STM, LEED and Mass spectrometry

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M. Edelwirth, W.M. Heckl

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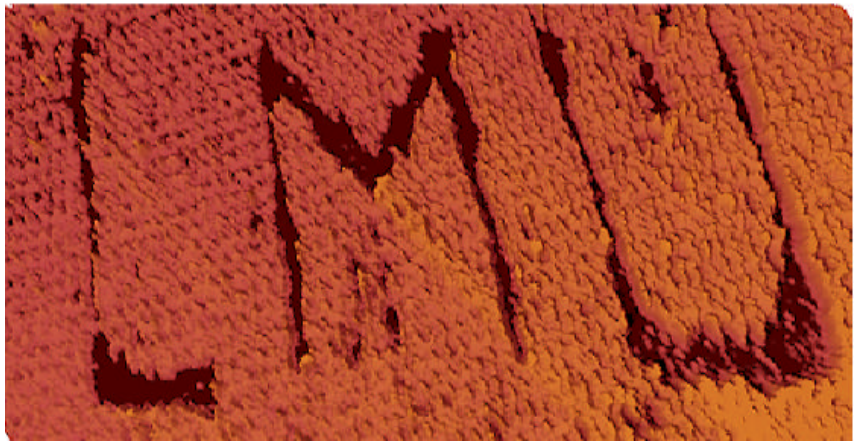


P. Cole, M. Reiter

Introduction

Motivation:

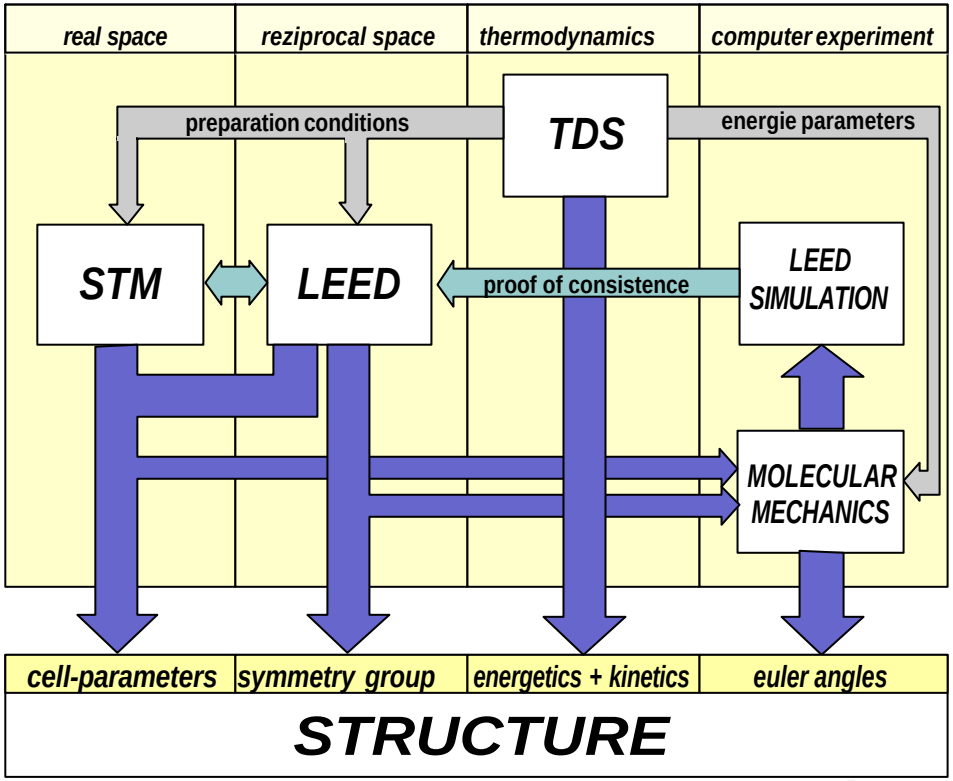
- Preparation of ordered monolayers by self-assembly
- Structure determination
- Nanomanipulation
- Idea: Preparation of masks from small molecules

Focus:

Small organic molecules (e.g. DNA-bases, liquid crystals)

on Ag, graphite, MoS_2

Experimental concept for the structure determination of self-assembled nucleic acid base layers



