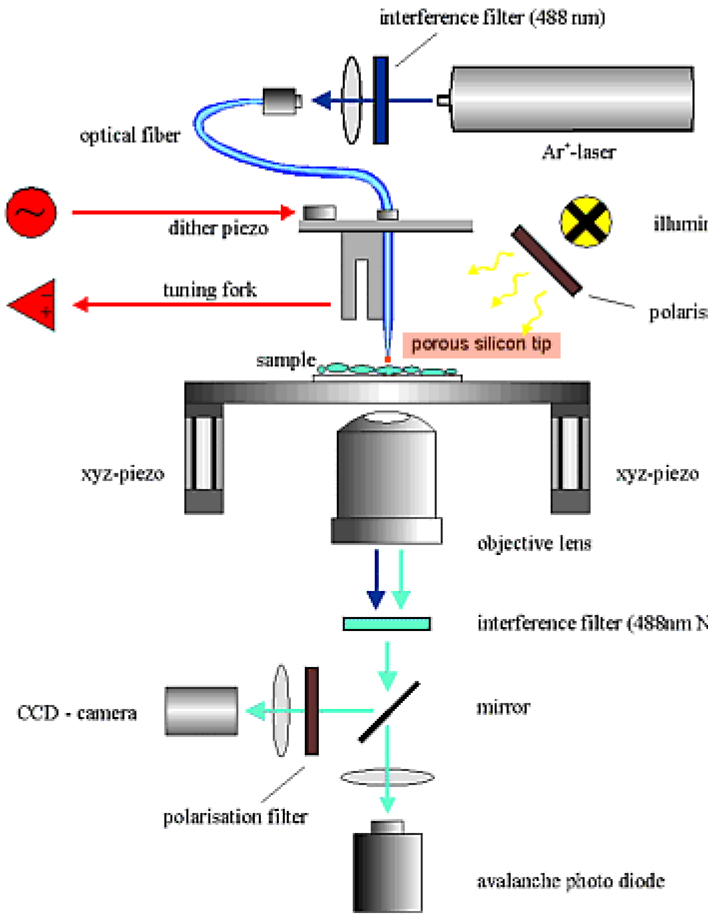
constant height mode
(flat surface, high resolution, fast scanning)

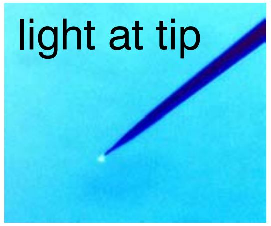


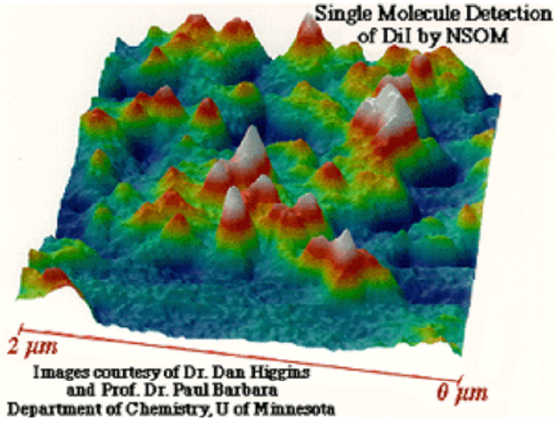
constant current mode
(rich topography, lower resolution)

Imaging Methods: Nearfield Scanning Optical Microscopy (NSOM)

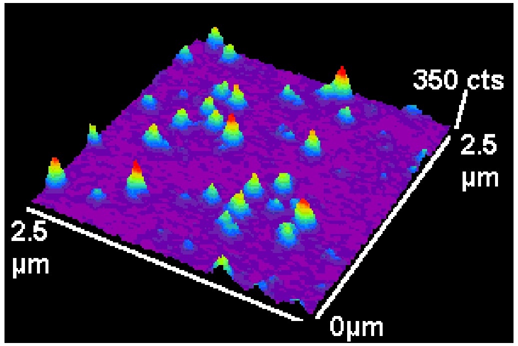
integration of optical microscopy tools with scanning probe techniques allows resolution far beyond optical diffraction limit, sample is excited by light coming from a wave guide tip with sub-micron aperture which is scanned over the surface, light coming from the probe is collected in an optical microscope objective, light intensity is recorded with respect to tip y–x–position

