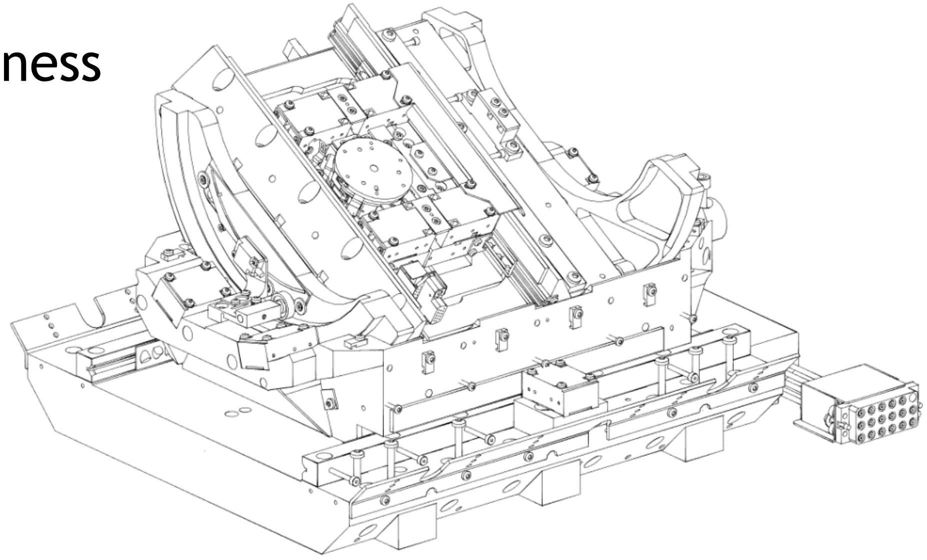
Vybrané kapitoly z elektronové mikroskopie

Sample manipulation

Frantisek Vaske

General requirements for sample manipulator

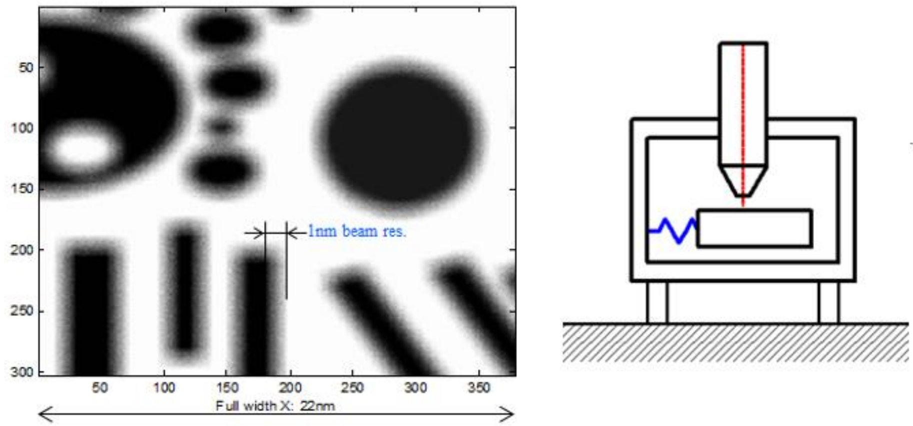
- Stands still – no vibrations
  - High stifness, low mass
- Stands still – no drift
  - Temperature stability
- Vacuum compatibility and tightness
- Non magnetic
- Moves the sample
  - Accuracy
  - Repeatability