UHV technique (Ultra High Vacuum) 1

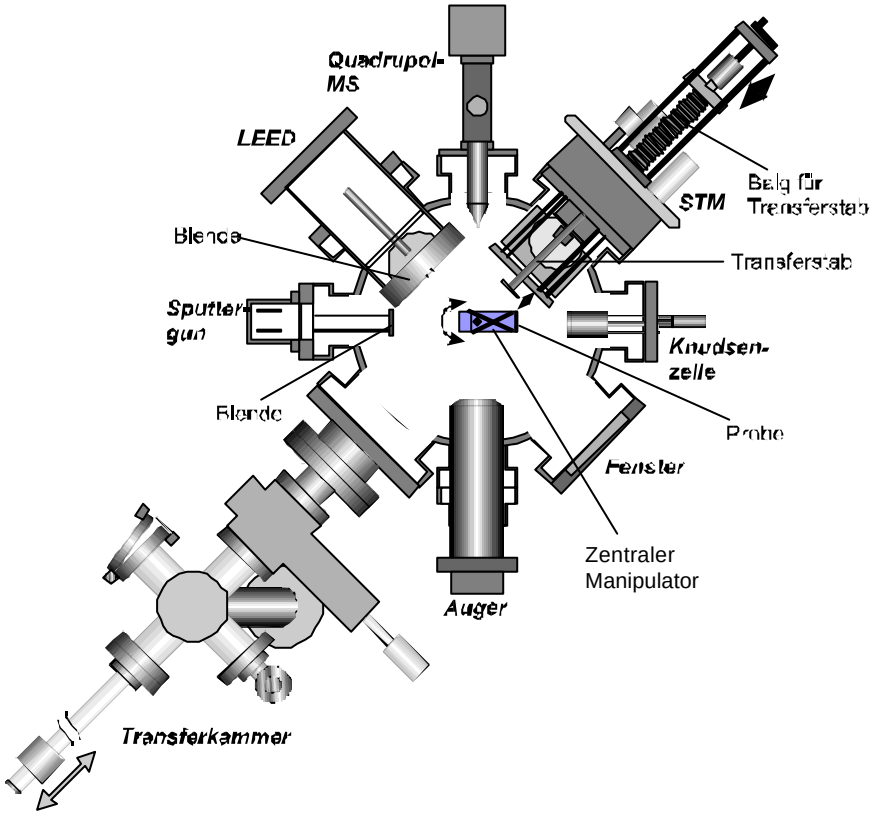
Why UHV ($p < 10^{-9}$ mbar)

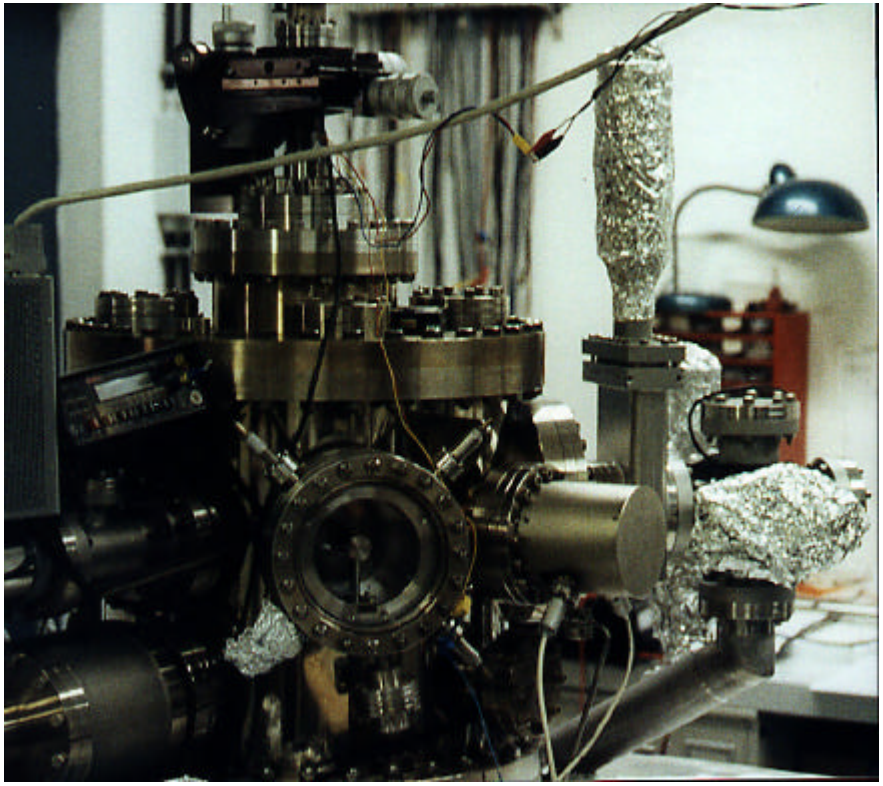
- Absolutely clean surfaces without H_2O, O_2 or other impurities
- Residual-gas-monolayer formation time more than 1 hour
- LEED, MBE, mass spectroscopy
- Sound insolation
- Low temperature experiments

Experimental effort

- Much more than in high vacuum (e.g. REM)
- Setup time several days
- Very few materials allowed, mainly stainless steel, Mo, W, Cu, Ceramics, glass, Teflon, viton
- No lubrication
- Only metal gaskets and valves
- Baking minimum 150°C for several hours
- Motion feedthroughs only with bellows
- Specialized workshop
- Direct motion usually with piezos and stick slip drives

