THE TRANSMISSION ELECTRON MICROSCOPE:

ITS EARLY DEVELOPMENT AND RECENT ACHIEVEMENTS

Stan Konings

First electron microscope

Ernst Ruska and Max Knoll (1928-1931)

First commercial electron microscope (Siemens 1939)

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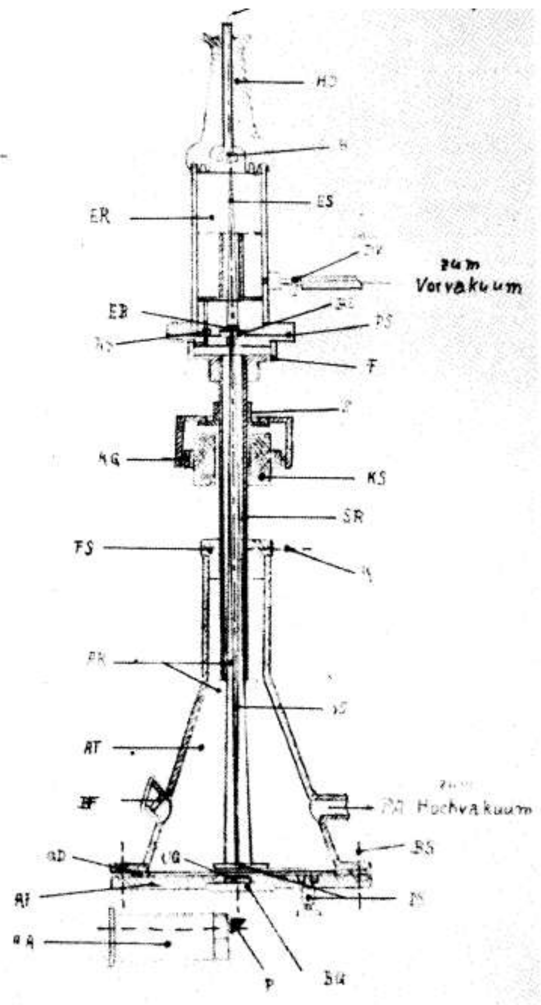
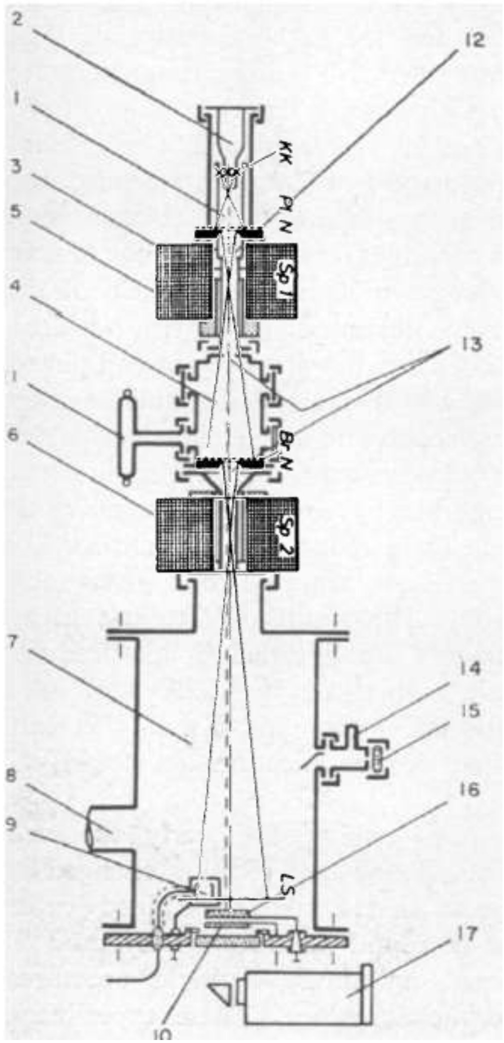
1-stage “microscope”

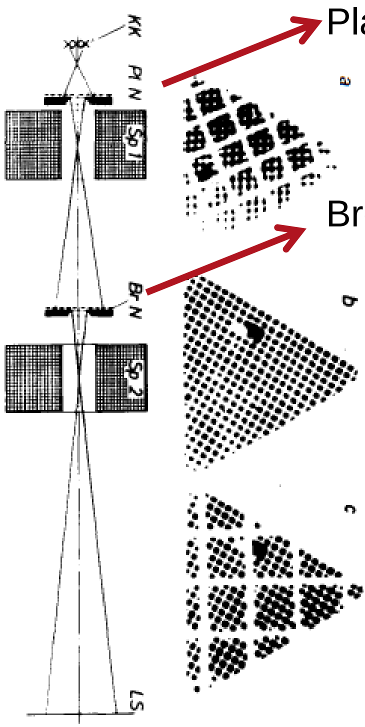
2-stage microscope



1929



1931



Platina grid

13 x Platina grid

Bronze grid

4.8 x Bronze grid

17 x Platina grid
4.8 x Bronze grid

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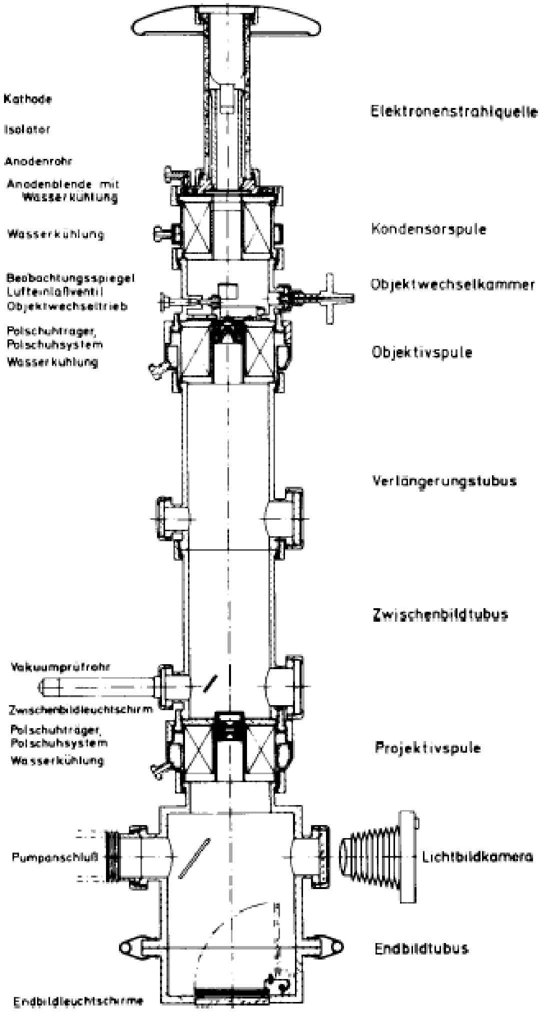
1933: Das übermikroskop