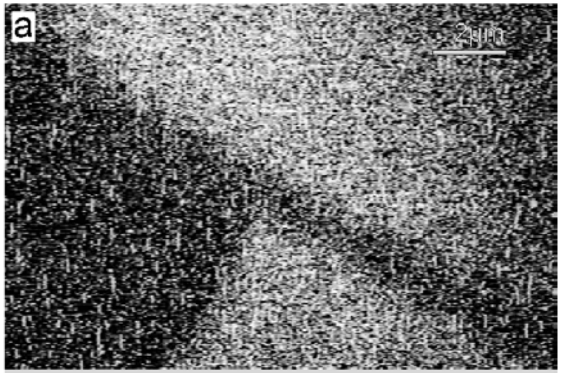




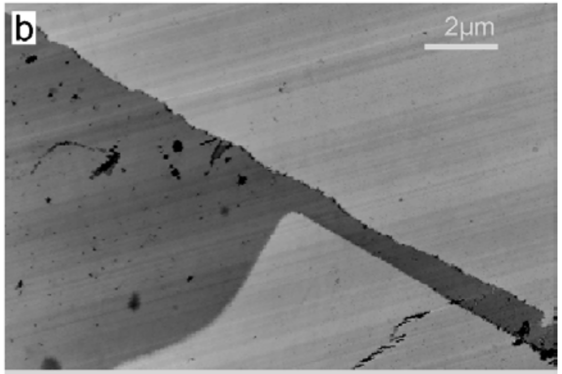


Images courtesy of Dr. Dan Higgins and Prof. Dr. Paul Barbara Department of Chemistry, U of Minnesota