UHV technique 2

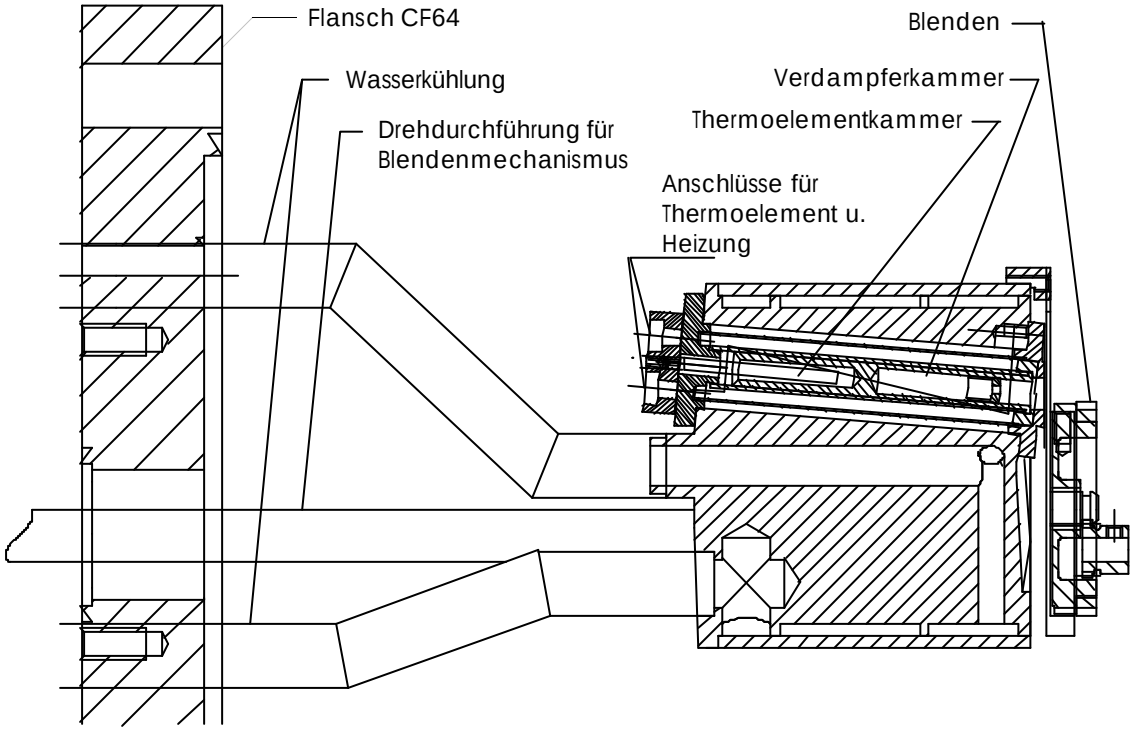




Preparation

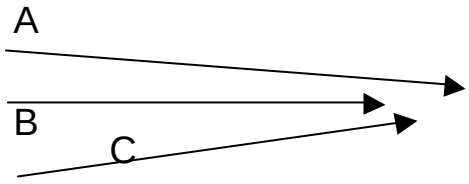
Substrate:

- Mechanical: splitting, grinding, polishing, ...
- Sputter / annealing cycles
- Test by LEED, Auger, STM
- Goal: atomically regular, flat and clean surface



Adsorbate:

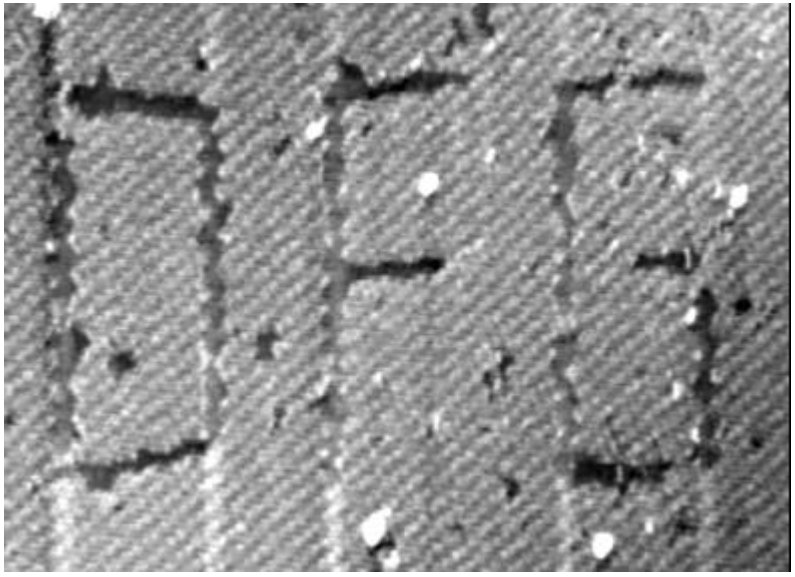
- Air: adsorption
- UHV: OMBE (Organic Molecular Beam Epitaxy)
- Cleaning by moderate heating
- With a specifically developed effusion cell an abitrary sequence or mixture of 3 different adsorbates is possible.
  ➧ co-adsorption experiments