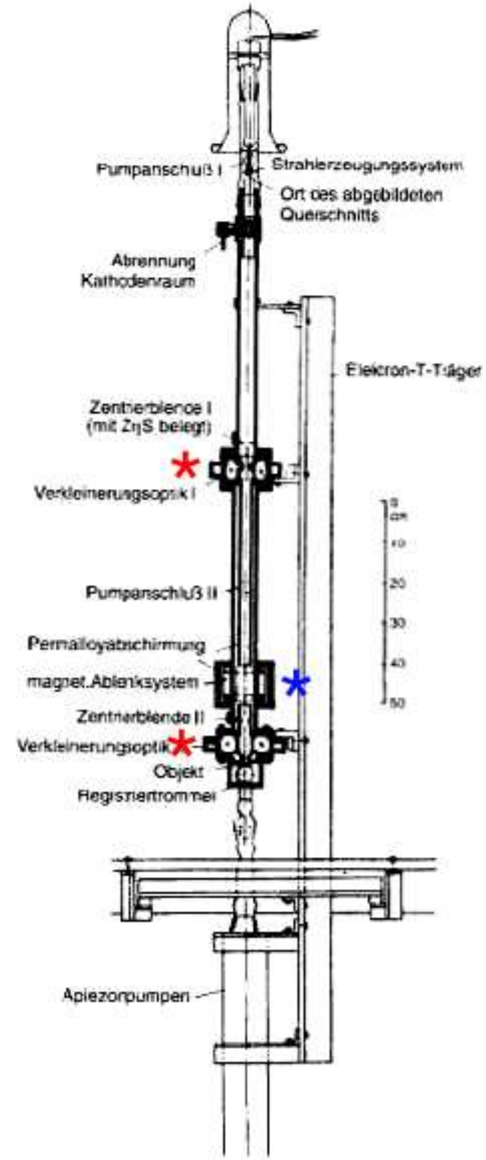


Magnification: up to 12000x

Driest and Müller: Z. Wiss. Mikroskopie 52, 53-57 (1935)

Krause: Naturwissenschaften 25, 817-825 (1937)

Scanning TEM

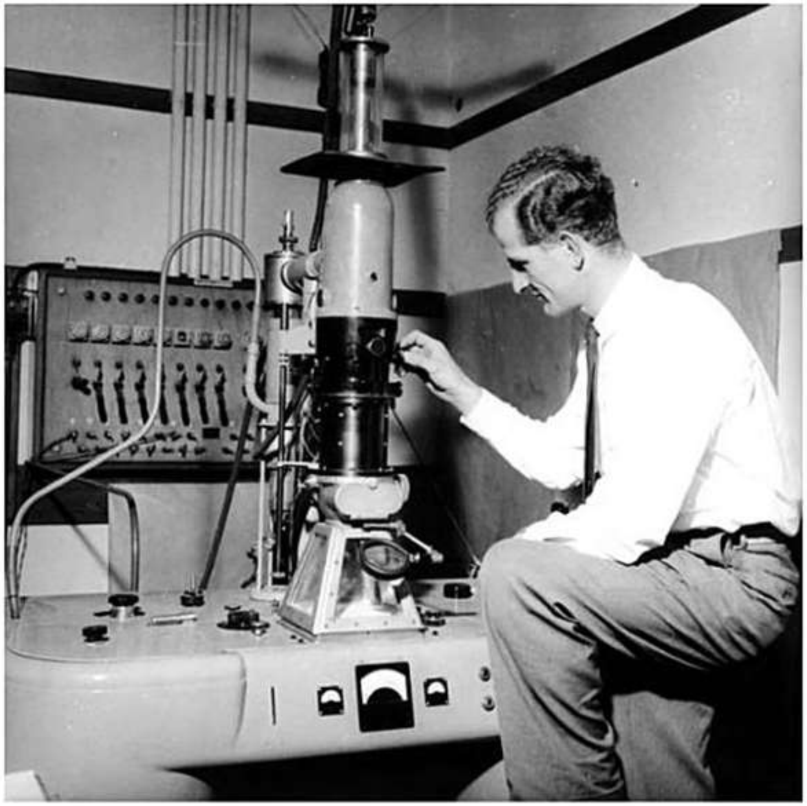


1938: The *scanning* transmission electron microscope invented by Manfred von Ardenne

Early adopter in the Netherlands: Willy Burgers

- First to build a “low magnification” electron microscope in the Netherlands : 1935 (Philips Research lab)
- Alpha-gamma transition in iron at high temperature

Jan le Poole: founding father of electron optics in the Netherlands



1941: build his first electron microscope
1957: prof. of electron optics group (Delft)

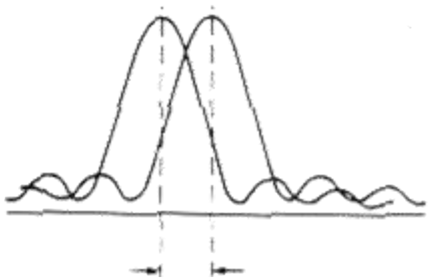
X-ray projection microscope
SEM with quadrupole lenses
Microprobe x-ray analyzer
Compact 1 MV microscope

Stigmators
Intermediate lens
Twin lens configuration
TEM / STEM switch

The quest for better resolution

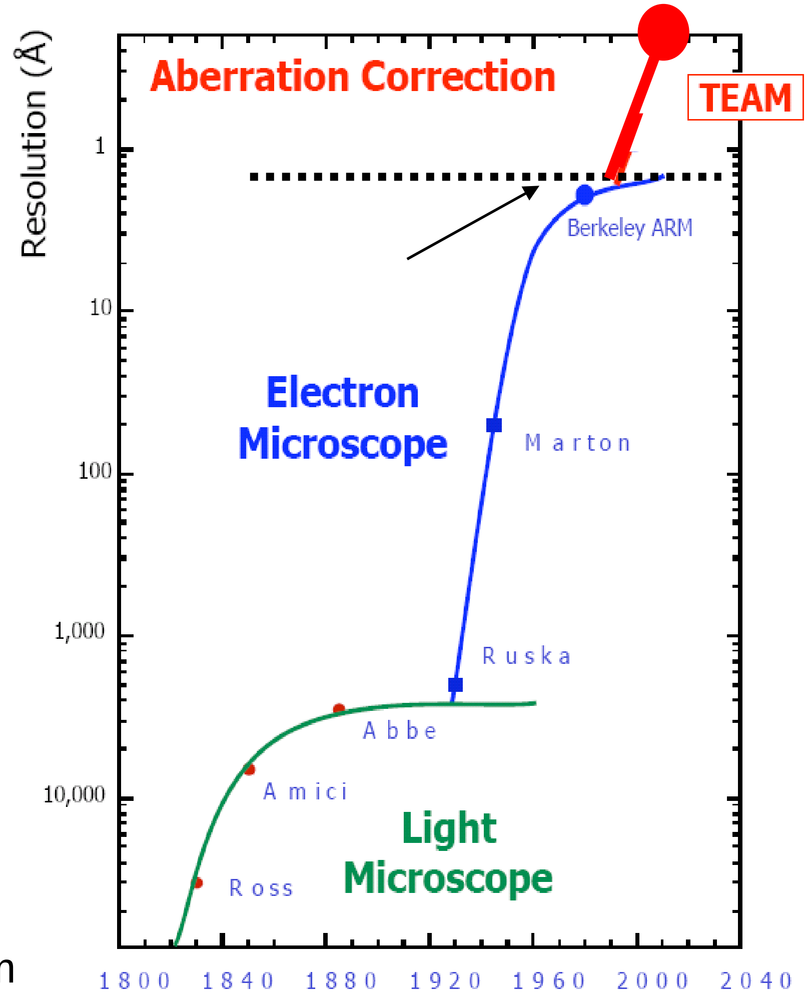
Why use electrons?