NFO - image: 15 µm x 10 µm

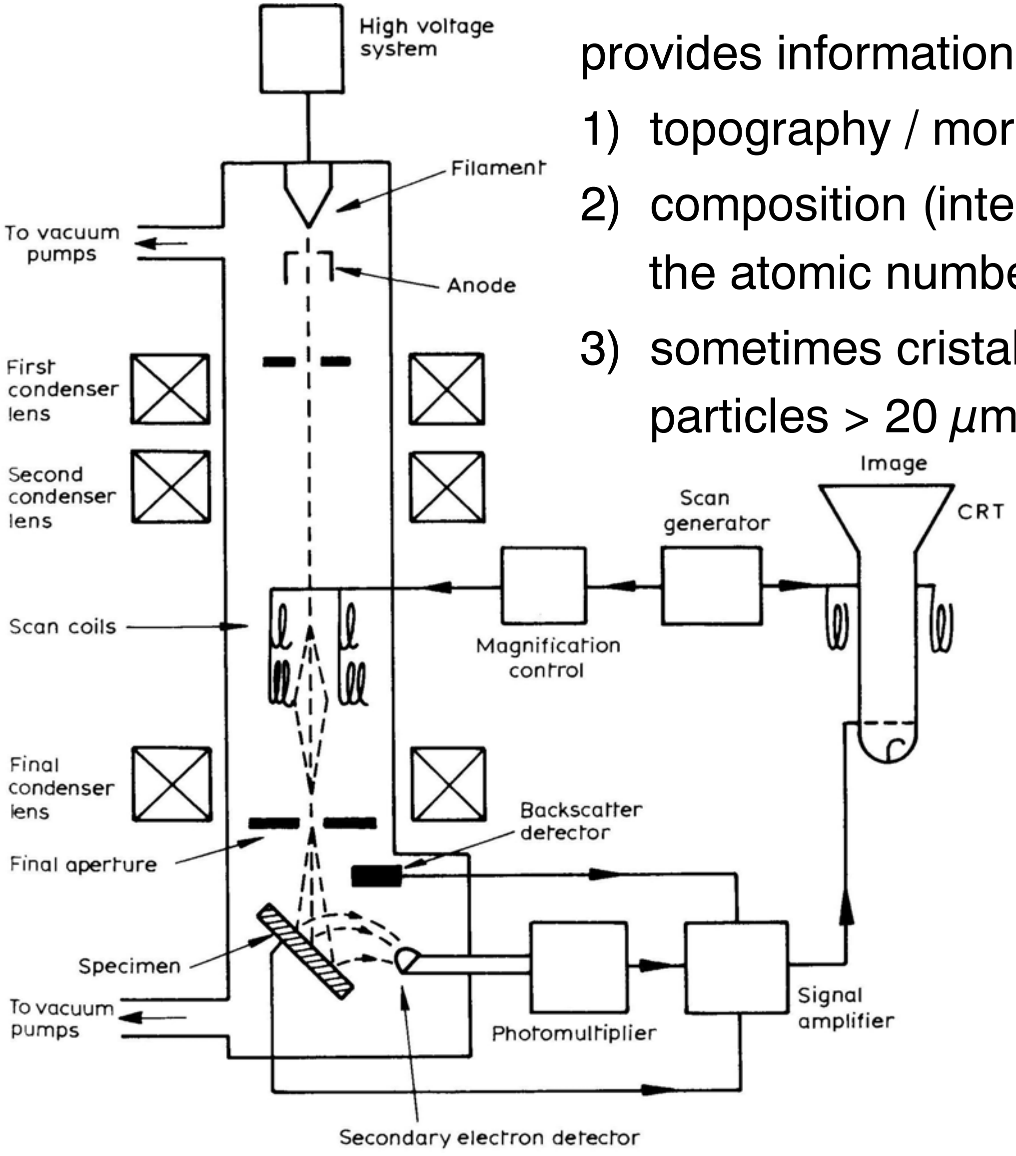


AFM - image: 15 µm x 10 µm

gold film on glass

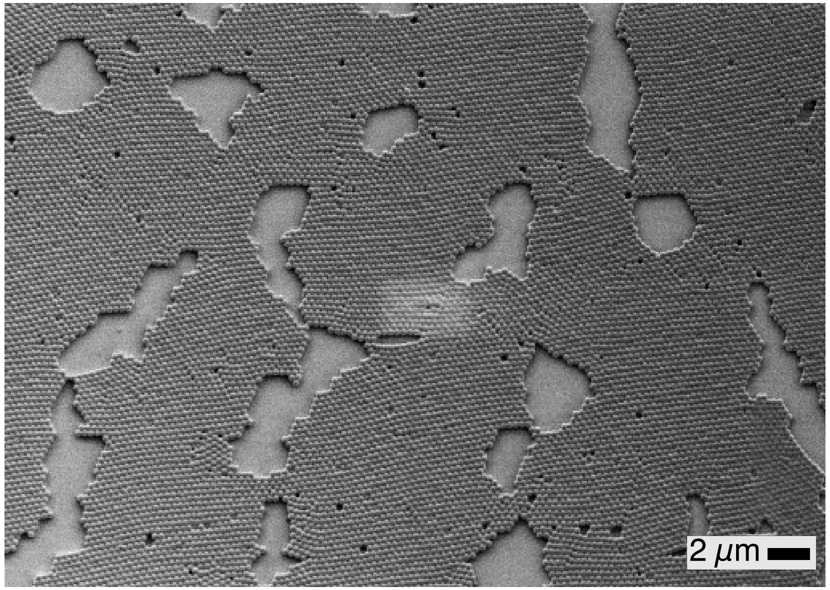
Imaging Methods: Scanning Electron Microscopy (SEM)

scanning of electron beam (0.2 – 30 keV) over a (usually conducting) specimen and detection of secondary low energy or backscattered electrons, resolution from mm down to about 5 nm



provides information on:

