Wir schaffen Wissen – heute für morgen

Paul Scherrer Institut

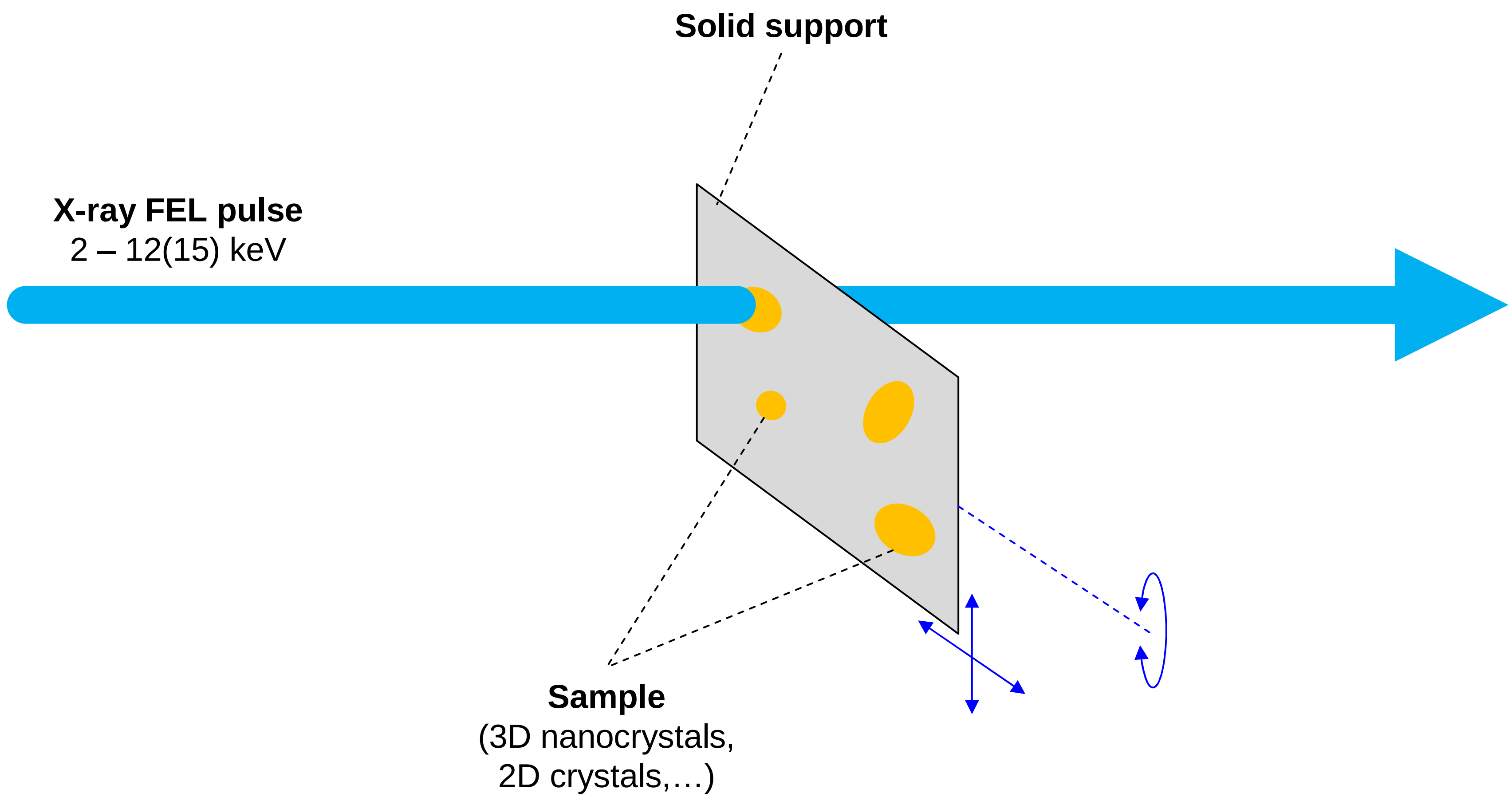
Jörg Raabe

How to Find Samples: Precise Positioning and Complementary Microscopy Techniques

Inhalt

- Challenges
- Sample positioning
- Focusing
- Finding samples

Challenges


Solid support
X-ray FEL pulse
2 – 12(15) keV
Sample
(3D nanocrystals,
2D crystals,…)

Challenges

- Mechanical Stability, 100Hz -> 10ms positioning time
  high resonance frequencies necessary
- Thermal stability:
  1°C temperature change result in 1µm length change
  for 10cm of typical construction material

Existing Instruments

- PolLux: Scanning transmission x-ray microscopy (STXM)
- OMNY: Test setup & Design of UHV/Cryo Version

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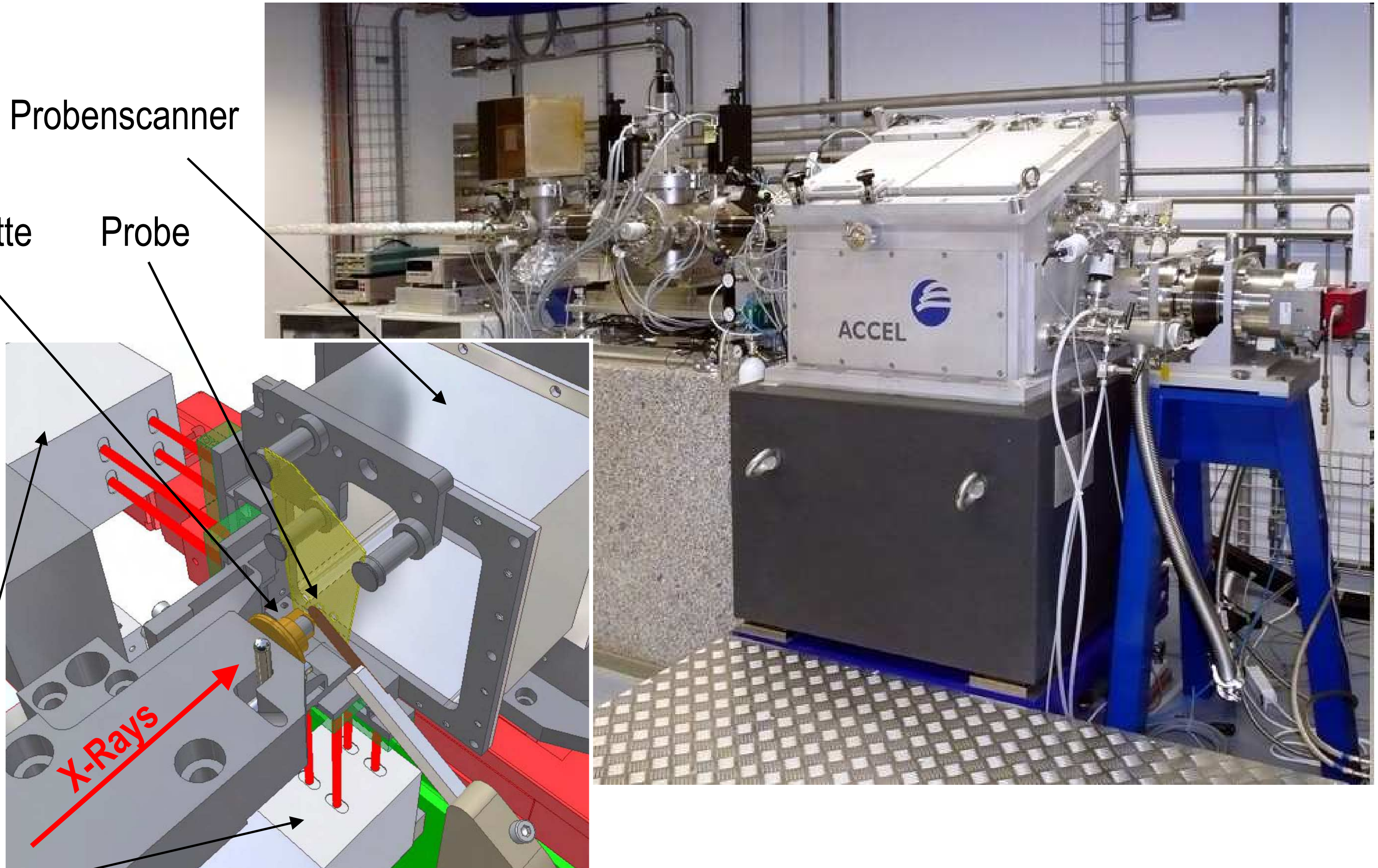
PolLux @ SLS