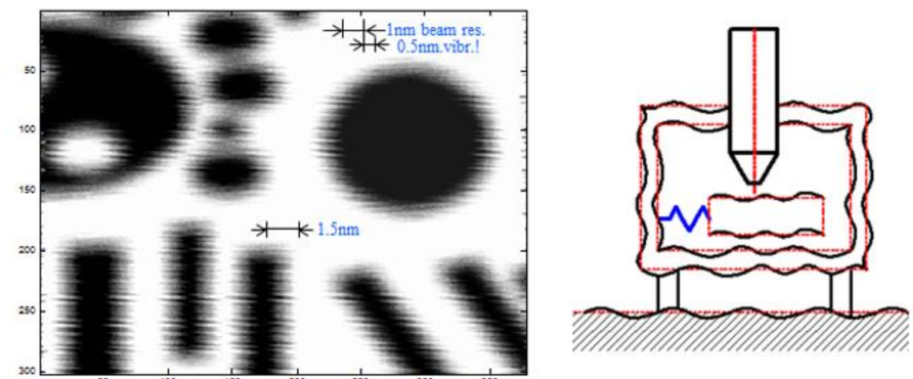


General requirements for sample manipulator
No vibrations

Sources of vibrations
- Floor vibrations
- Accoustics

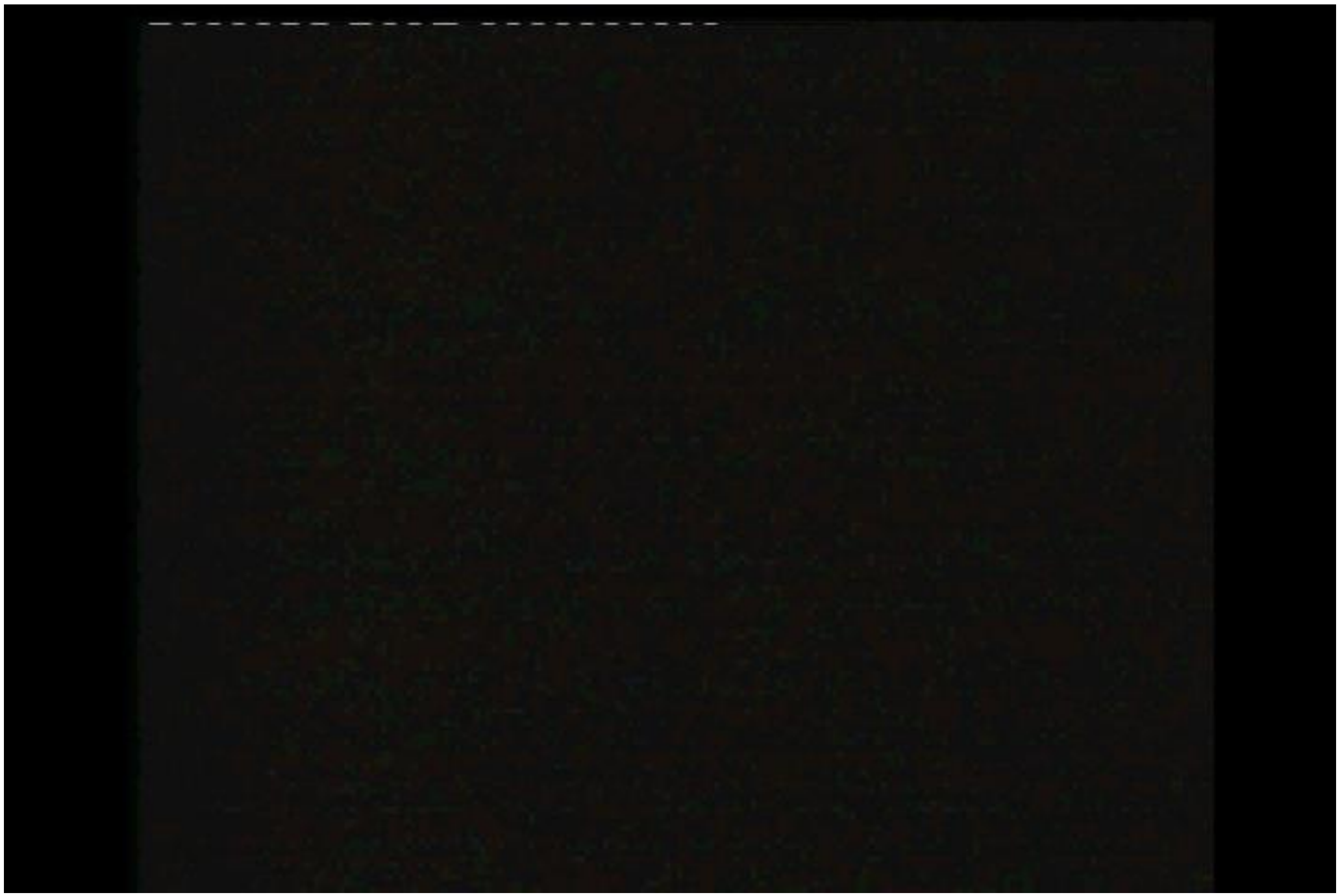
Loss of resolution
Image flags – SEM specific (resolution, dwell)





General requirements for sample manipulator

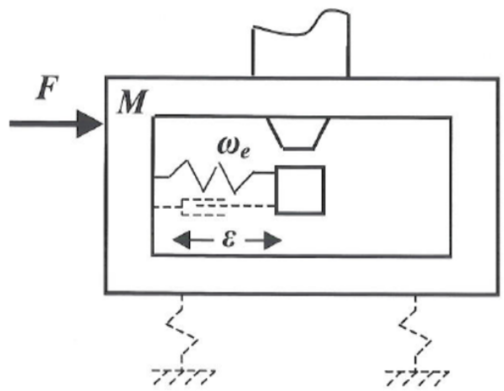
No vibrations

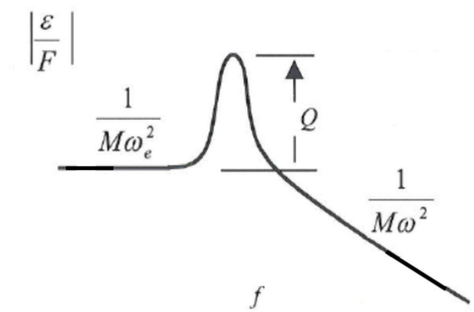


General requirements for sample manipulator
No vibrations

Increase stiffness
- Lightweigth stage
- Friction brakes
- Herthzian contact in bearings

Increase damping
- Tuned mass damper



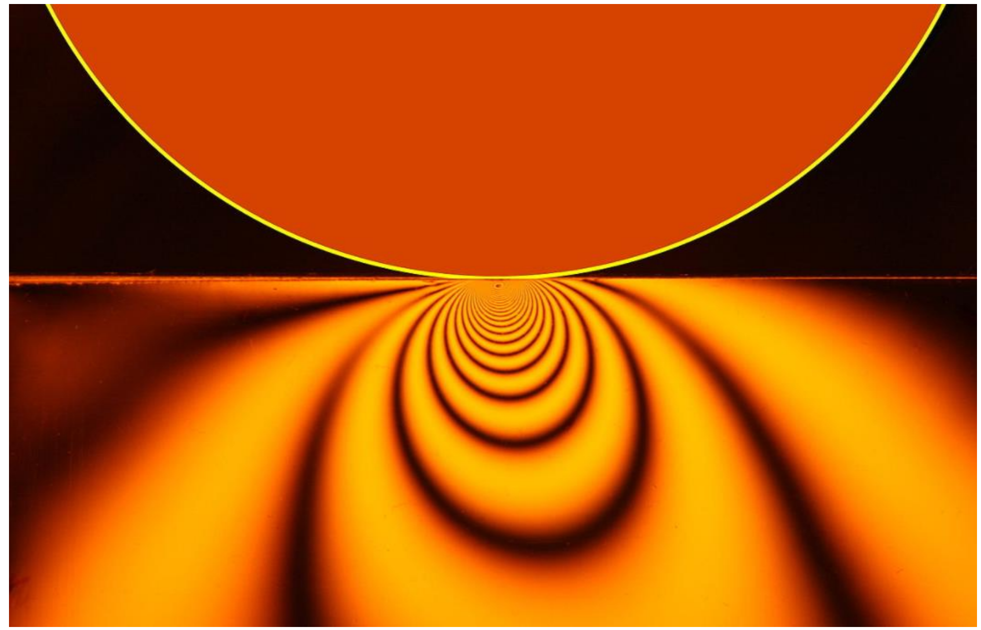


Strive for:

High M high ω_e Low Q

General requirements for sample manipulator

No vibrations



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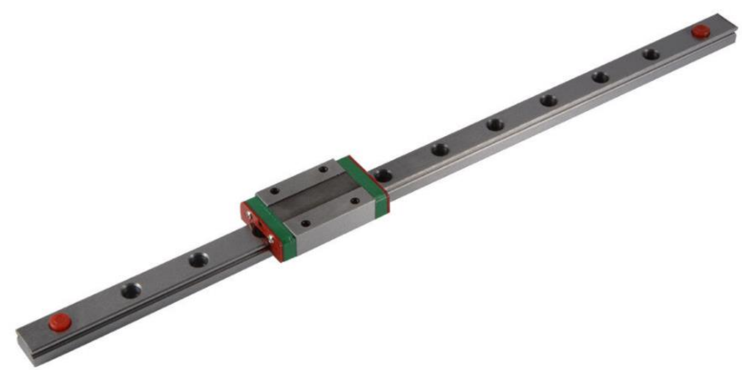

General requirements for sample manipulator
No vibrations

Increase stiffness
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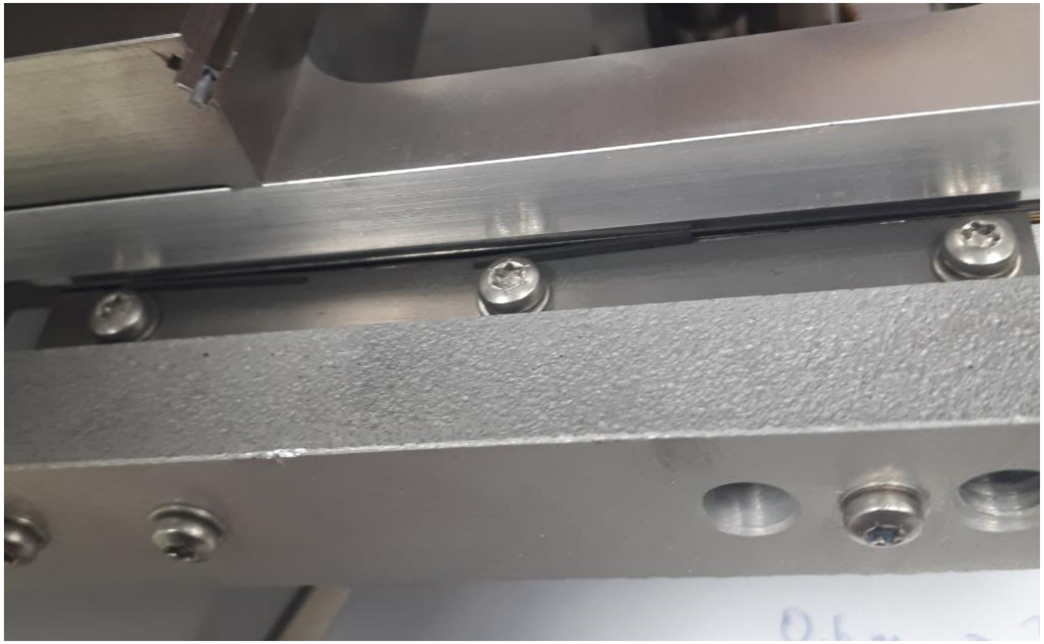