$$R = 0.612\,\lambda / n \sin \alpha$$



$$\lambda = \frac{h}{\sqrt{2m_0 eU}} \frac{1}{\sqrt{1 + \frac{eU}{2m_0 c^2}}}$$

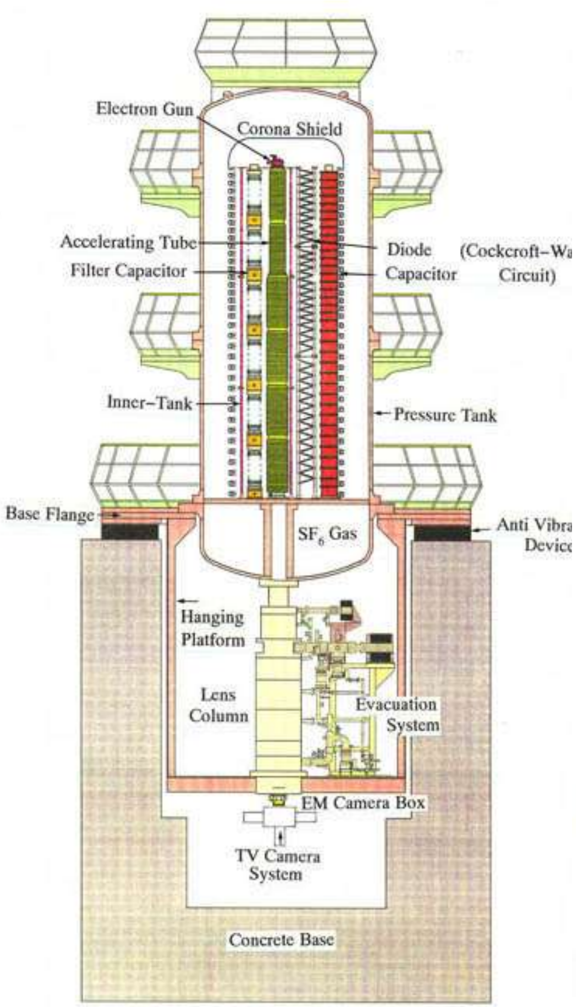
Accelerating voltage

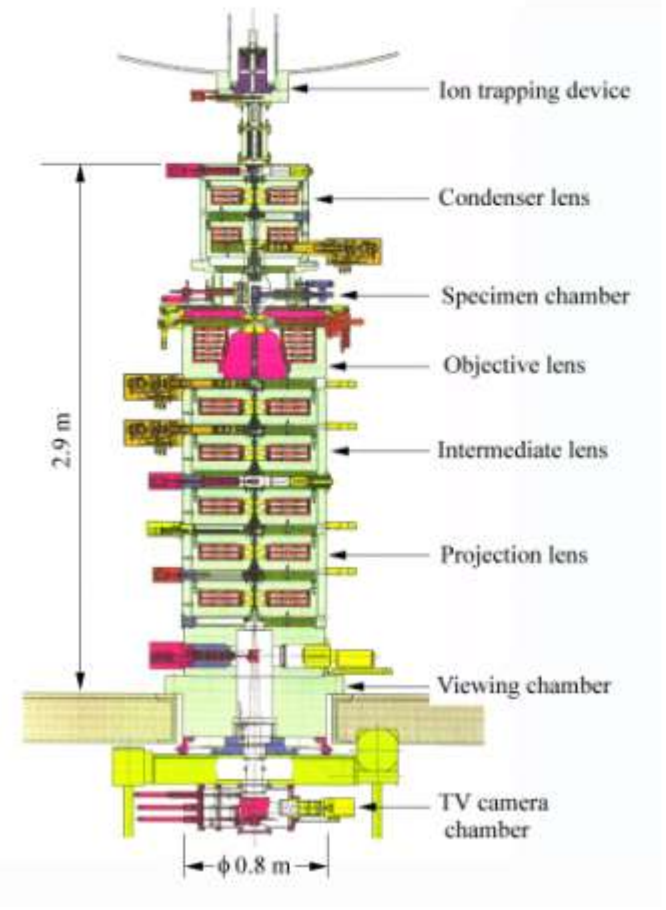


At 75 kV and an opening angle of 20 mrad: R = 264 pm

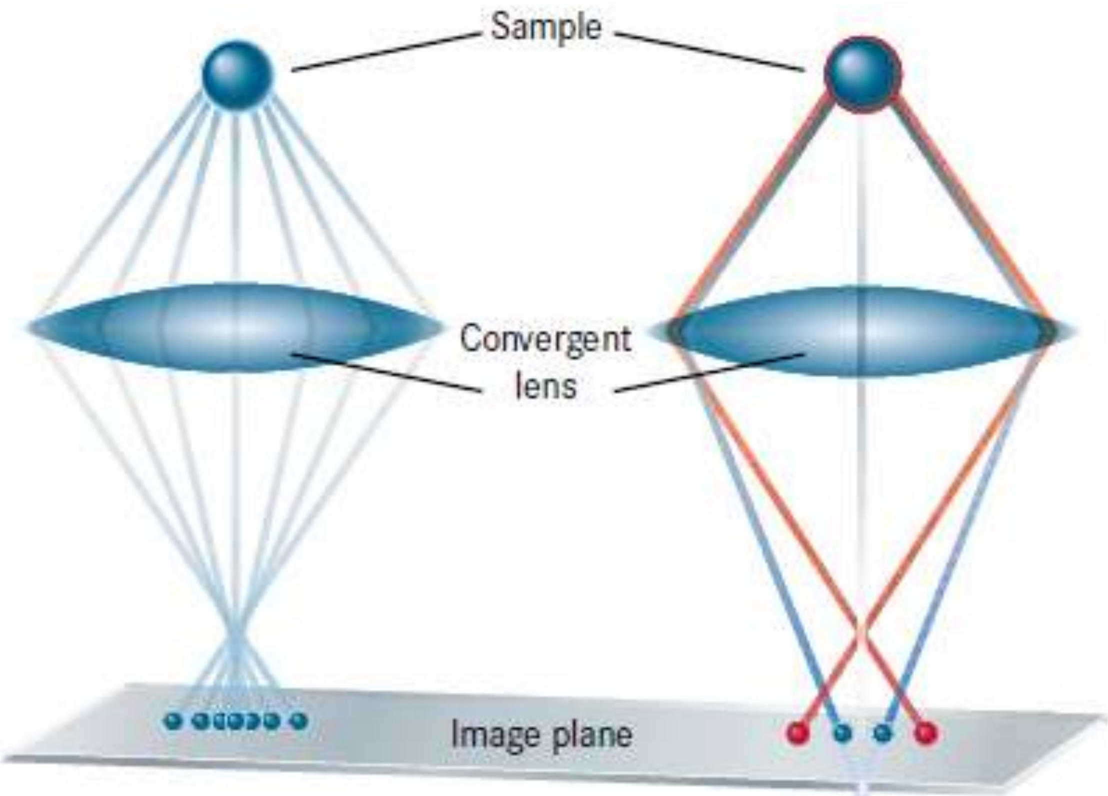
At 3000 kV and an opening angle of 20 mrad: R = 20 pm