Endstation

Probenscanner

Zonenplatte

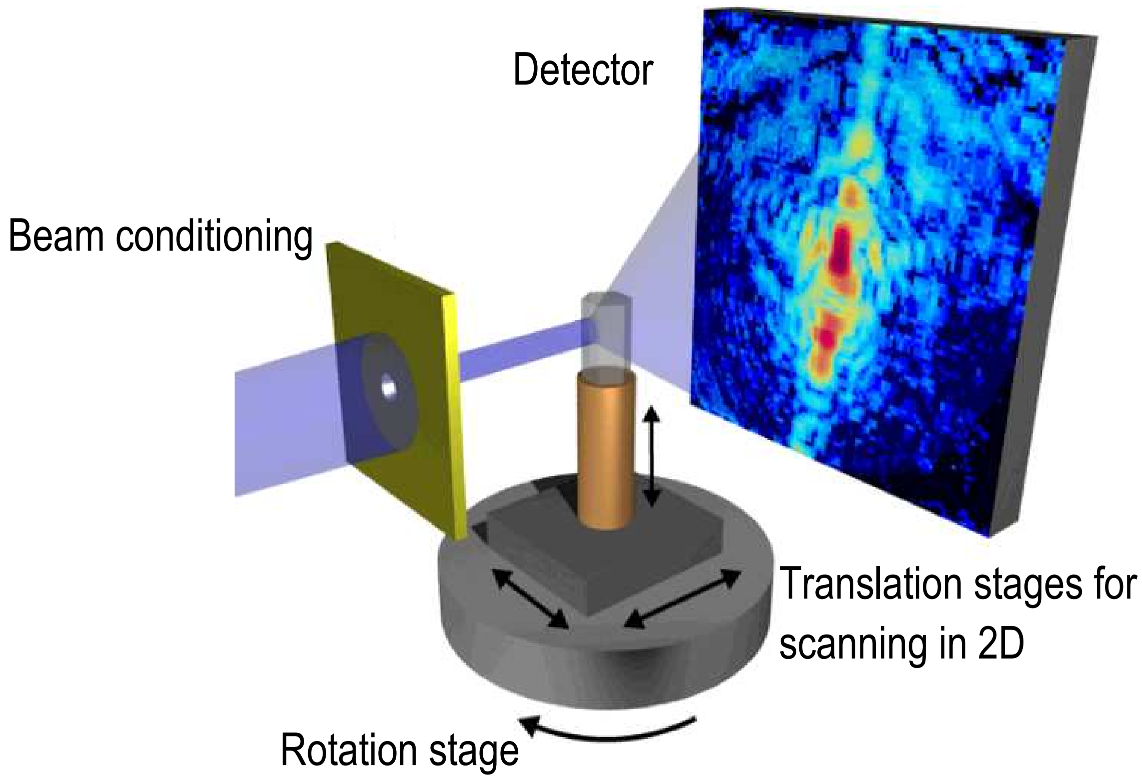
Probe



Interferometer

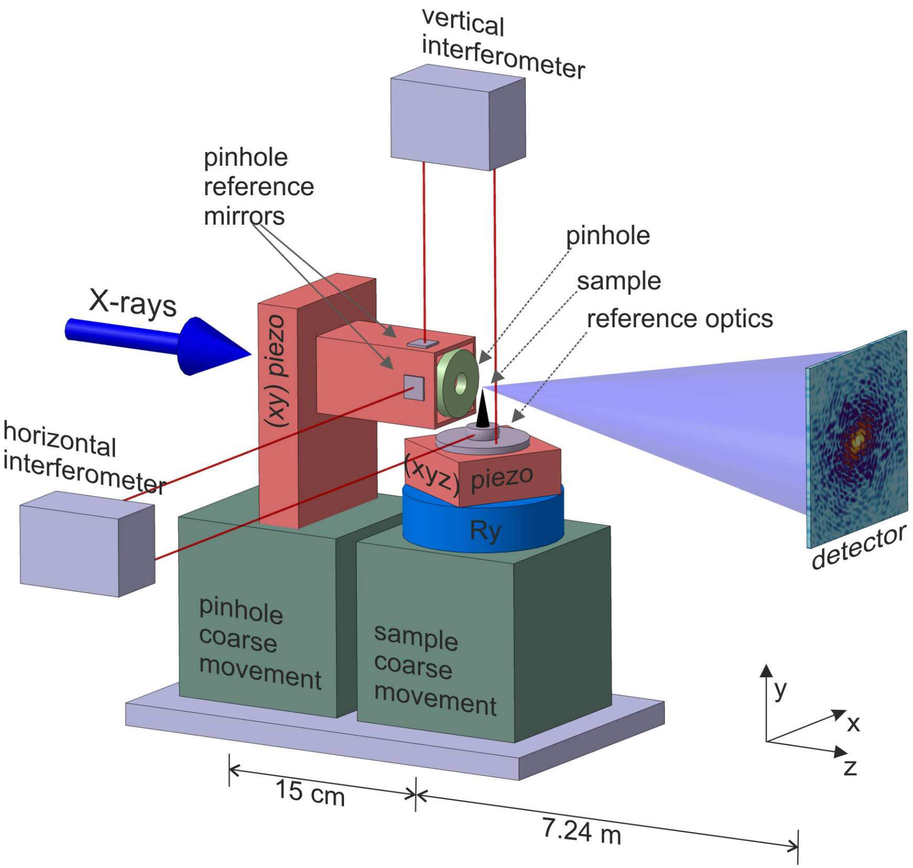
Energiebereich 250-1600 eV

OMNY Test Setup



OMNY Test Setup

Limited to atmospheric pressure and room temperature



Position of sample to optics measured by laser interferometry and actively stabilized