General requirements for sample manipulator
No vibrations

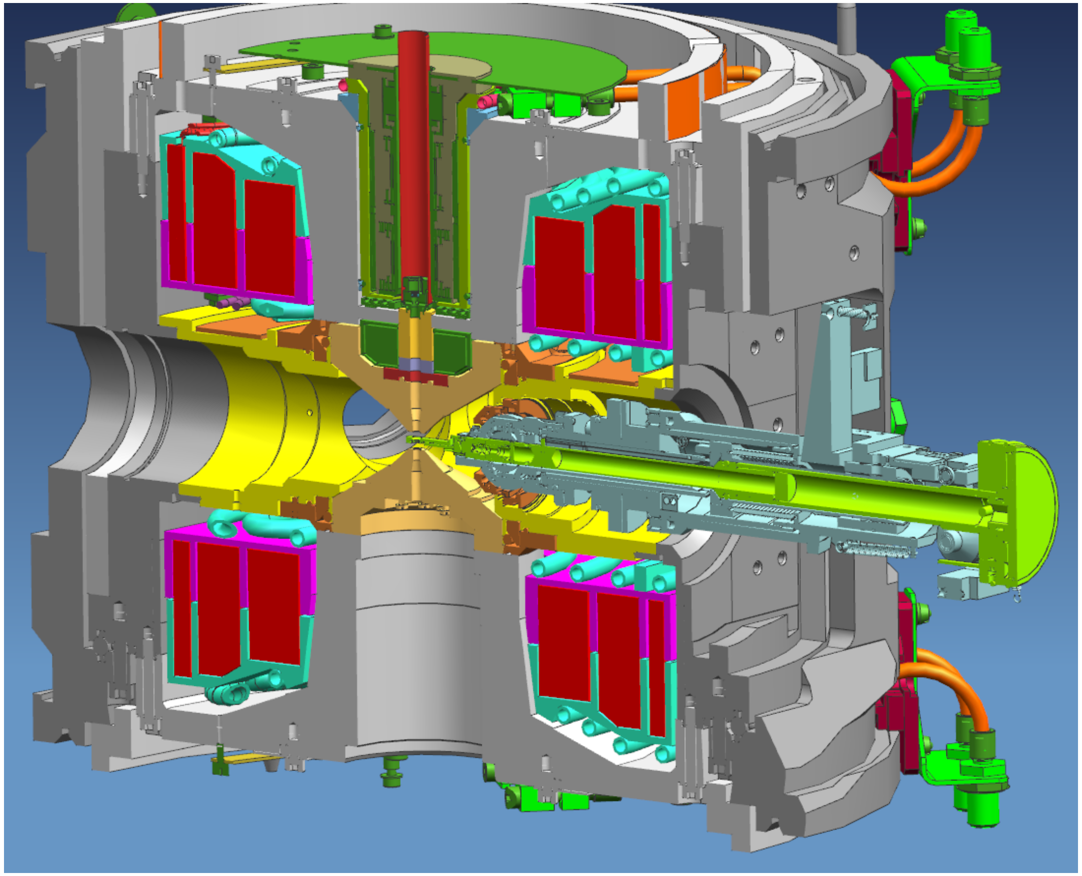
Increase damping

- Tuned mass damper

General requirements for sample manipulator
Thermal drift

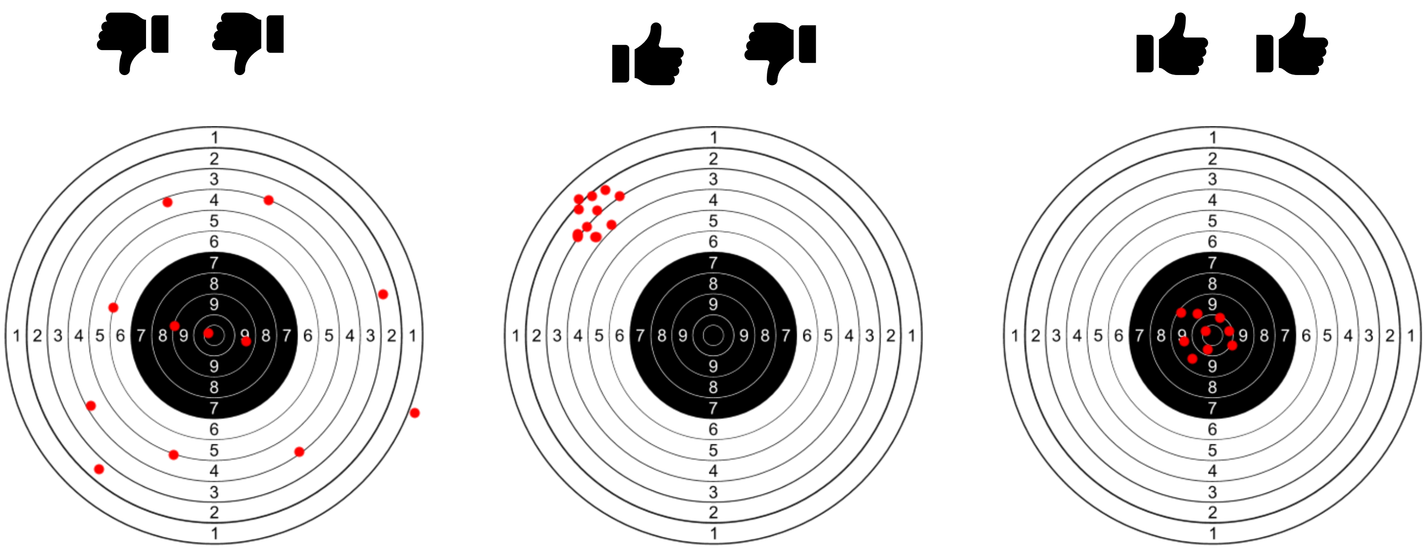
- **Thermal drift**
  - Caused by thermal expansion
    - Temperature stability
    - Smart design

$$drift = L_0\,\alpha\,(T_1 - T_0)\,/t$$



General requirements for sample manipulator
Motion

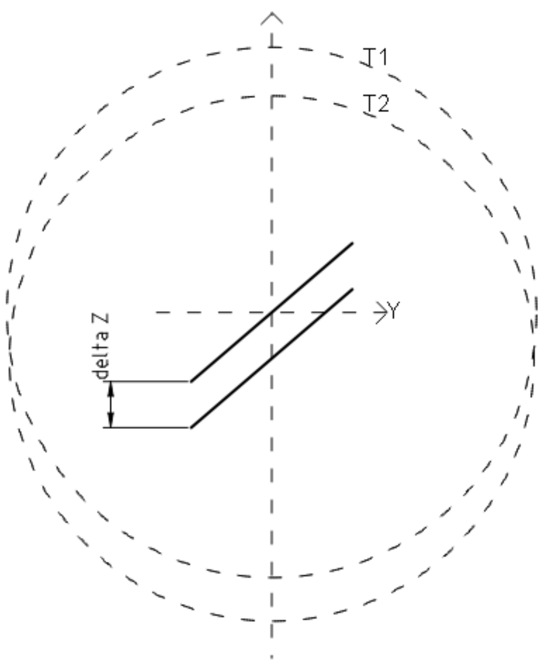
- Accuracy
- Repeatability – more important, can be calibrated
- Feedback control, mapping