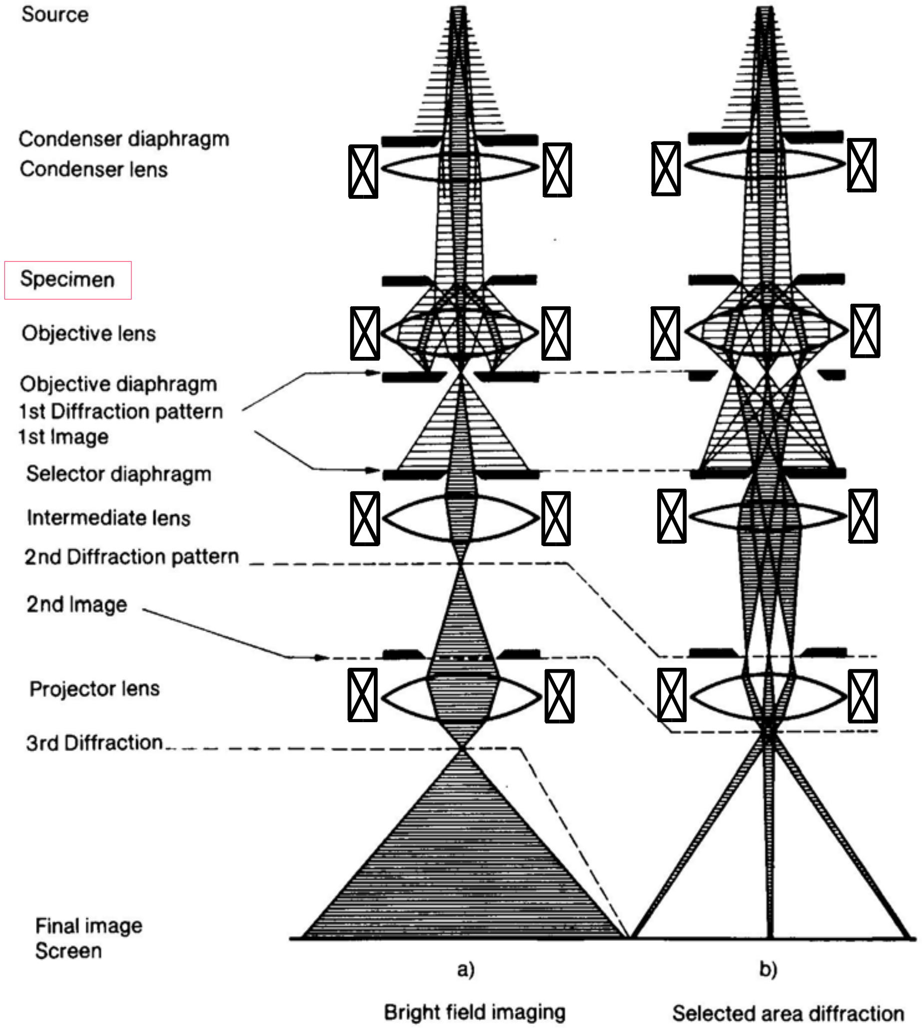
1) topography / morphology (surface profile, structural features)
2) composition (intensity of backscattered electrons correlates to the atomic number of elements within the sampling volume)
3) sometimes cristallographic information (single-crystal particles > 20 μm)



monolayer of colloidal polymer particles (280 nm)

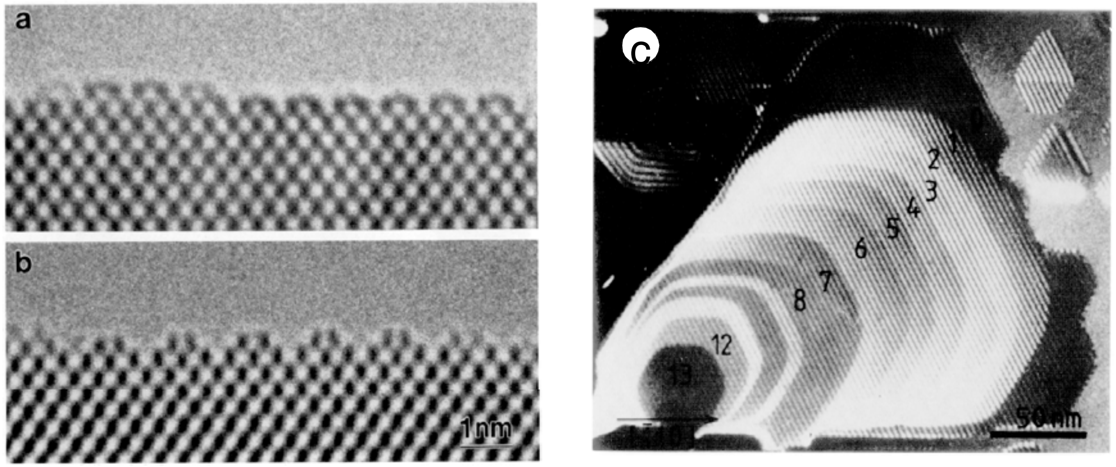
Imaging Methods: Transmission Electron Microscopy (TEM)

transillumination of a thin specimen (~ 30−100 nm) with high energy electron beam allowing high resolution imaging or electron beam diffraction in crystalline samples: acceleration voltage 100 keV → $\lambda = 3.7$ pm; 1 MeV → $\lambda = 0.87$ pm



a) Bright field imaging

b) Selected area diffraction



a) TEM profile images of CdTe crystal edge (100) face along [110] projection, 2x1 Cd−rich reconstruction (140°C)