3 MV transmission electron microscope Osaka





Lens aberations limit spatial resolution



Spherical aberation *Cs*   Chromatic aberation *Cc*

Picture from: JARA

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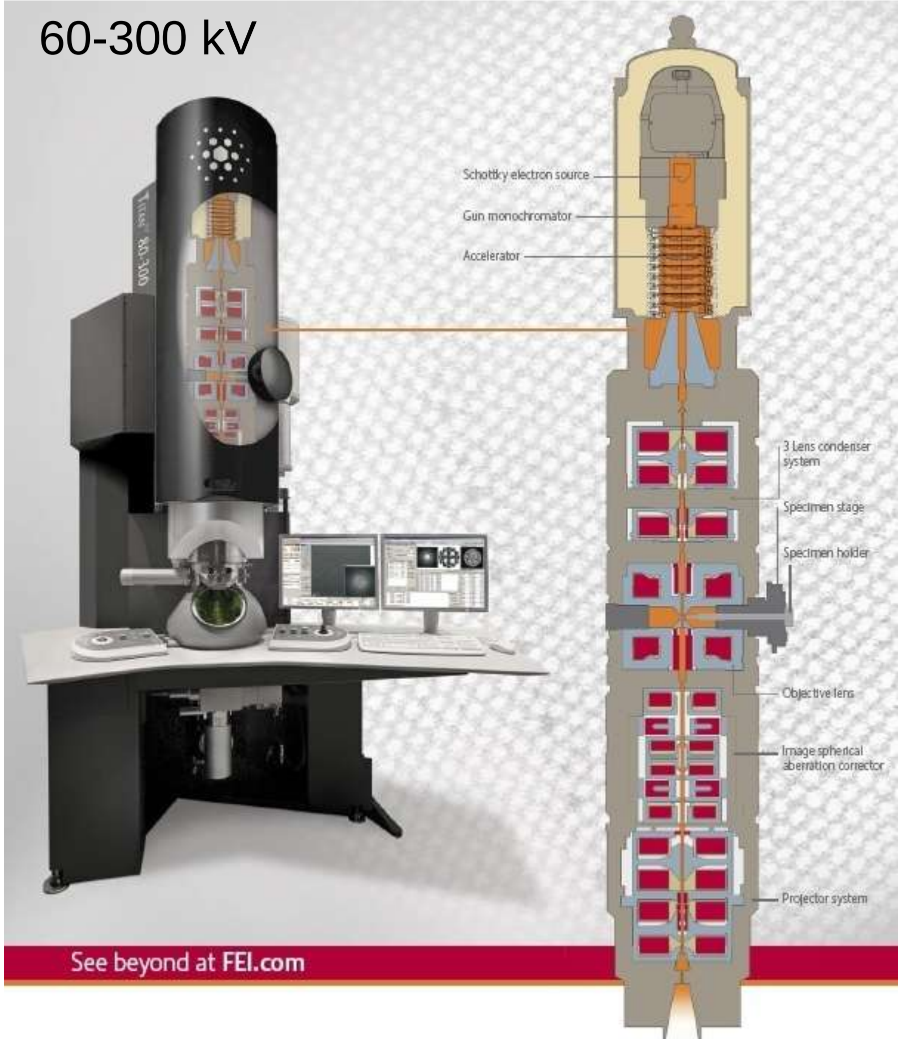
Aberration correction for TEM and STEM



Hexapole lenses for spherical aberration correction

Dodecapole lens for chromatic aberration correction

CEOS
Corrected Electron Optical Systems GmbH

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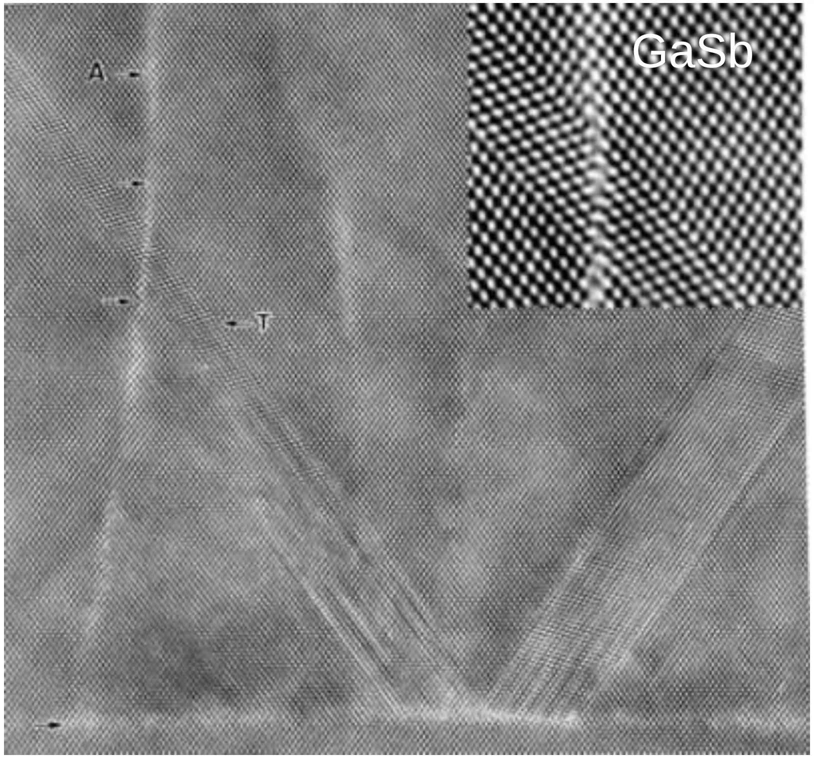
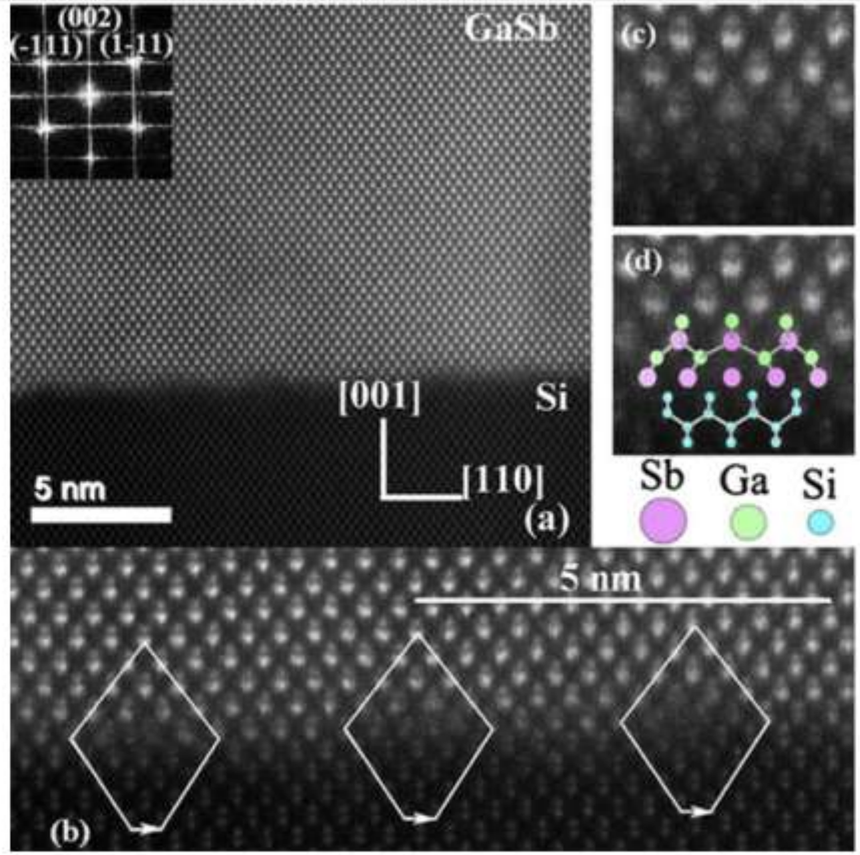
TEM