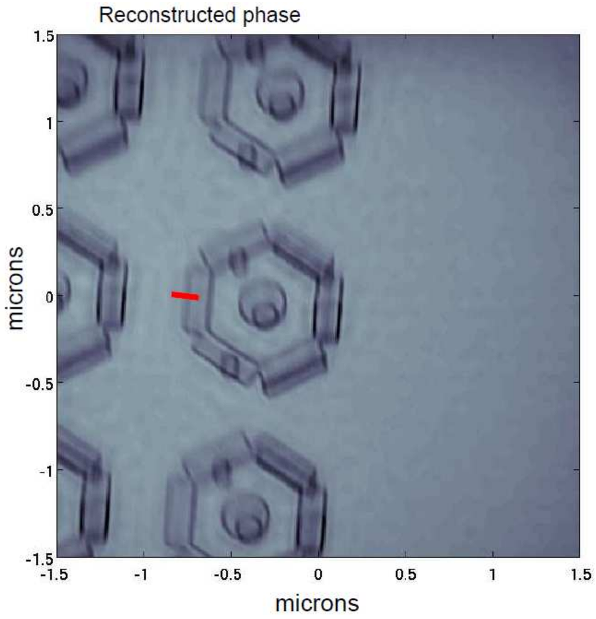
Position stability routinely reached:
8 nm (stdev) horizontally, 3 nm (stdev) vertically

→ high resolution imaging should be possible

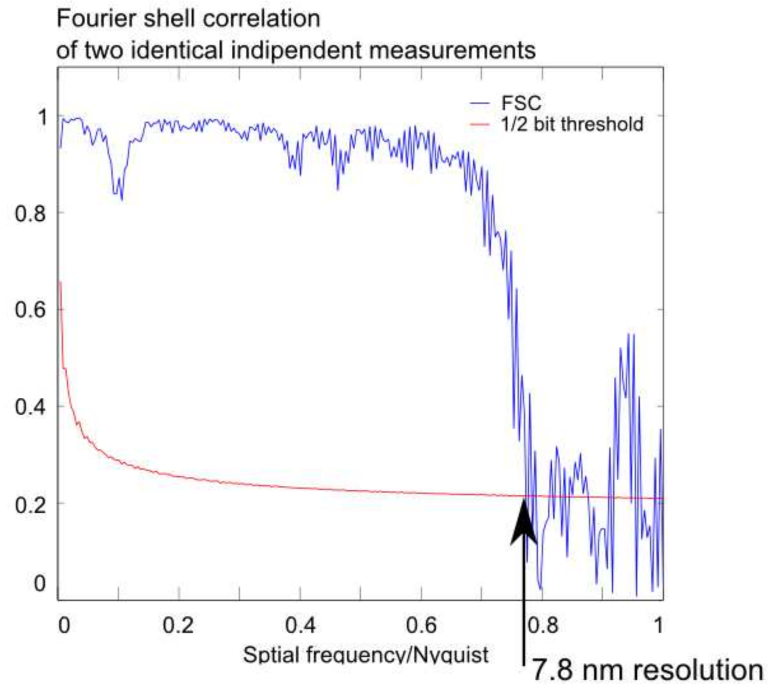
Further information can be found in Mirko Holler, Joerg Raabe, Ana Diaz, Manuel Guizar-Sicairos, Christoph Quitmann, Andreas Menzel, and Oliver Bunk, "An Instrument for 3D X-ray Nano-Imaging", Review of Scientific Instruments, accepted for publication.

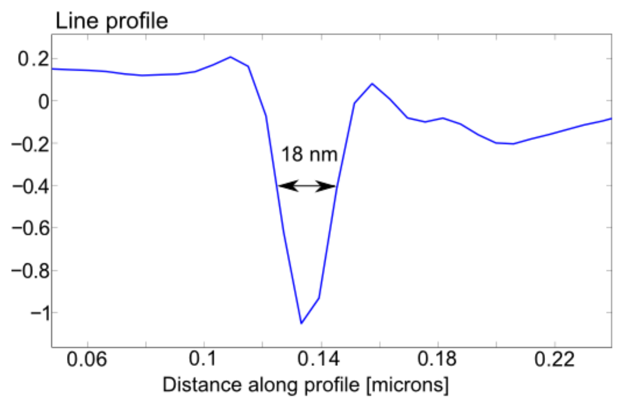
OMNY Test Setup – 2D imaging

2D lithographic test object:
concentric circles of hydrogen silsesquioxane HSQ, at two height levels subsequently coated with a conformal layer of iridium of about 17 nm in thickness by atomic layer deposition.

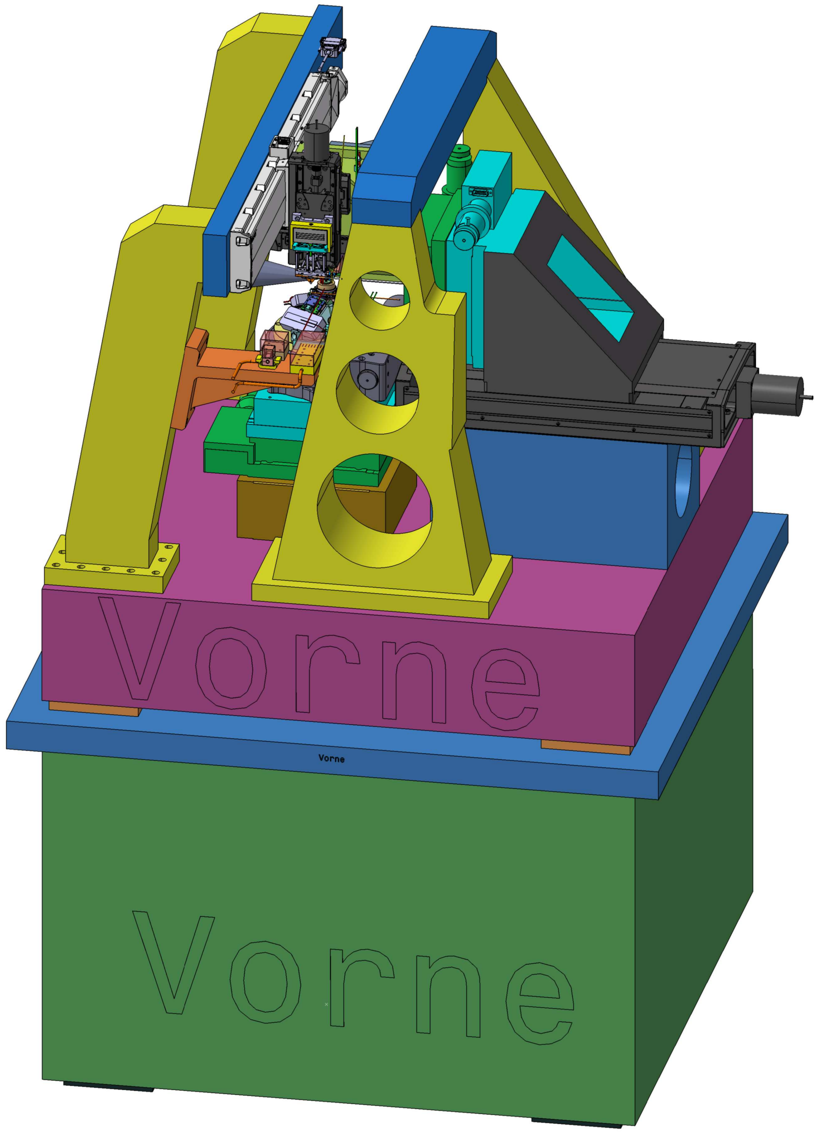


Phase of the reconstructed test structures (radians)
at a field of view of 3x3 μm^2, reconstructed to 6 nm pixel size
FZP illumination, X-ray beam Ø 2 μm