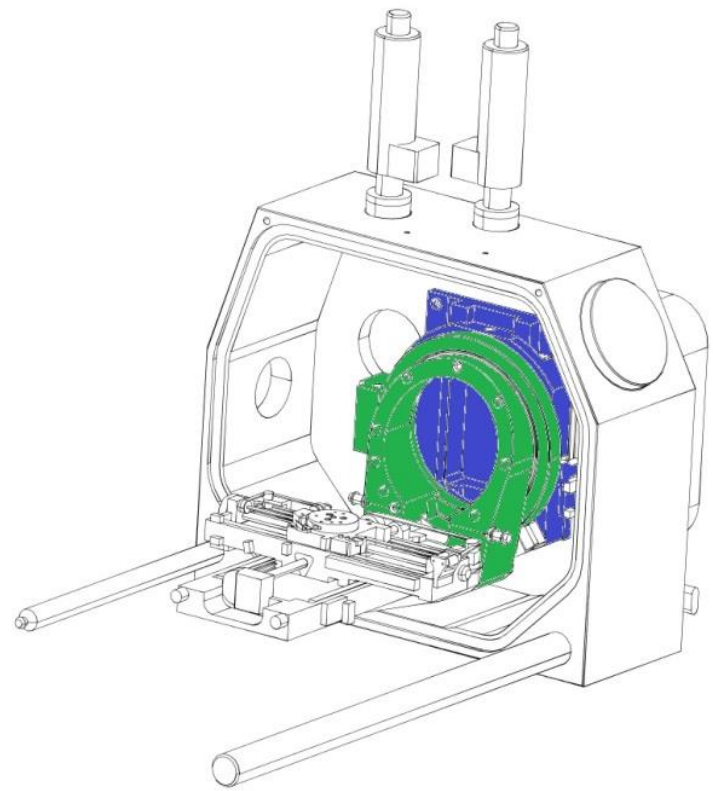


General requirements for sample manipulator
Eucentricity

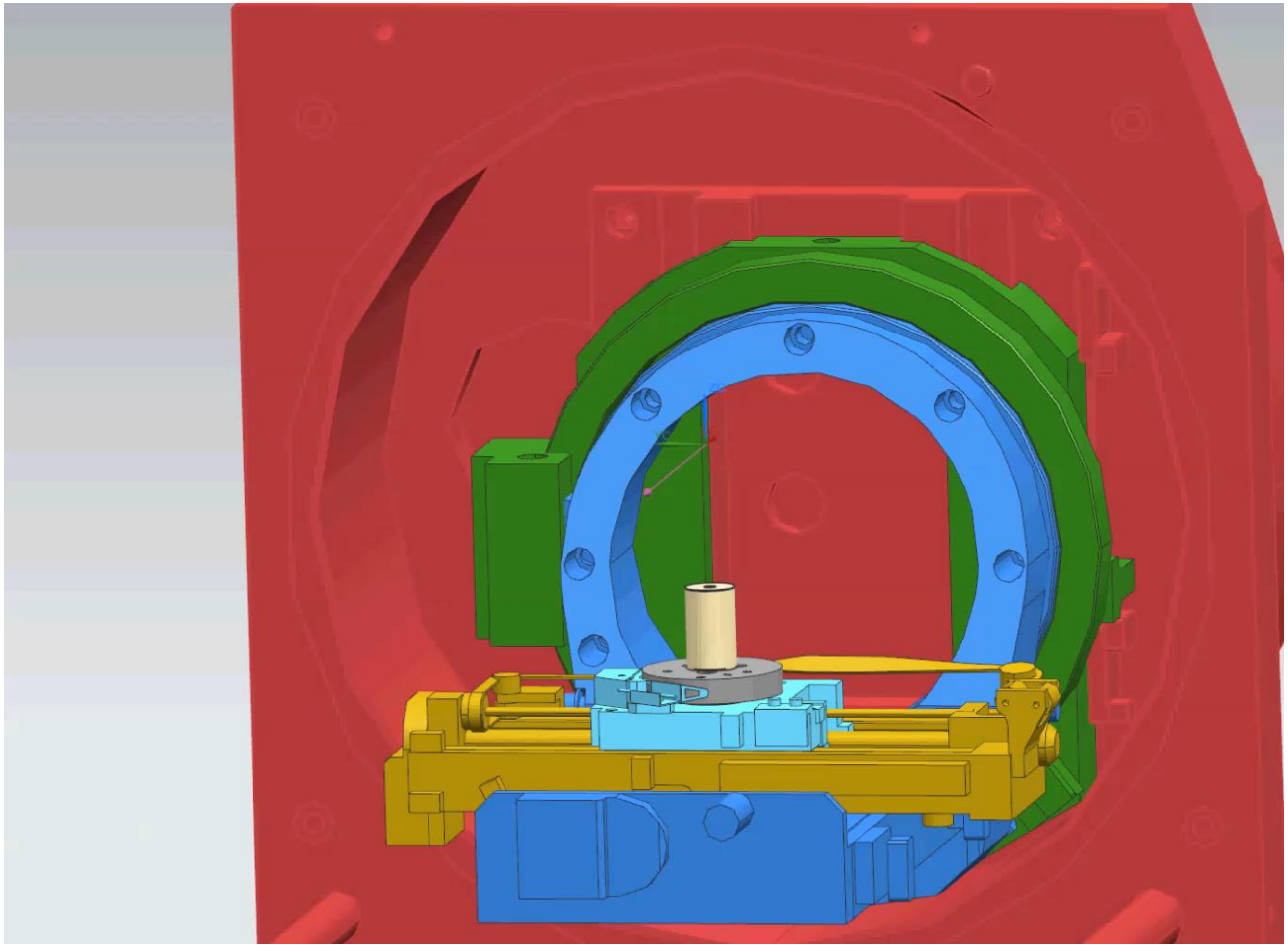
Non Eucetric stage

- Z stacked before T
- Compensation in Y needed





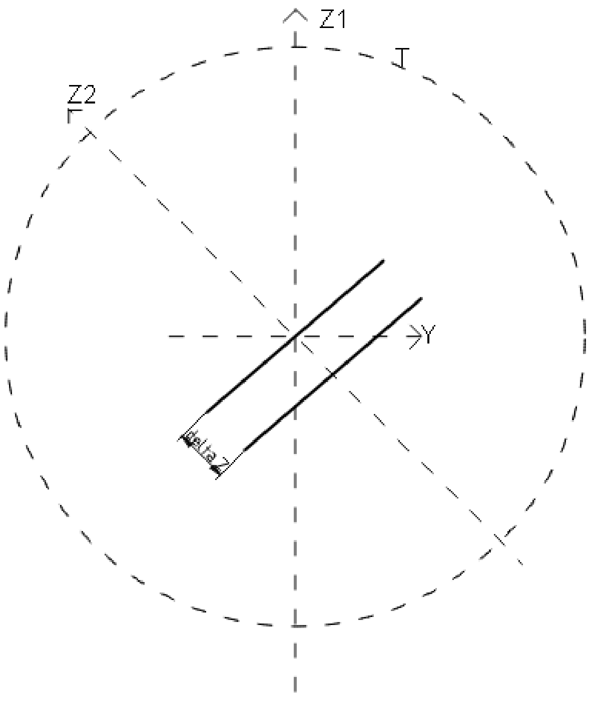