STEM

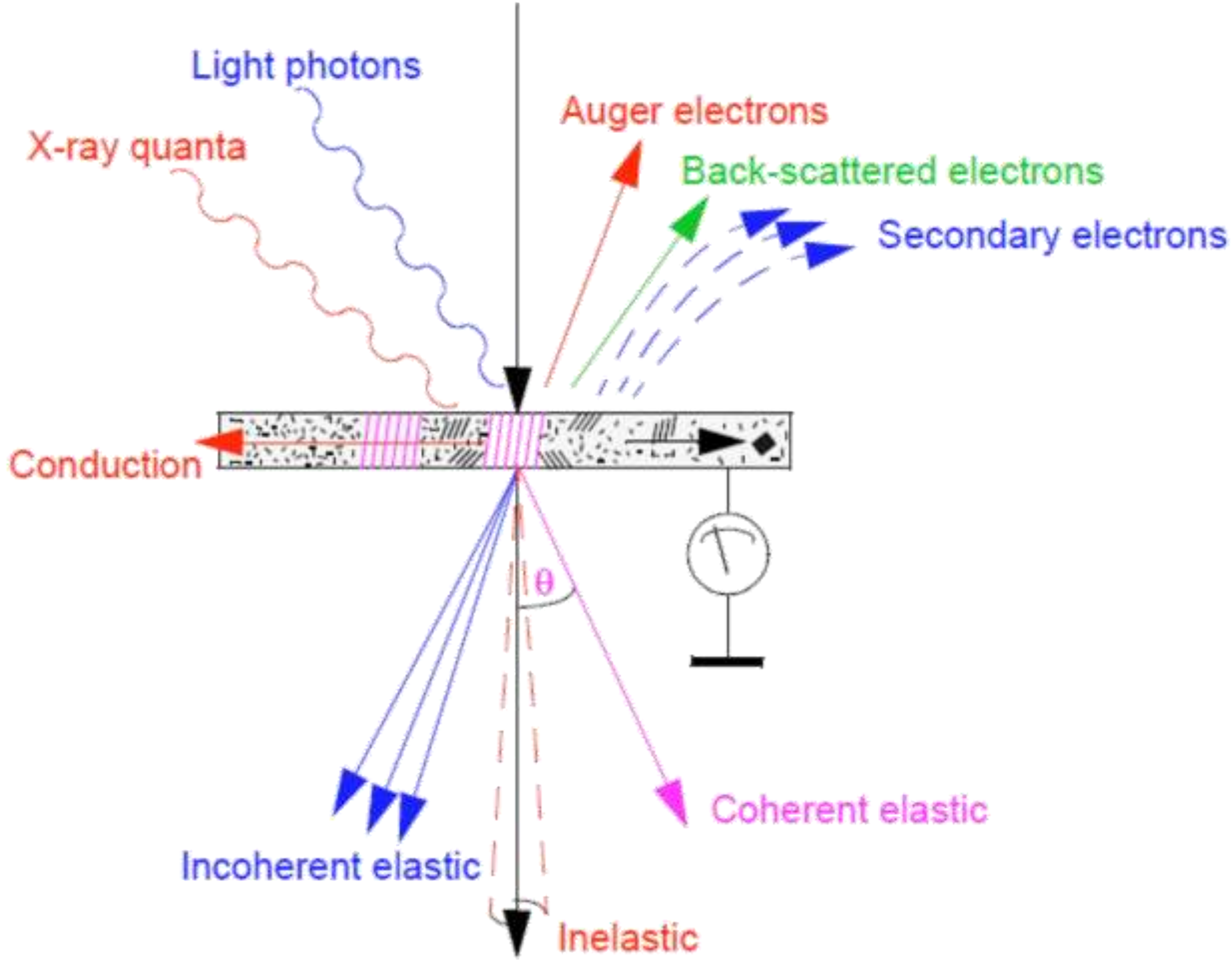




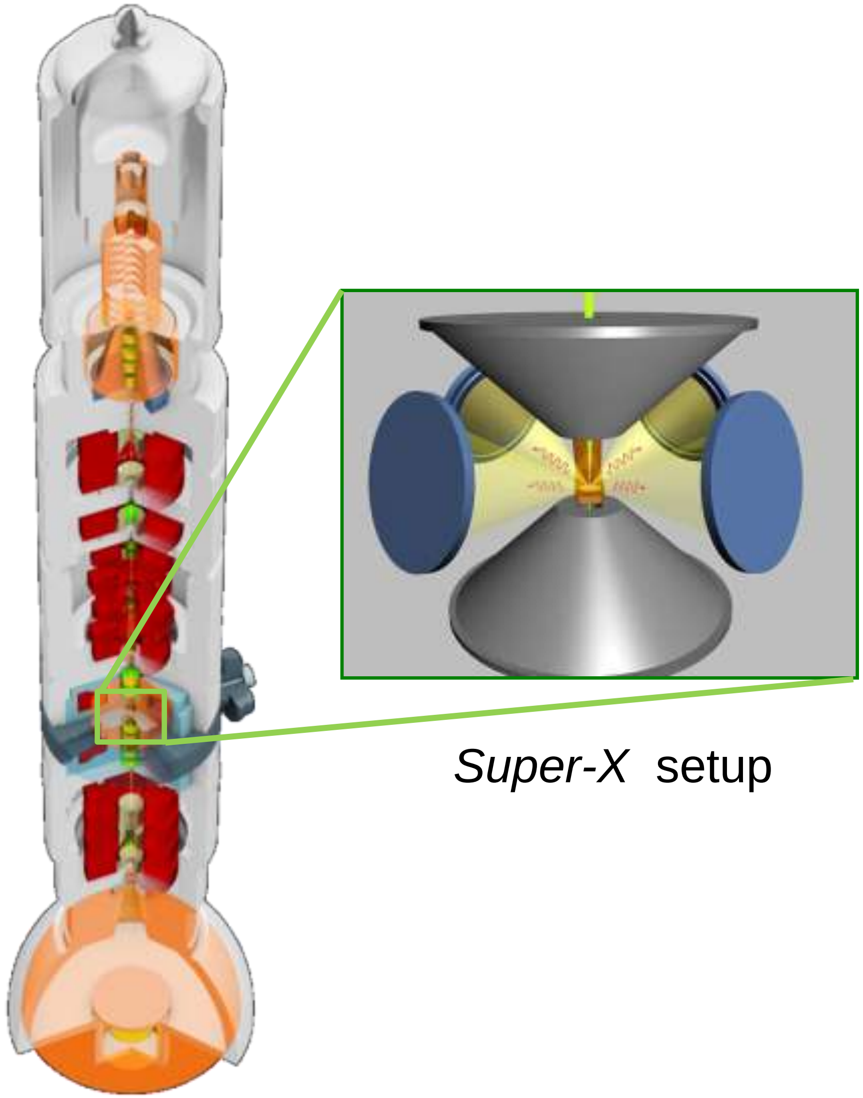
Pic: Canadian Centre for Electron Microscopy

Aberation-corrected TEM and STEM show the atomic arrangement at high quality

Grab all signals!