Triple effusion cell;
Temperature range: 10°C up to 1000°C

STM (Scanning Tunneling Microscope) 1

- Imaging and manipulation with the same instrument
- Manipulation of moleculs with small forces
- Organic adsorbates need a very stable and sensitive instrument (✹UHV).
- Nanomanipulation also possible in air and at room temperature
- Typical Parameters:
  - system: PTCDA on HOPG at ambient conditions
  - current: 10pA - 1nA, voltage: -1V to +1V
  - seconds / picture: ~10
  - pixels: 512 x 512
  - tunneling distance for imaging: ~5Å
  - reduction of gap-resistance for manipulation from 10^{10}Φ to 10^{9}Φ (Bias :1V to 0.1V)
  - Time for the whole experiment: several nights



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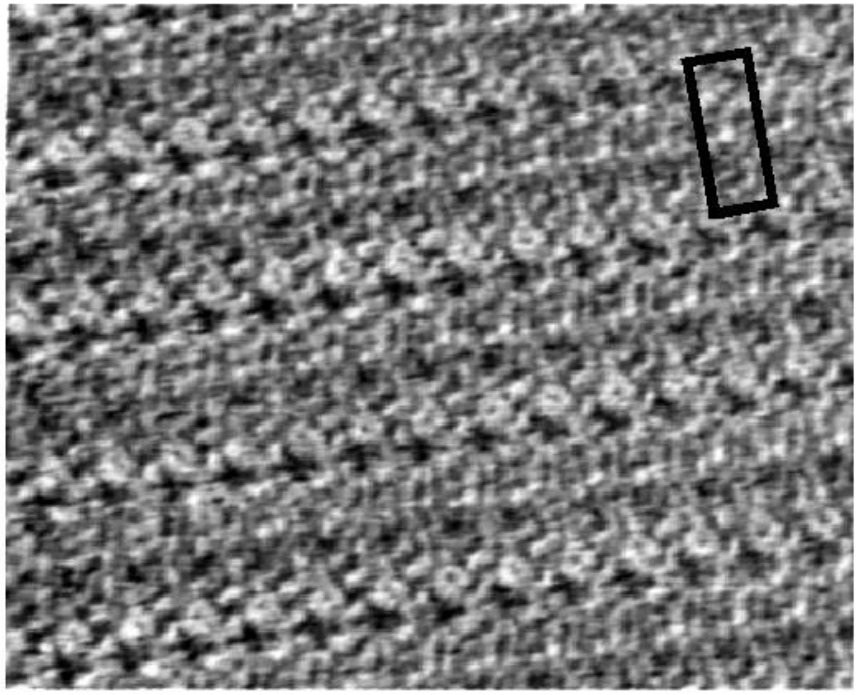
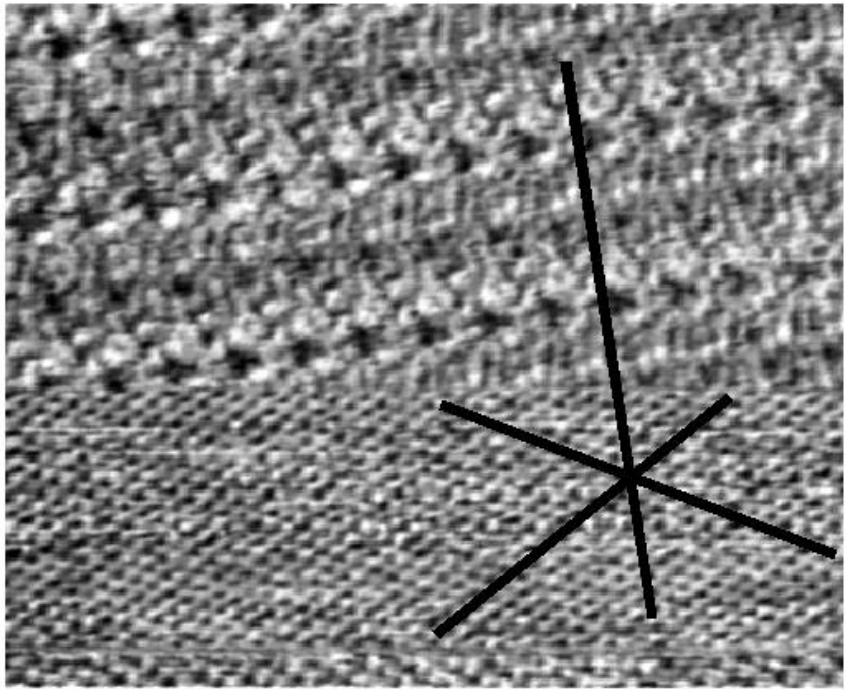
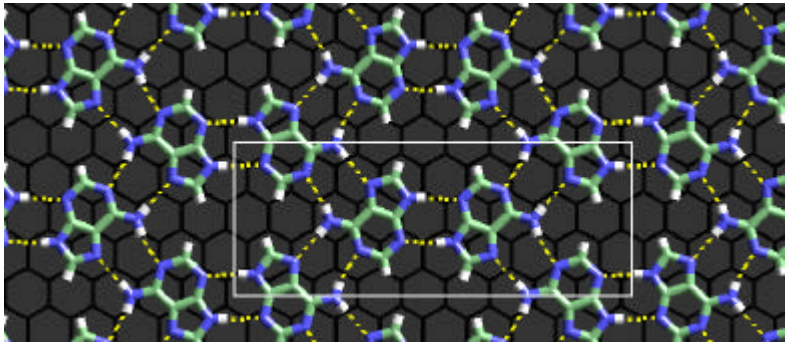
- With STM one obtains information about the local structure in real space, incl. defects!
- Although the (well known) principle of a STM is simple and one can see C-atoms with a cheap home-built STM, learning to use it for research usually takes up to several months and going to UHV is a full time job.