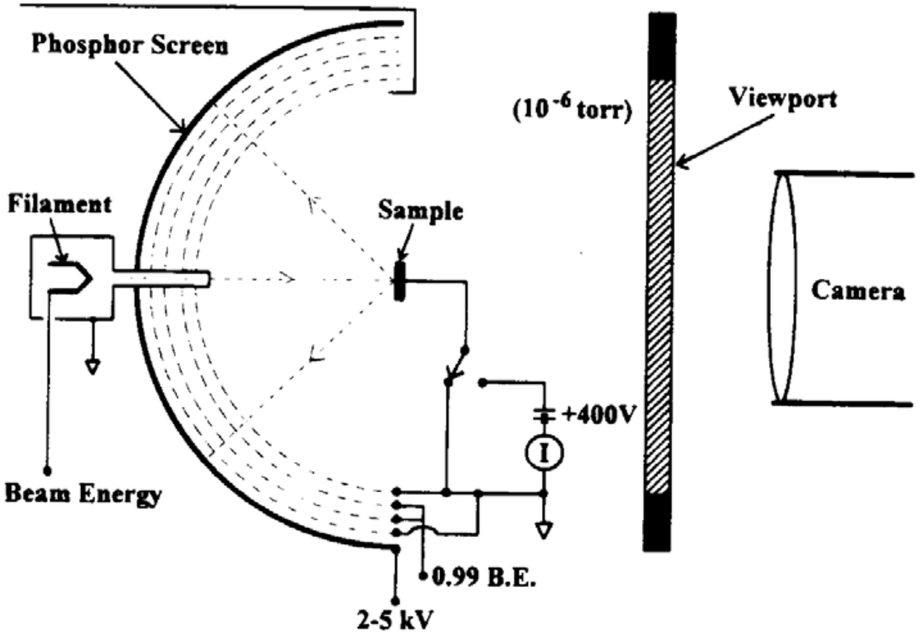
b) like a), 3x1 Te−rich reconstruction (240°C)

c) darkfield TEM of thin Ag layers deposited onto thin MoS_2, numbers give thickness in monolayers

Imaging Methods: Low Energy Electron Diffraction (LEED)

LEED is used to study the symmetry, periodicity and atomic arrangement of solid crystal surfaces and thin films. The LEED pattern symmetry, peak position and intensities give direct information on surface lattice parameters and the position of atoms in the surface unit cell.

LEED principle: low energy electrons (10−500 eV) are impinging onto a substrate surface and ~ 1 % (high interaction of electrons with matter) are elastically reflected to a phosphor screen, a diffraction pattern can be observed if lateral order at surface is beyond 20 nm