Energy-dispersive X-ray spectroscopy (EDX)



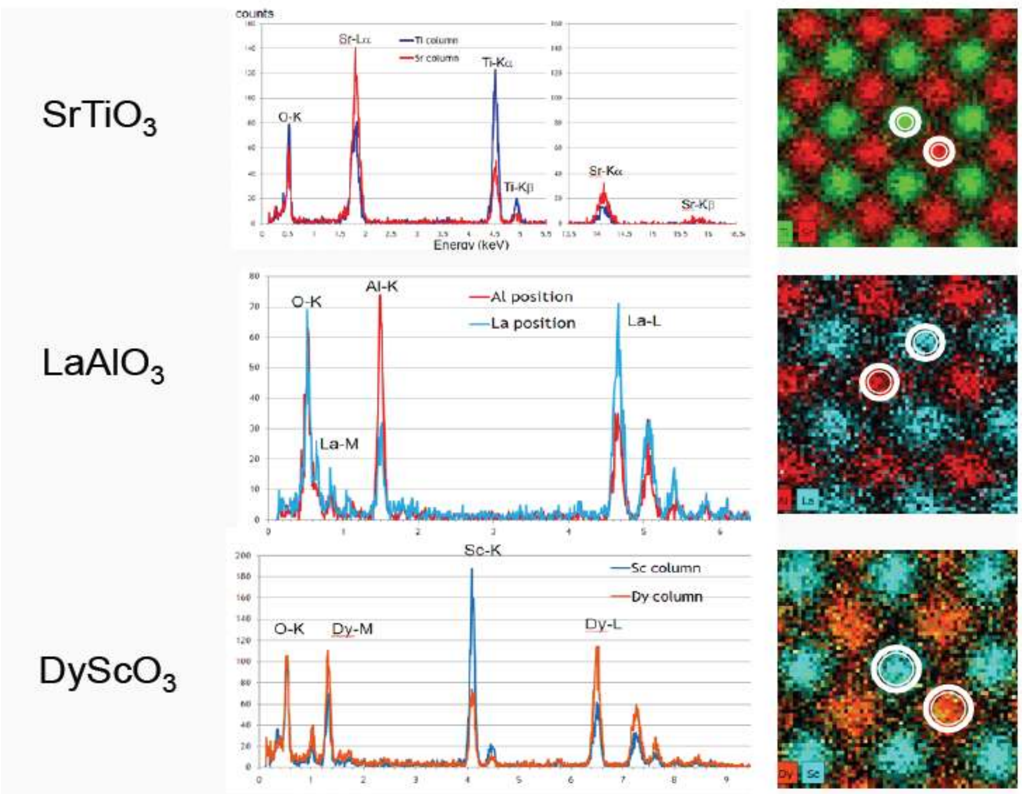
Super-X setup

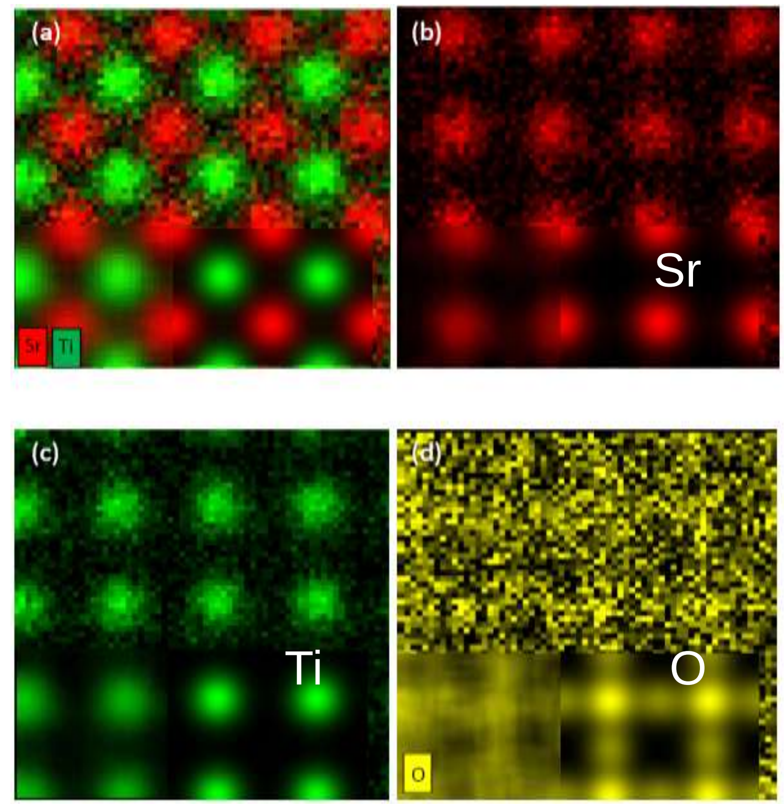
Most important components for mapping at *atomic level*

- Silicon Drift Detector
- High brightness gun (X-FEG)
- Probe corrector

(Cs corrector for STEM imaging)