STM 2



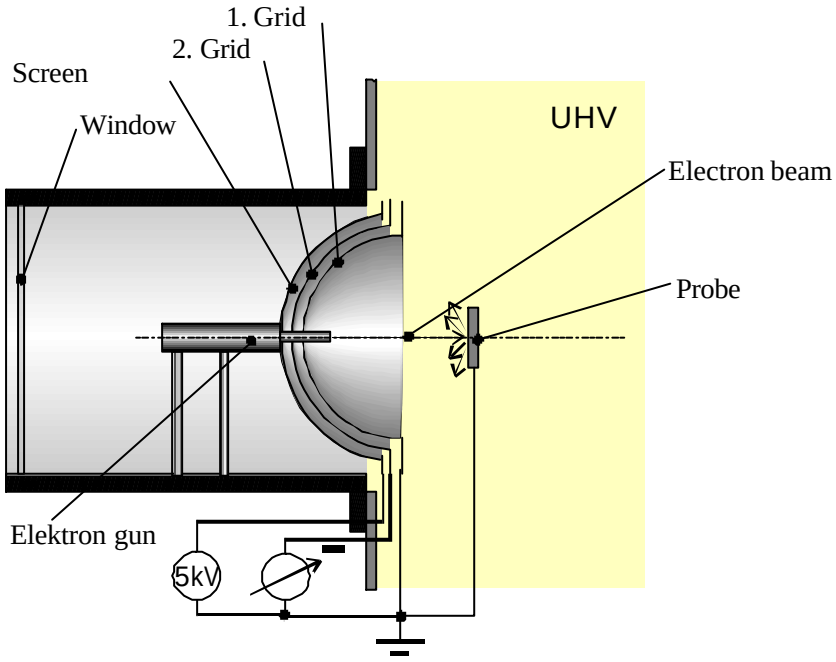




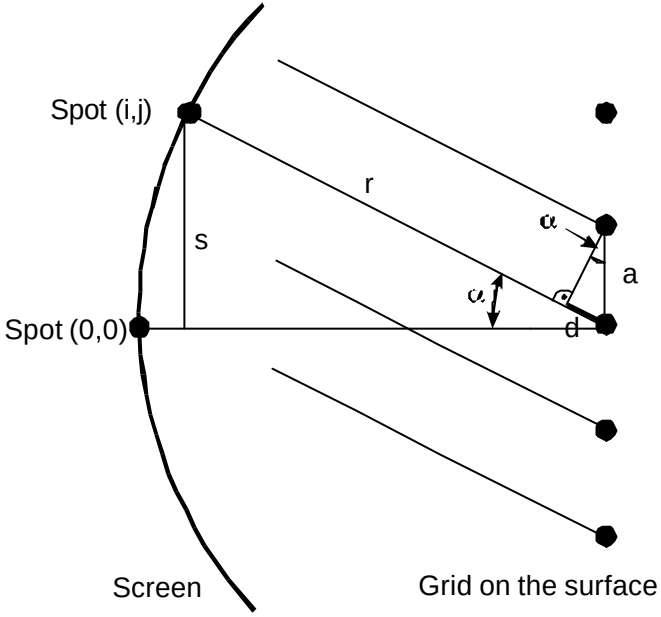
Adenine on graphite, 110Å x 90Å,
unit cell 22.1 Å x 8.5Å, ♋=90°,
♑=0°

LEED (Low Energy Electron Diffraction) 1

Schematic:



Geometric evaluation:

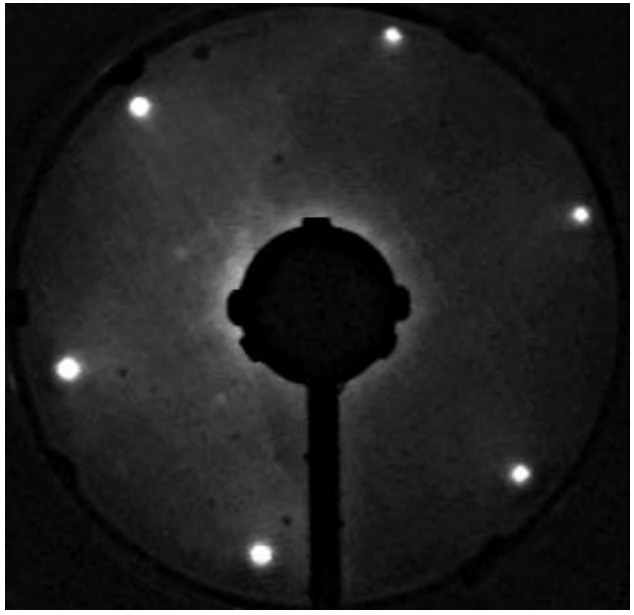


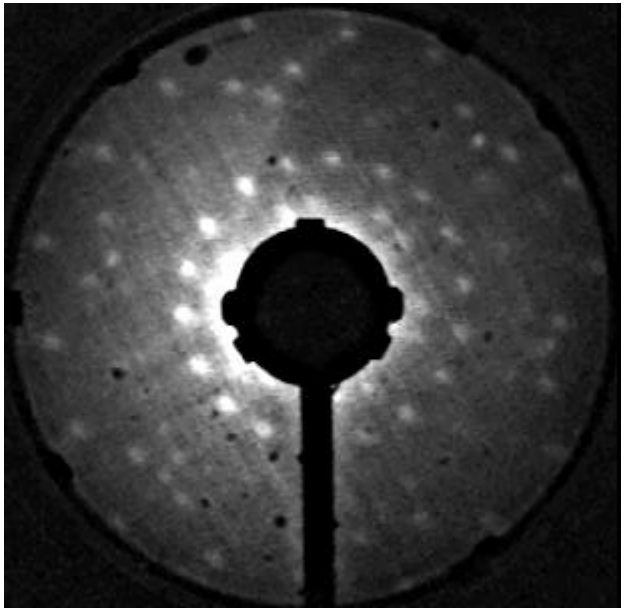
Bragg: $n \lambda = a \sin(\vartheta) = a\,(s / r)$

- Information about periodic structures (unit cell parameters): symmetry group, extension, direction.
- Reciprocal space, superposition of all domains of a large area, ~1mm^2