Thanks to Joan Vila-Comamala for sample preparation at LMN, PSI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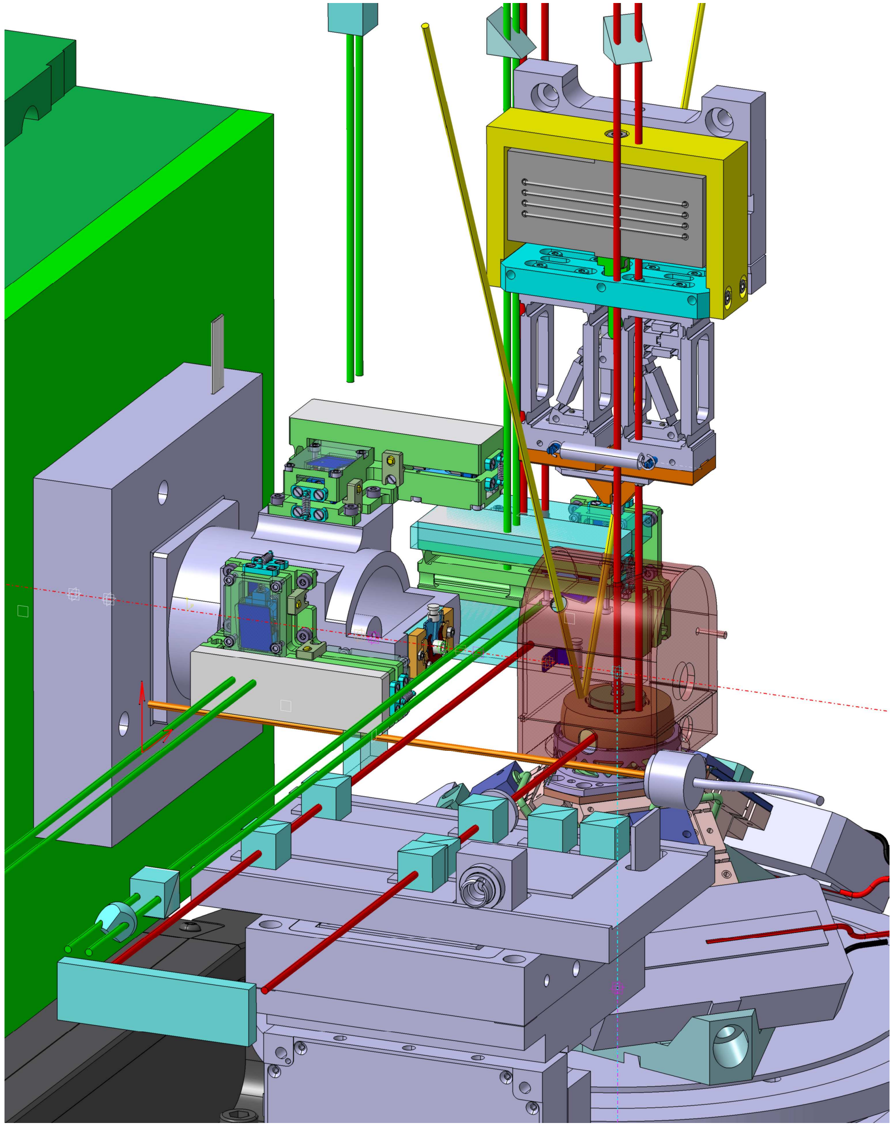
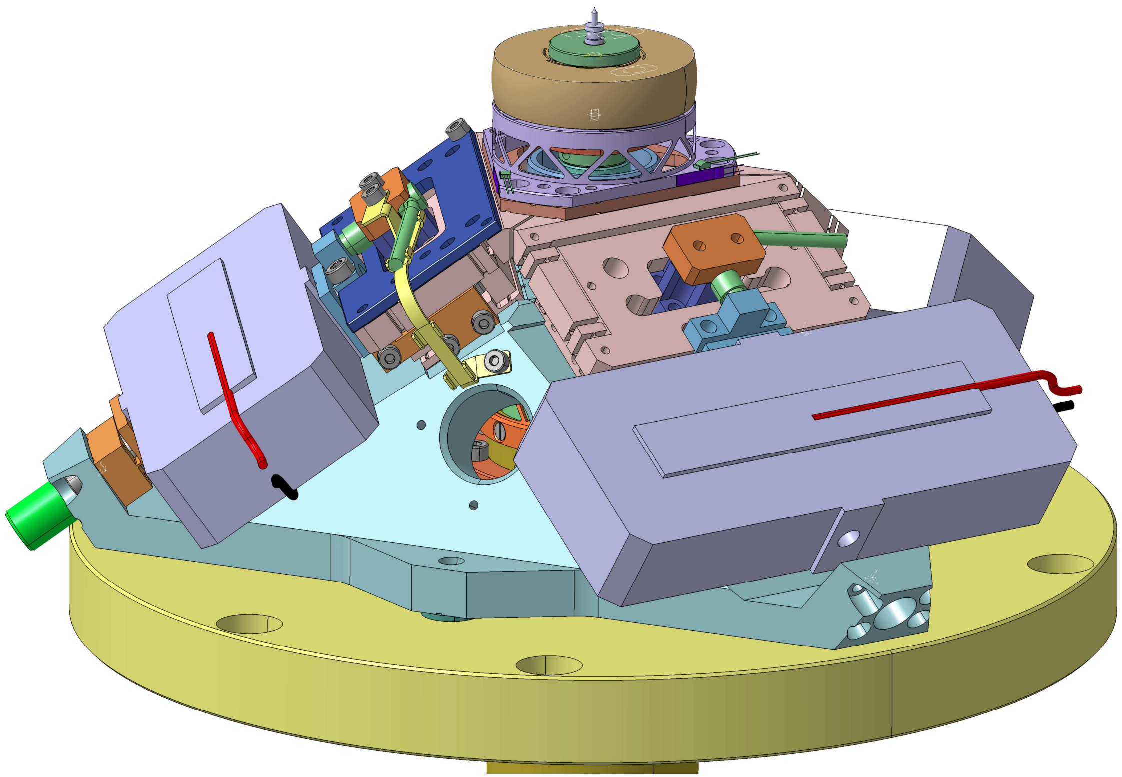