General requirements for sample manipulator
Eucentricity

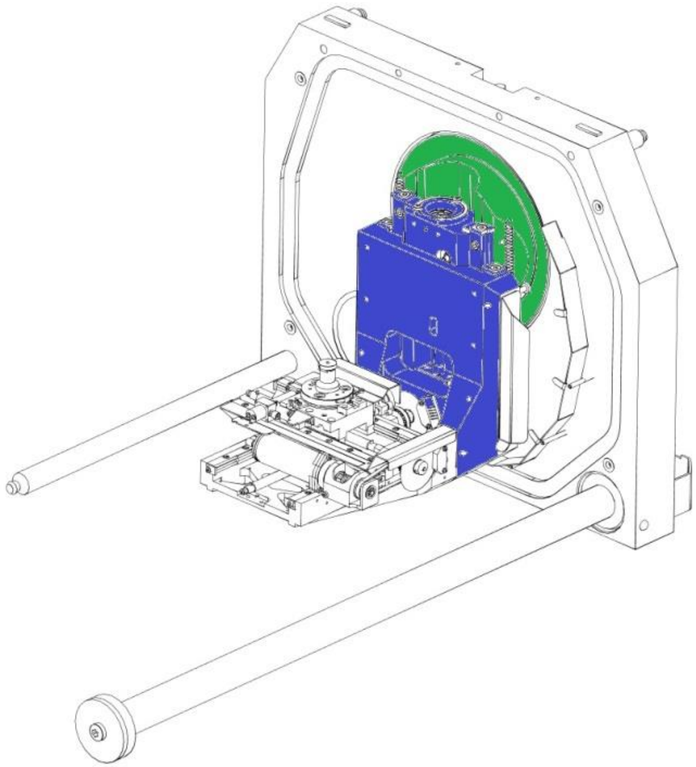


General requirements for sample manipulator
Eucentricity

Eucetric stage

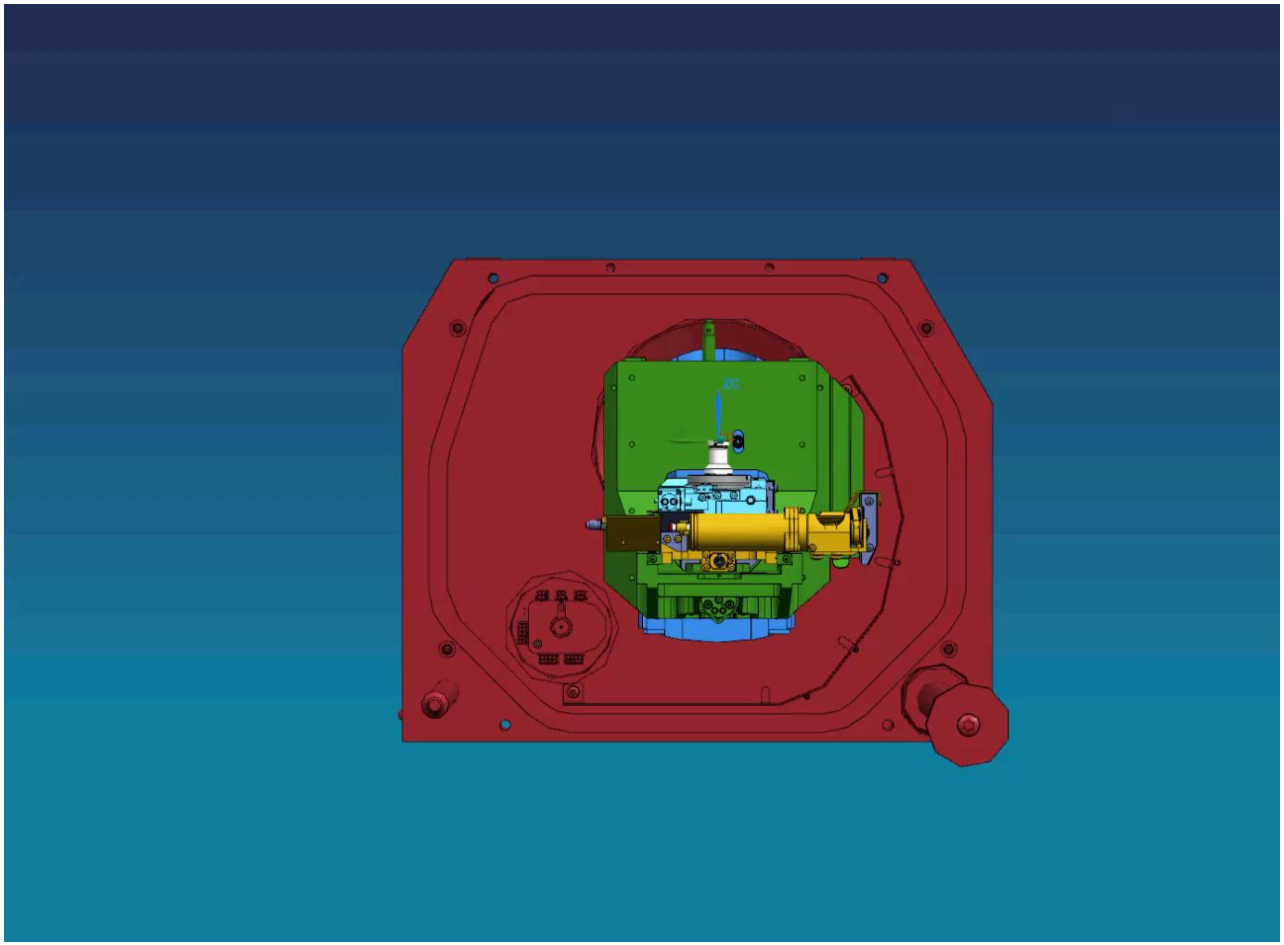
- T stacked before Z
- Preferable for SEM/FIB