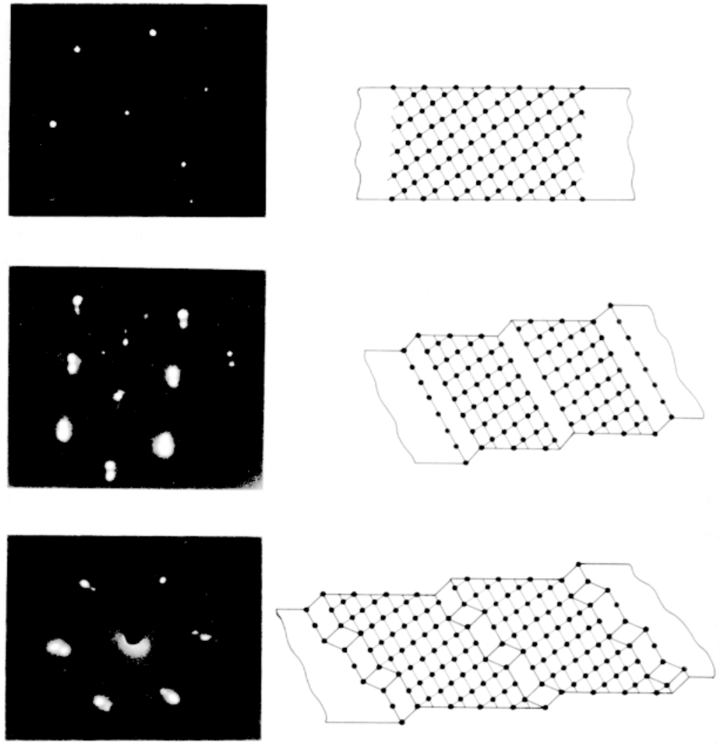


schematics of LEED device

low defect density

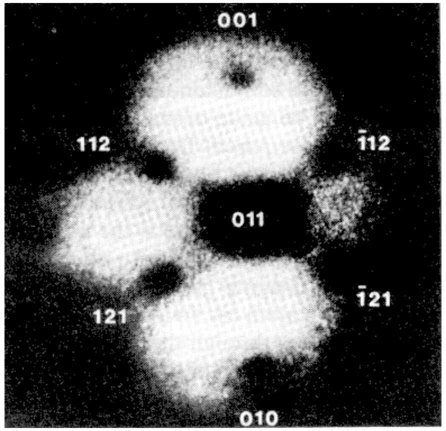
regular steps

regular steps
with kinks



assignment of LEED pattern to defect density of platinum surface

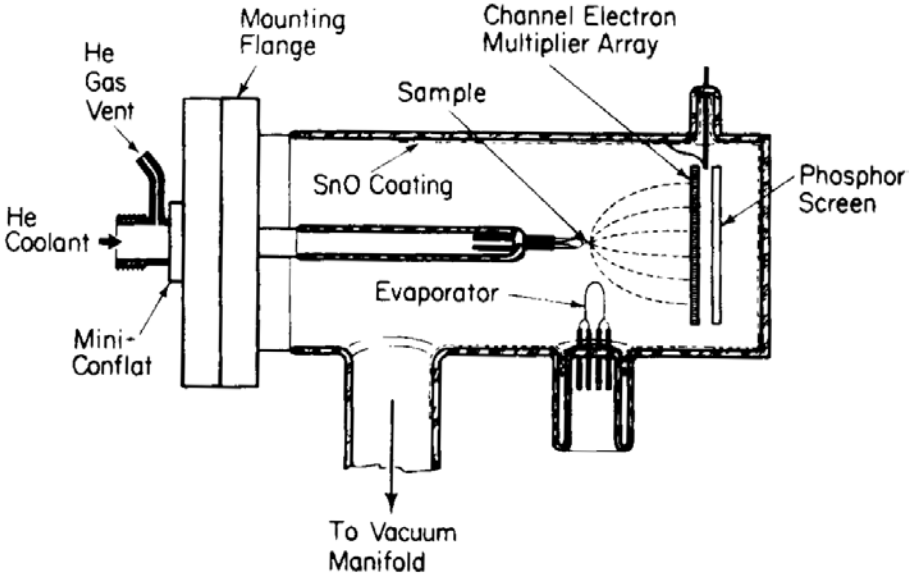
Imaging Methods: Field Emission Microscopy (FEM) and Field Ionization Microscopy (FIM)



FEM of crystalline W tip

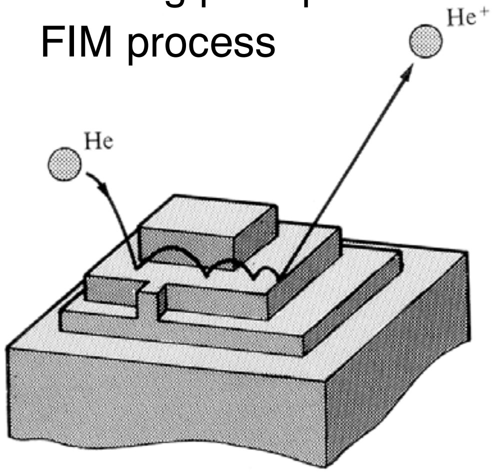
FEM: to a sharp metal tip (radius <1000 nm) in high vacuum (~10^{-11} torr) a high potential (≥1.5 kV) is applied, electrons are emitted depending on local work function (surface structure dependent) and impinge on fluorescent screen in point projection geometry → spots on screen can be assigned to exposed crystal faces of tip

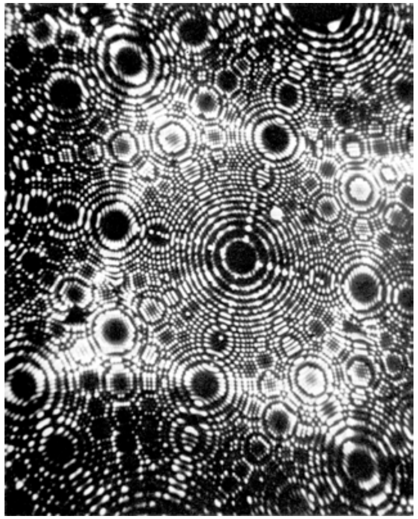
FIM: to metal tip (r ~10 nm) at ~20 K in He (or Ne, Ar, H_2) atmosphere (down to 10^{-10} torr) a potential (<20 kV) is applied (reverse polarity to FEM), gas ions at the surface get ionized and accelerated away from tip to phosphor screen in point projection geometry



schematics of FIM device (similar to FEM)

working principle of FIM process





FIM of crystalline Ir tip