LEED 2

Adenin on graphite; 56,6 eV and 28.3 eV

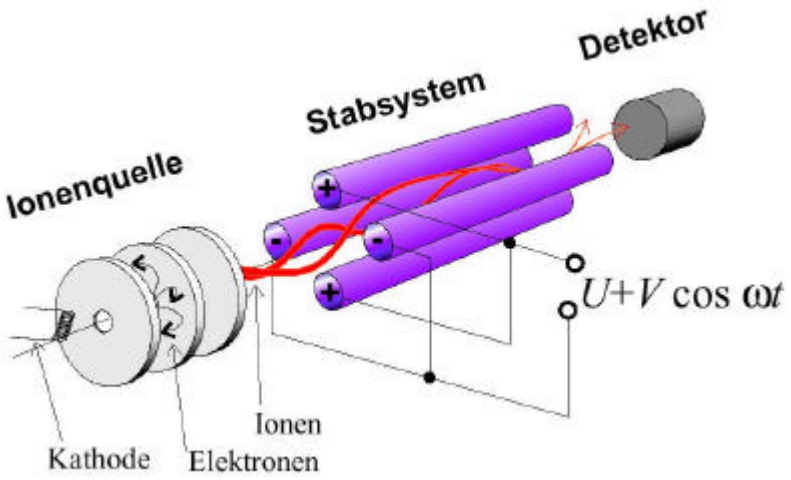




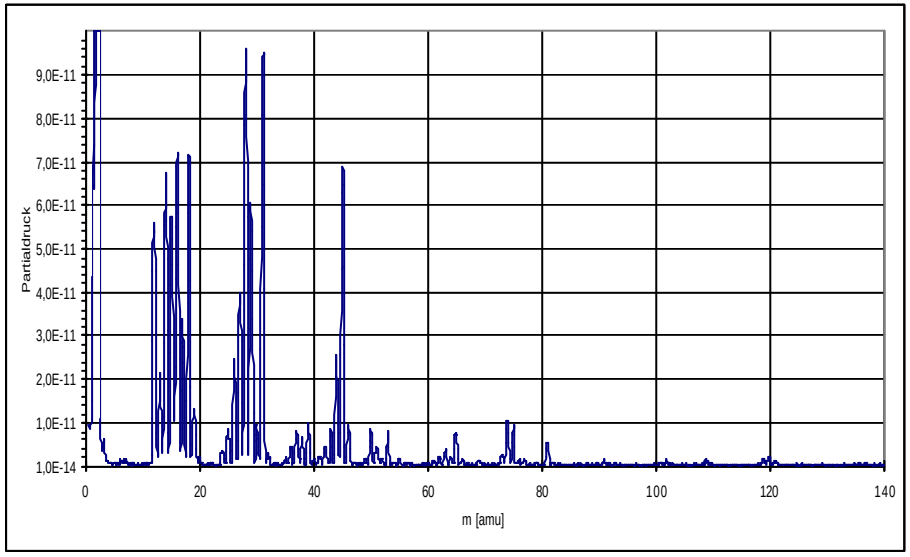
Unit cell: 22.1Å x 8.5Å, 90°

QMS (Quadrupole Mass Spectrometer)

- Partial pressure down to $1x10^{-14}$ mbar
- Resolution < 0.2 amu
- Typ. scan speed: 0.3 - 5 amu/s
- Fragmentation pattern by ionisation
- Absolute calibration difficult, but possible
- Standard equipment in UHV



Typ. mass spectrum of an unbaked UHV-Chamber



Two operating modi:

1.) Scan: partial pressure vs amu

- Residual gas analysis (RGA)
- Control of pumps

2.) Single Peak: partial pr. vs time

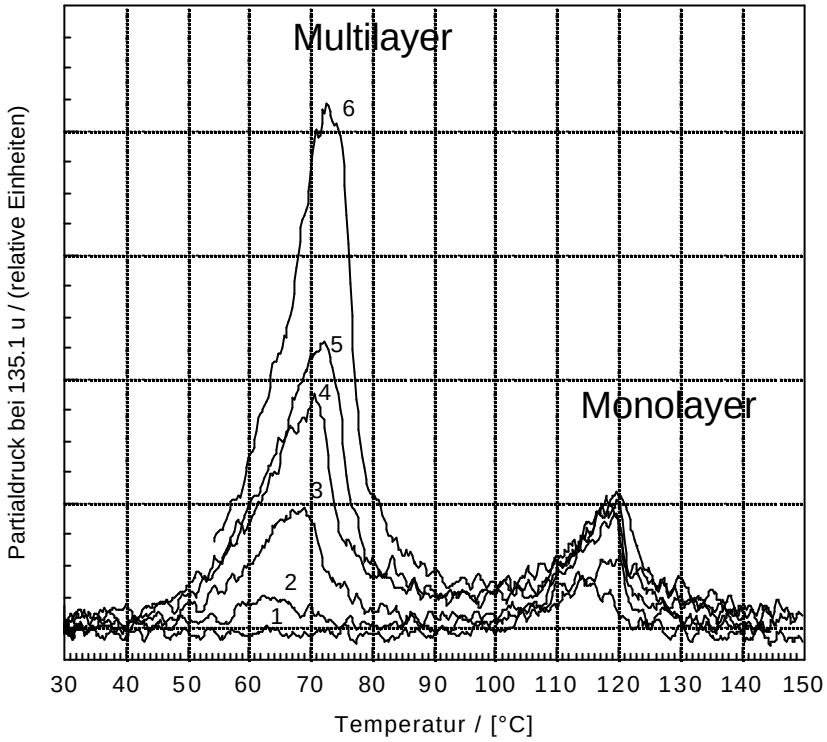
- Leak Test (He)
- TDS

TDS (Thermal Desorption Spectroscopy) 1

Goal: Activation energy of desorption at different coverages

Experimental steps:

- Cleaning of the substrate
- Preparing the adsobate layer(s) with MBE
- Adjusting QMS on main peak of the adsorbate spectrum
- Heating with linear increase of temperature (e.g. 1°C/s) during recording partial pressure and temperature versus time
- The position, size and shape of the different peaks contains the Information about the binding energy



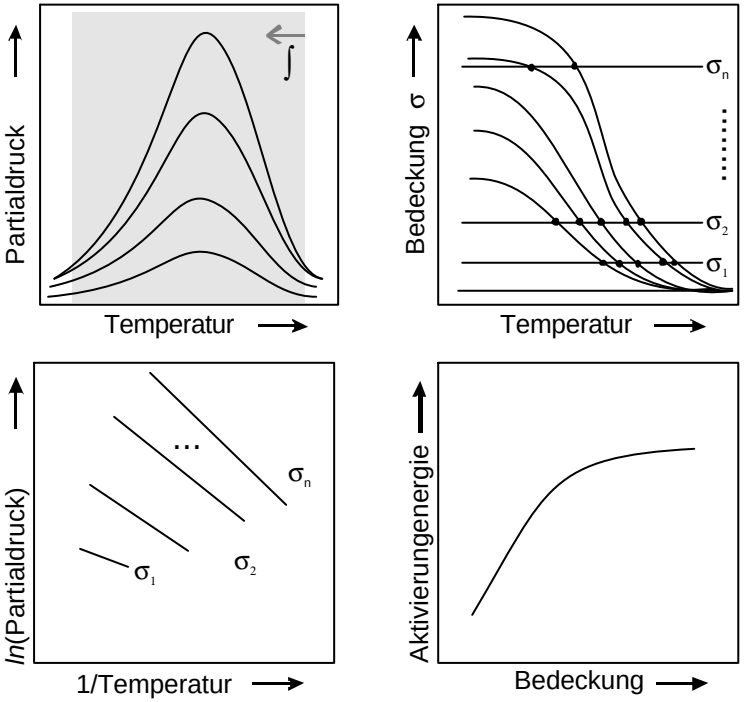
Adenin on Ag(111); 20, 40, 60, 80, 100 and 180 s MBE-time (#1-6)