General requirements for sample manipulator

Eucentricity



General requirements for sample manipulator
Eucentricity

General requirements for sample manipulator

System compliance

Vacuum compatibility to reduce contamination

- Avoid soft plastics, Zinc (brass), magnesium, cadmium, anodizing, paints…
- Use PEEK, steel, aluminium alloys, bronze, chemical nickel plating

Magnetic compatibility to not to distort beams

- Avoid magnetic materials – steel, iron, electrolytic nickel plating
- Use Aluminium alloys, stainless steel
- AC fields are problematic (motors)
- DC shifts beams predictably

Chemical resistance to gas chemistry

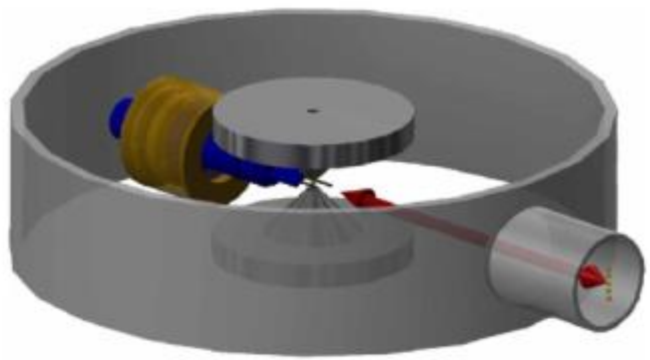
- Non reactive surfaces – gold plating if needed

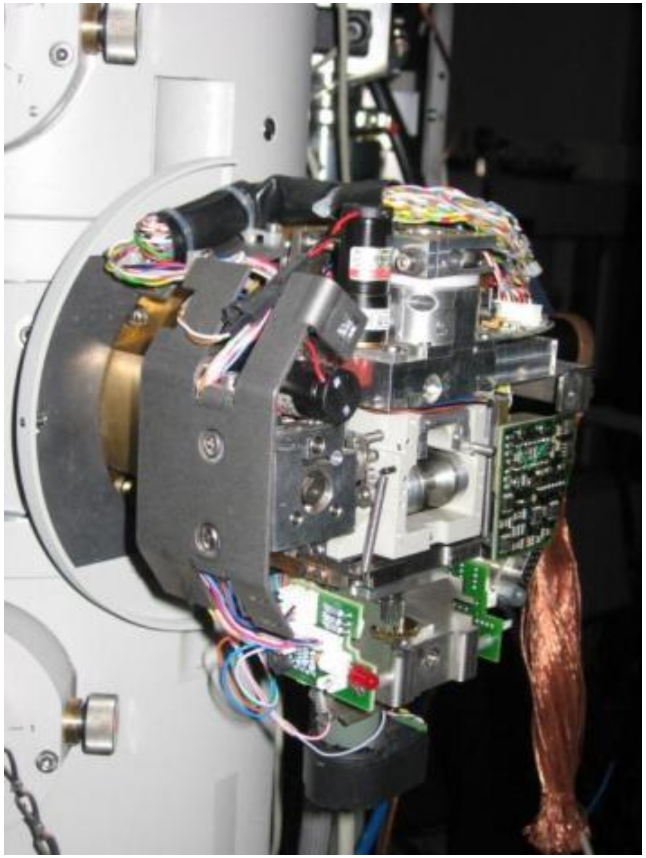
Vacuum tightness

- Consider electrical feedthrough, dynamic seals
- 1e-8 mbar*l/s

TEM stages

- **Mounted on the column**
  - At atmosphere
- Inserts sample on a holder into objective lens
- Additional functionality achieved by sample holders