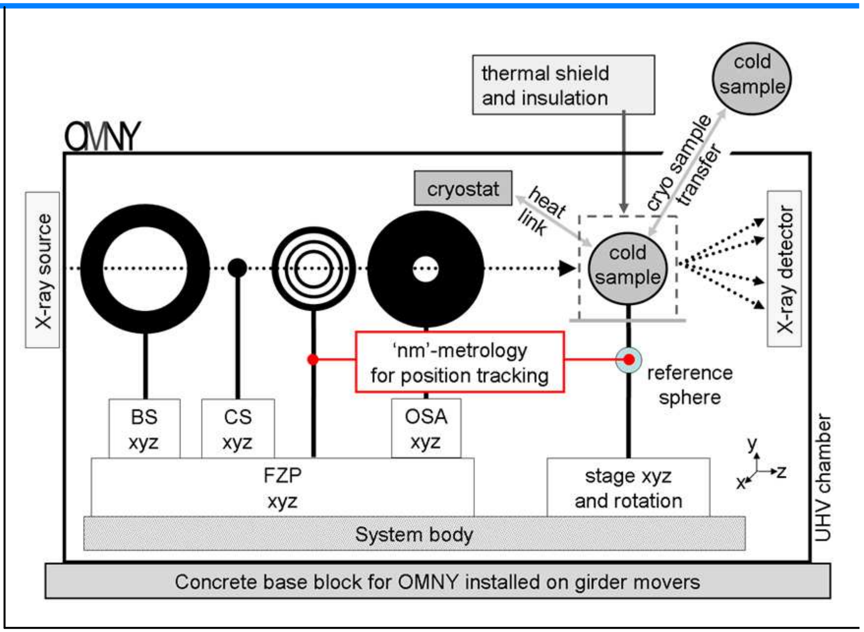
OMNY Scanner

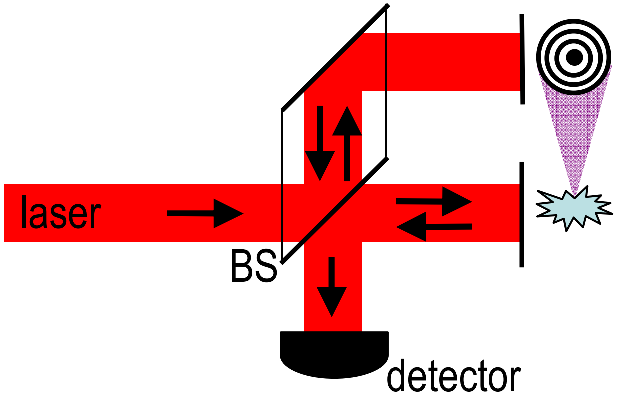


Metrology for OMNY

For high resolution imaging
accurate information on the position
of the sample in respect to the beam required
i.e. the relative position of the sample
to the Fresnel zone plate

Required:
- sub-nm resolution at high bandwidth
- exteroceptive: include thermal drifts in the measurement

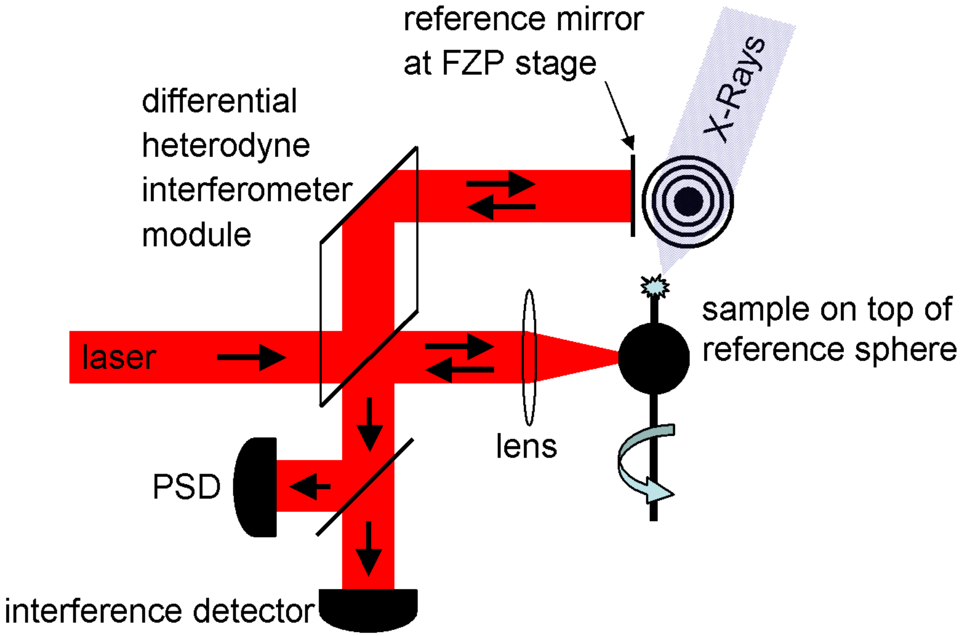


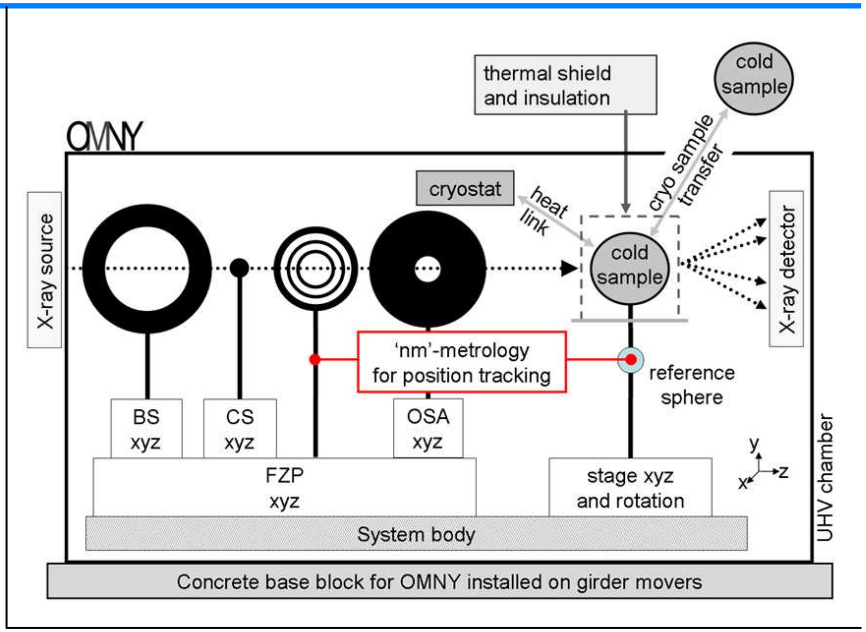


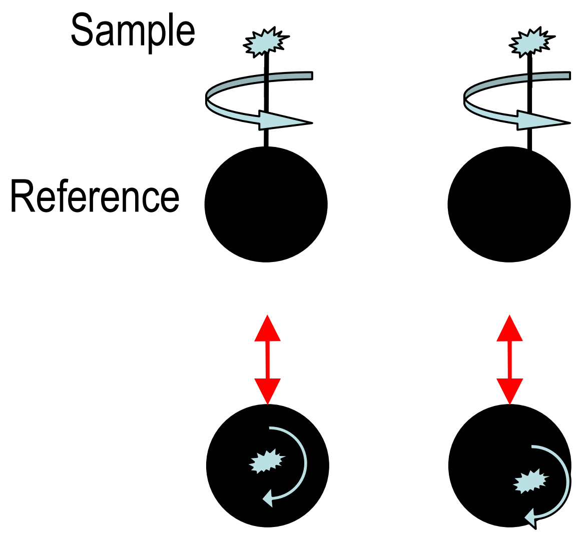
Heterodyne laser interferometry
- Resolution: 0.3 nm
- Noise: 2 nm (stdev)
- non-contact, long range
- linear scale

Metrology for OMNY: Tracking interferometer

Wobble motion is detected by laser beam deflection with a position sensitive detector (PSD)
Closed loop: interferometer tracks the reference sphere and keeps pointing a its center
→ interferometer data at all rotation angles