EDX of several perovskite materials

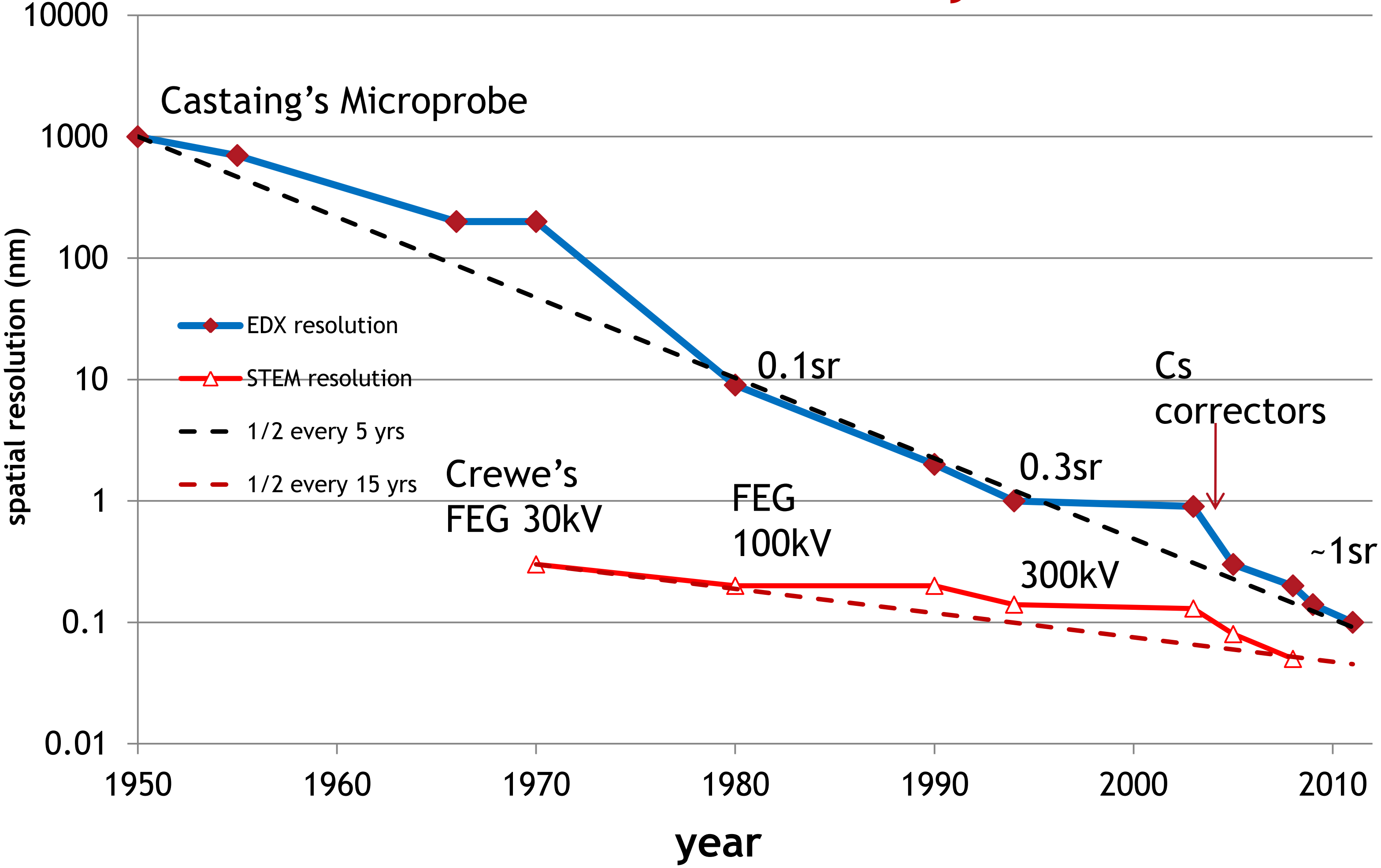




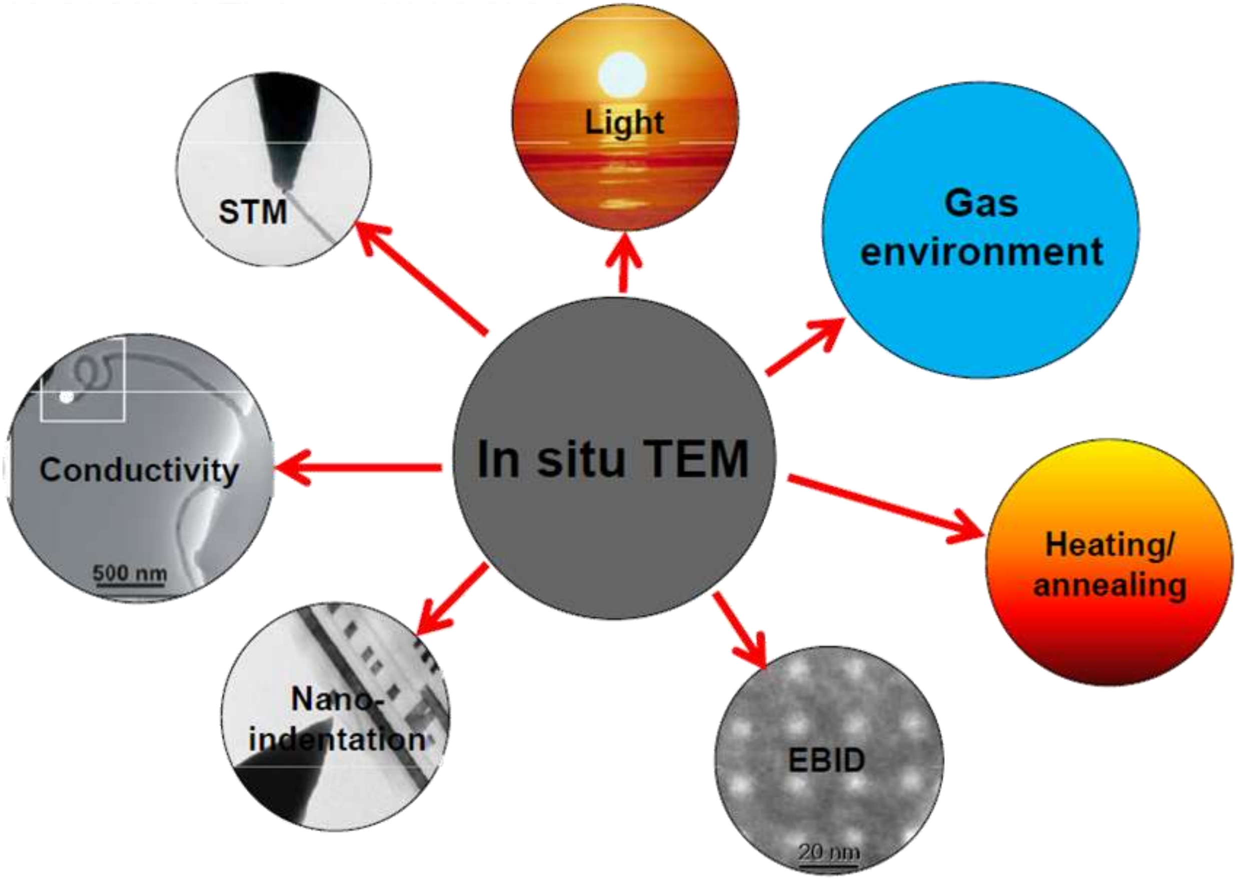
Raw data (top)
averaged map (lower left)
and simulation (lower right)

A.J. D'Alfonso et al. PRB 81 (2010)