TDS 2

Evaluation of the activation energy:

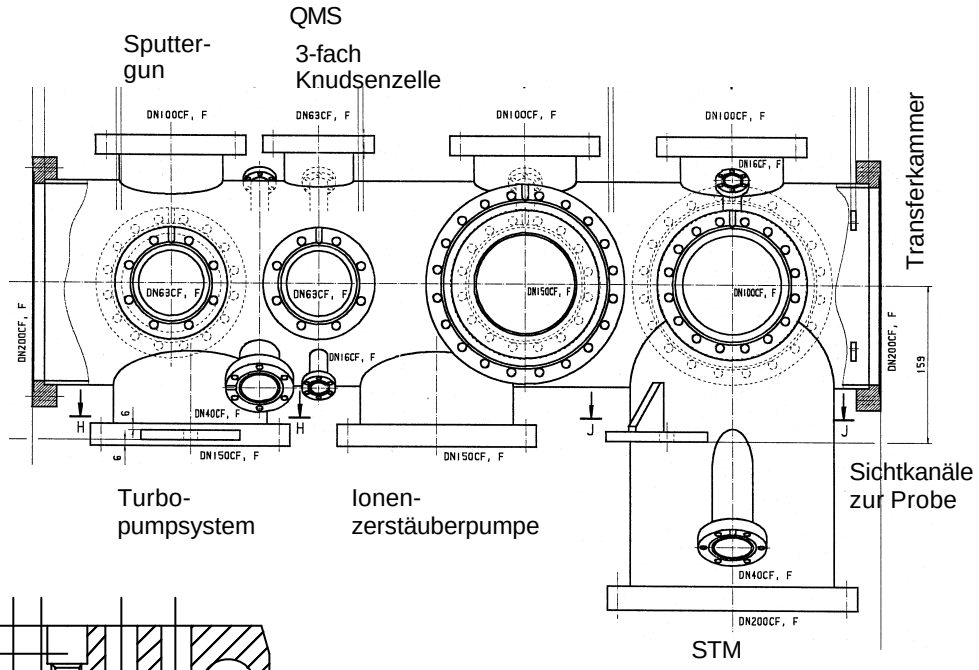
- Peak-maximum-method (Readhead): simple, but the energy (E) assumed to be coverage independent

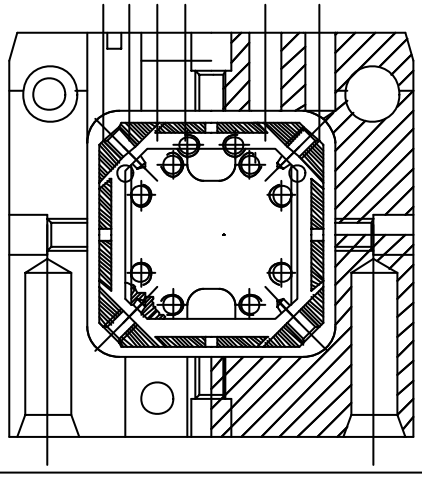
  Polany-Wigner expression leads to an implicite expression for the energy and maximum temperature
- Complete Analysis: needs a lot of measurements, shows the coverage dependence of E



Concept of new UHV chamber

- Compact
- Linear arrangement of experiments
- Positioning of sample only with only one reliable linear feedthrough
- Triple effusion cell
- STM, LEED, TDS, QMS, sputter-gun
- Transfer-chamber
- Fast experiment cycle time





Sample stage:
- temperature drift compensated
- mobile
- 8 electrical contacts
- resistive heating
- sample size max 6 x 6 x 4 mm^3
- heat radiation shield

Conclusion

What we can do:

- Standard UHV-technique
- Mass spectrometry
- Thermal desorption spectroscopy
- LEED
- Auger
- STM (AFM), additional feature: nanomanipulation at room temperature
- Image processing for scanning probe microscopy
- Numerical simulations of organic adsorbates (force field calculations)

Near future:

Variable temperature (LN_2 to 500°C) UHV-STM, tunneling spectroscopy, nanomanipulation