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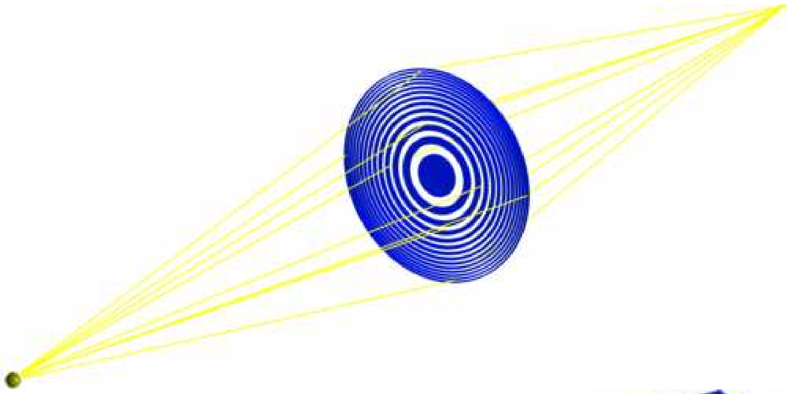
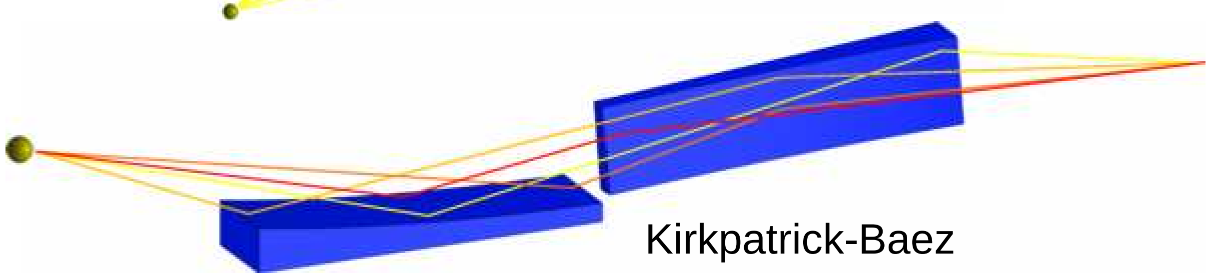
Focussing

- Zone plates

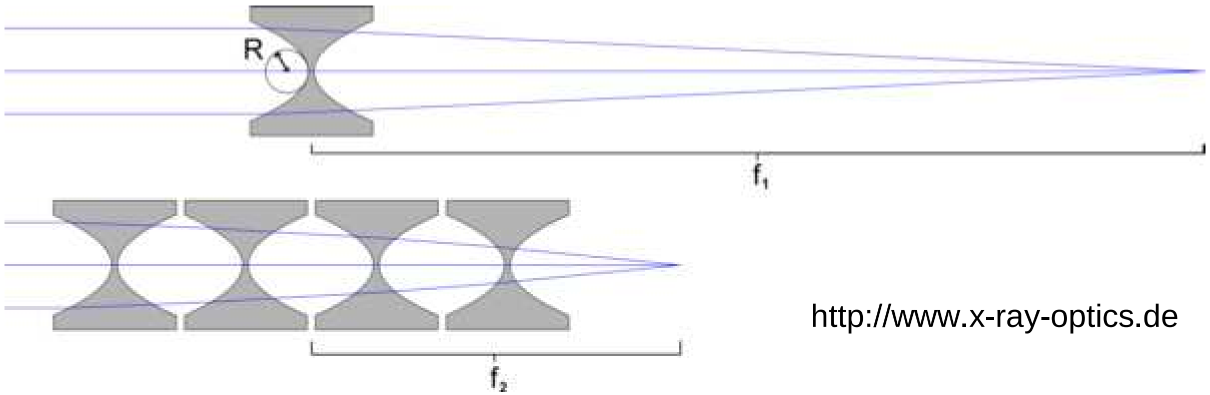


- Mirrors



Kirkpatrick-Baez

- Refractive optics

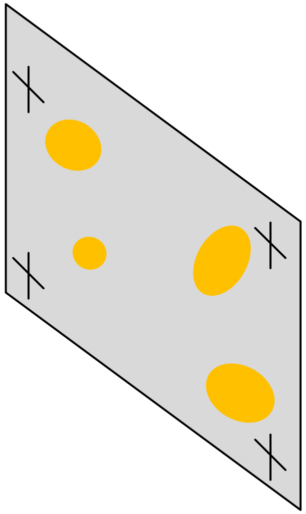


http://www.x-ray-optics.de

Finding Samples

<u>Ex situ:</u>

Measuring Positions of particles with respect to reference marks

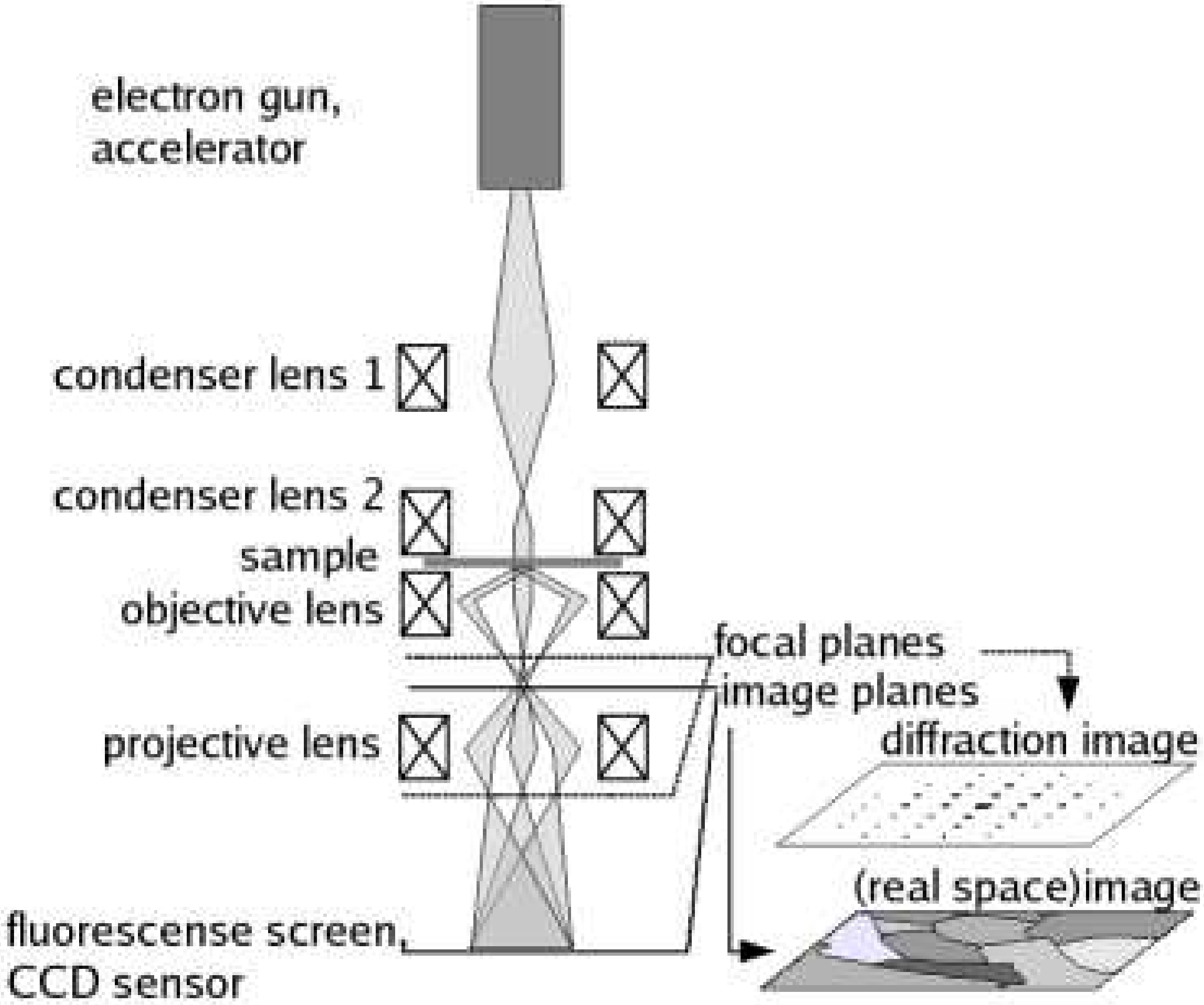


<u>In situ:</u>

Integrate complementary microscope into the endstation:

- TEM
- SEM
- Visible + x-ray microscope

TEM


electron gun,
accelerator
condenser lens 1
condenser lens 2
sample
objective lens
focal planes
image planes
projective lens
diffraction image
(real space)image
fluorescense screen,
CCD sensor

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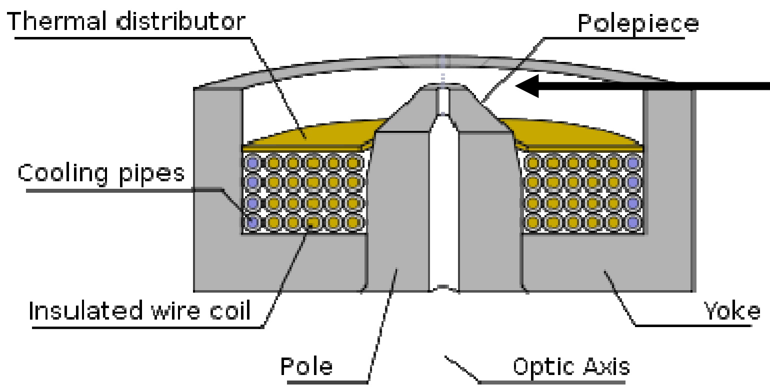
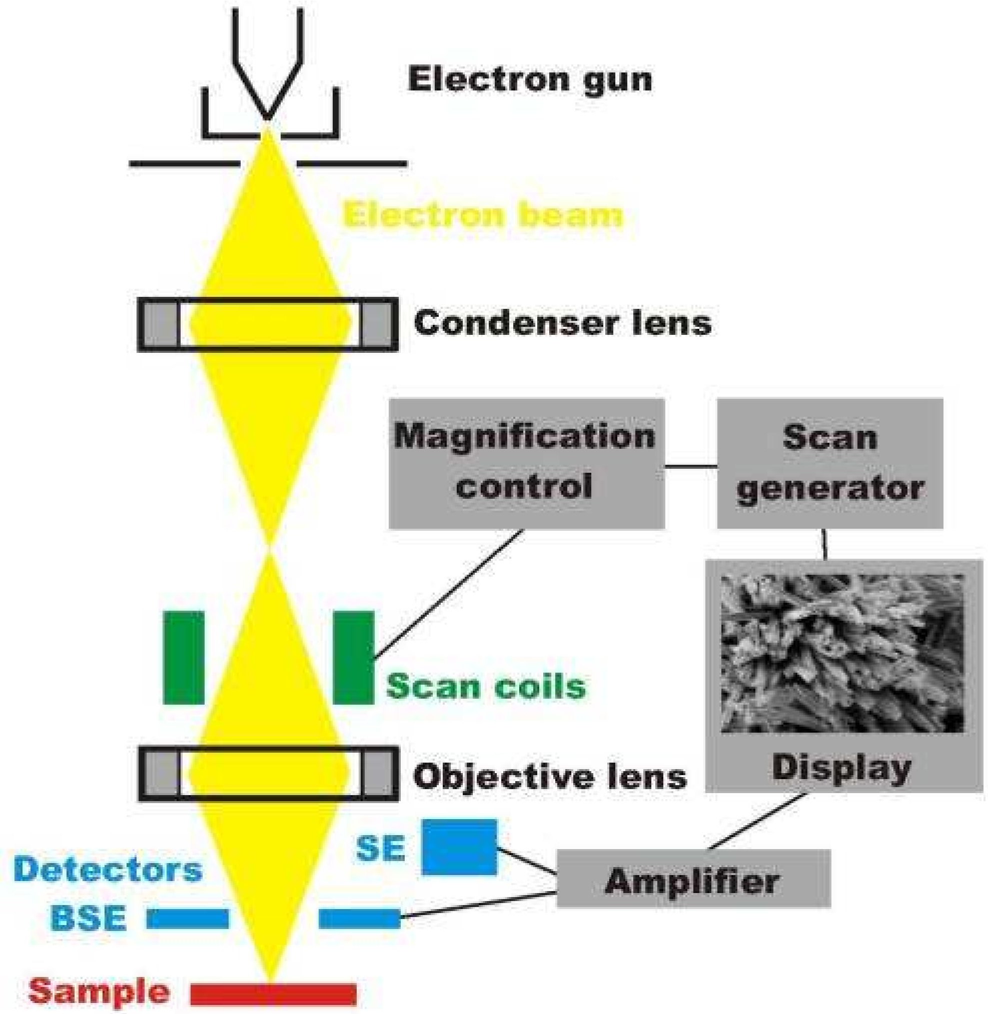
Objective lens of a TEM