Trend in X-ray microanalysis and STEM resolution over the last 60 years

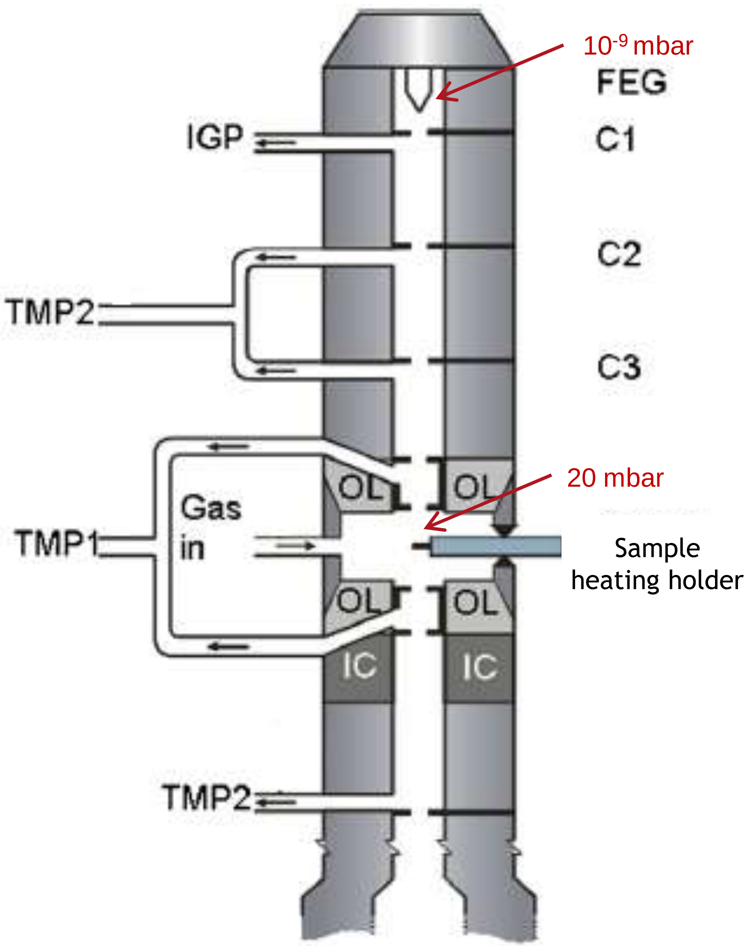


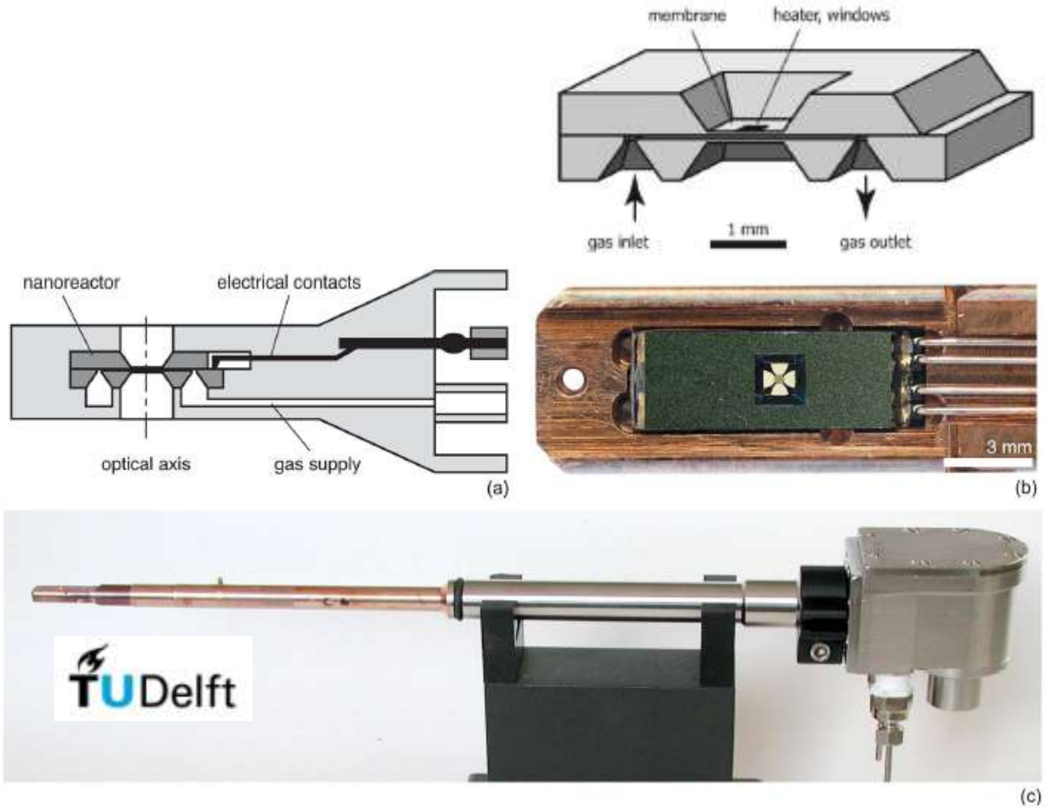
In-situ TEM



STM
Light
Gas environment
Conductivity
500 nm
In situ TEM
Heating/ annealing
Nano-indentation
EBID
20 nm

Environmental TEM: gas + heat





Creemer et al, J MEMS, 2010

Differential pumping system
Gas flow through microscope

Nanoreactor with thin electron tranparent windows
Gas flow integrated on holder

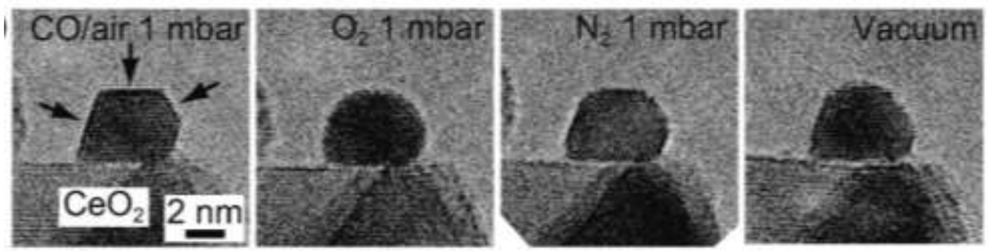
Environmental TEM: tool for catalysis

Catalysts are often in the form of nanoparticles (1-10 nm)

- *Structure of metal species is non uniform*
- *Metal - support interaction*
- *Deactivation due to coalescence*
- *Where are the active sites? the role of defects (vacancies, dislocations), steps and edges*

Typical gases: H_2, O_2, H_2O, CO, CO_2, C_2H_2, CH_4 ......
Typical temperatures: 500-800 ˚C

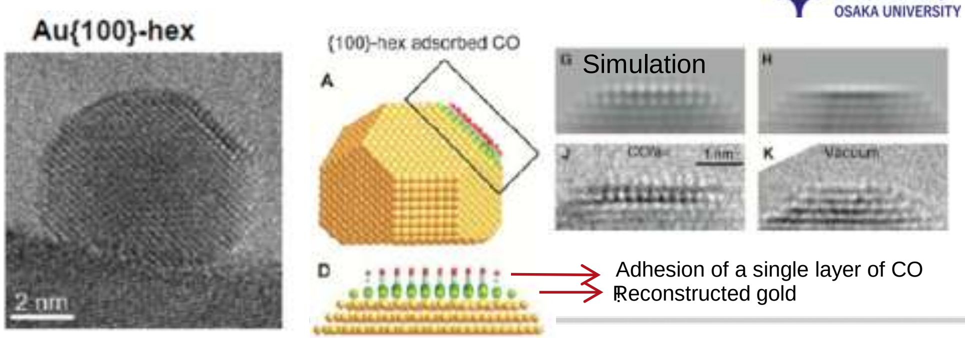
Nanoparticles change shape under the influence of gas



Uchiyama *et al.* Angew. Chem. (2011)

CO oxidation by gold nanoparticles on CeO_2

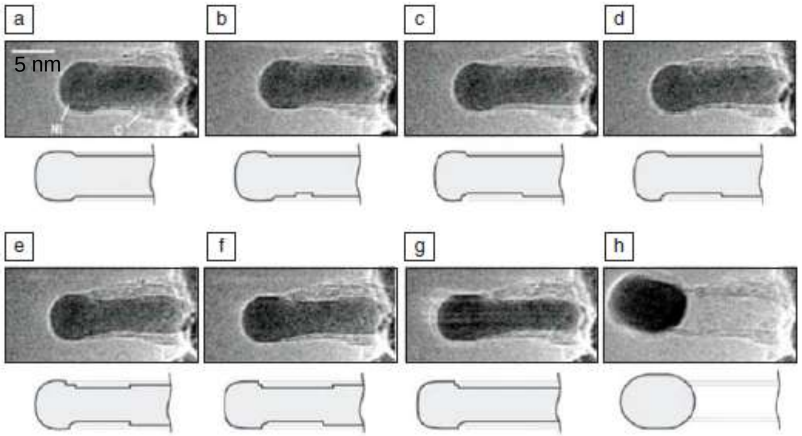
大阪大学 OSAKA UNIVERSITY



Adhesion of a single layer of CO
Reconstructed gold

Yoshida *et al.* Science, 335, 317 (2012)

Growth of carbon nanotubes



Nikkel catalysts with CH_4/H_2 @ 2.1 mbar and heated to 536 ˚C
Time scale 2 frames/sec

Helveg *et al.* Nature, 427, 426 (2004)

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Thank you !