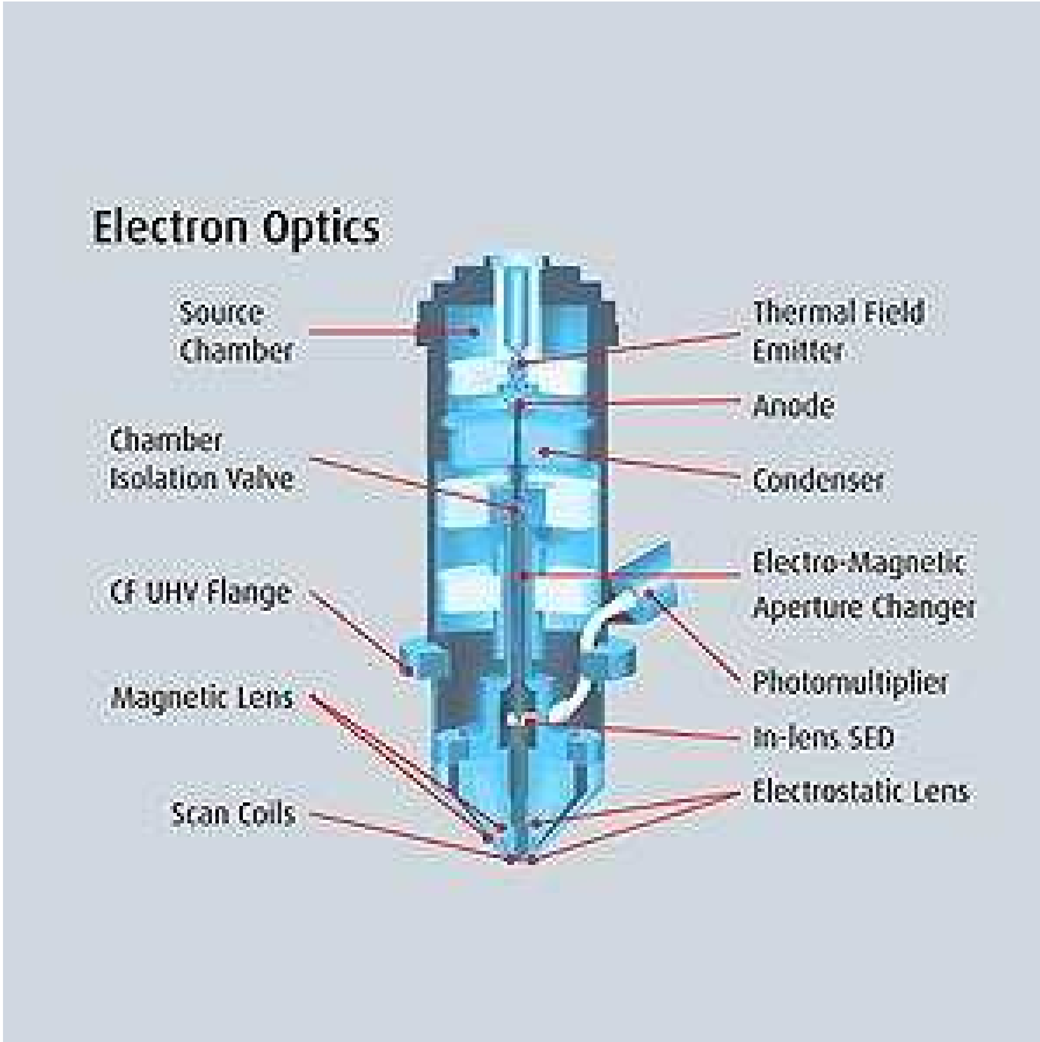
Typical gap for sample 5-10mm



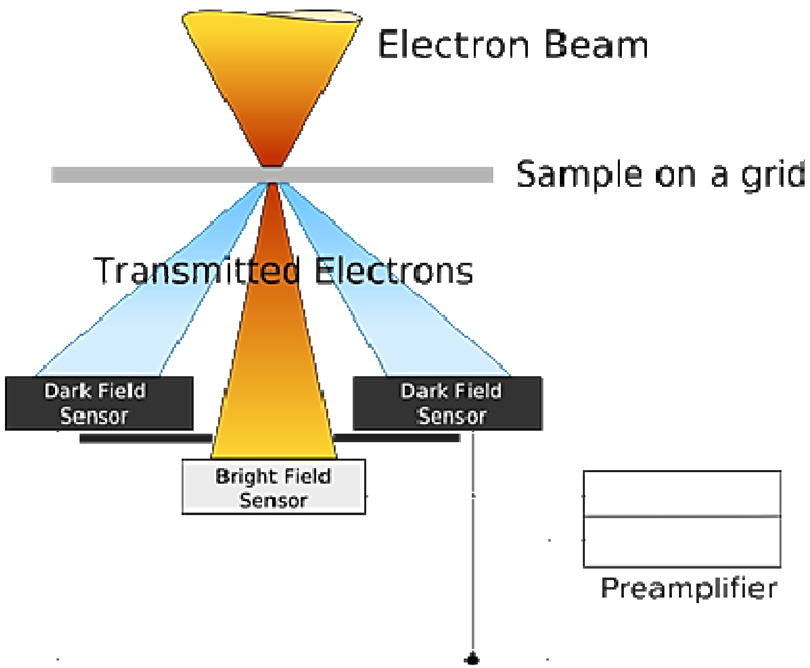
http://www.microscopy.ethz.ch

UVH SEM

Omicron: Modified Zeiss Gemini

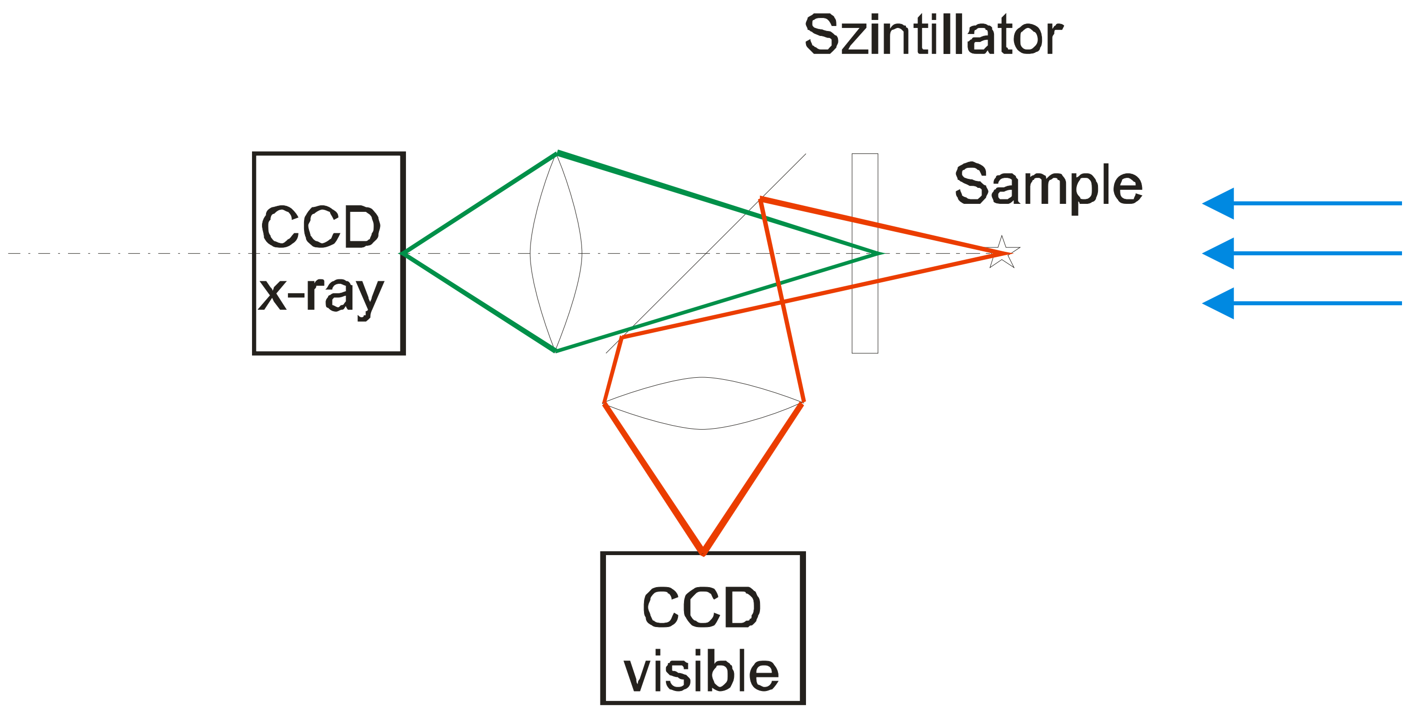


STEM in a SEM



http://www.tescan.com

Combined visible and x-ray Microscope



Conclusions

- Stable and precise positioning System with exteroceptive metrology system
- Complementary in situ microscopy
- Cryo sample transfer

Thanks to the OMNY Team:

- Mirko Holler

- Markus Vitins
- Hansueli Walther
- Thierry Lachat