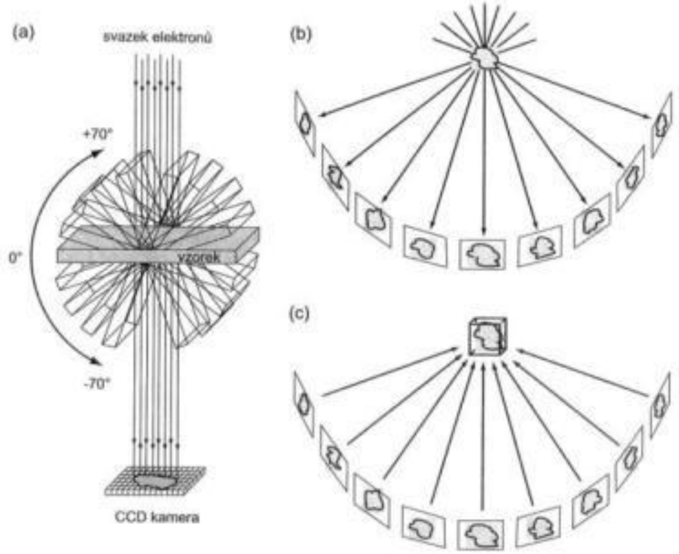




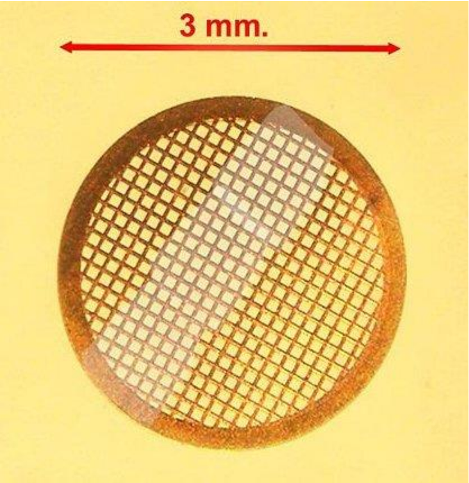
TEM stages

- Movement ranges to accomodate standard ø 3 mm sample (TEM grid) and use case
- Sample size is a compromise between magnification needed and practicality
- Tilt axis for 3D reconstruction

9.10 ELEKTRONOVÁ TOMOGRAFIE A 3D REKONSTRUKCE 199

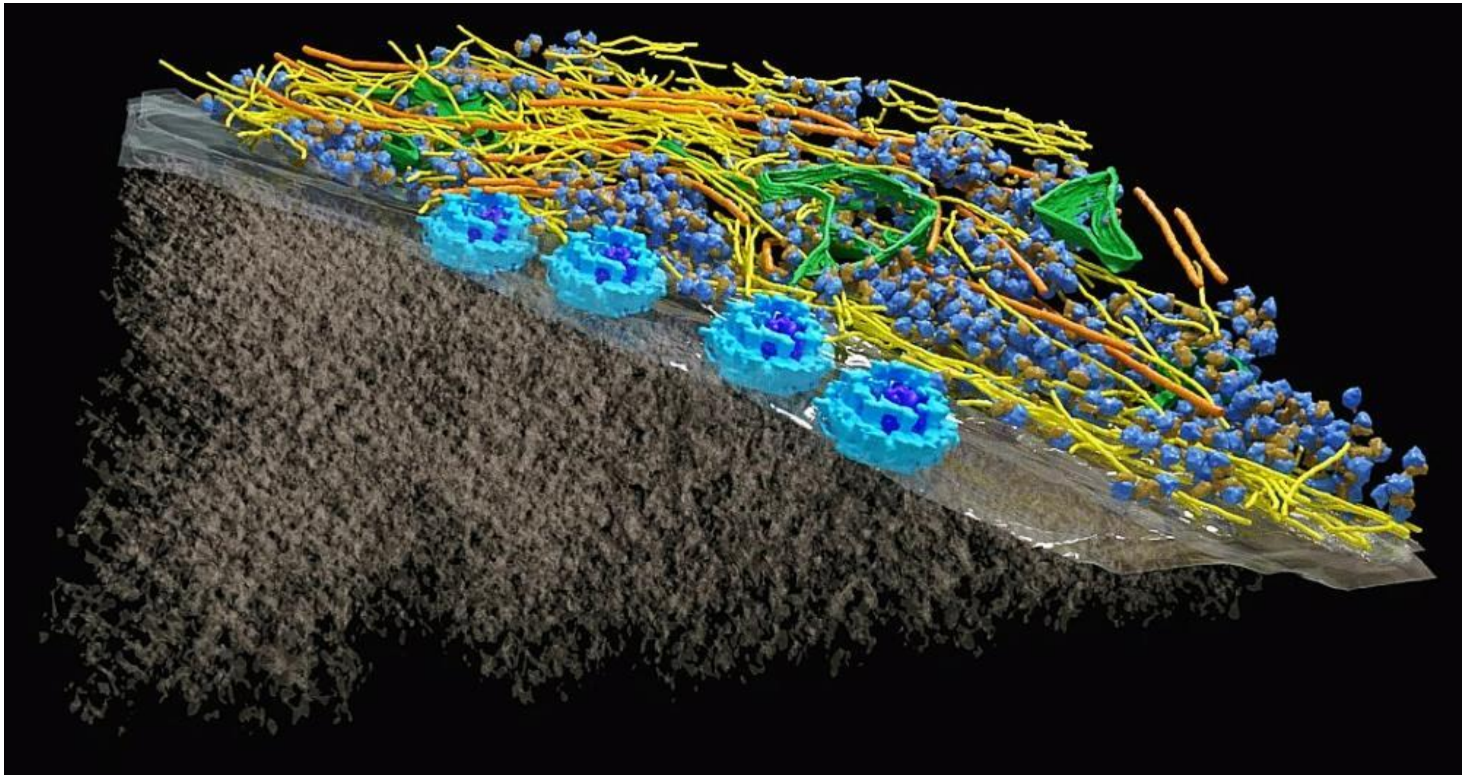


Obr. 9.41. Schéma elektronové tomografie a 3D rekonstrukce: (a) Registrace série snímků při postupném naklápění vzorku v rozmezí úhlů ±70°, (b) Průměty objektu na jednotlivých snímcích, (c) Zpětná počítačová rekonstrukce původního trojrozměrného objektu. Upraveno podle [SB05].



TEM stages

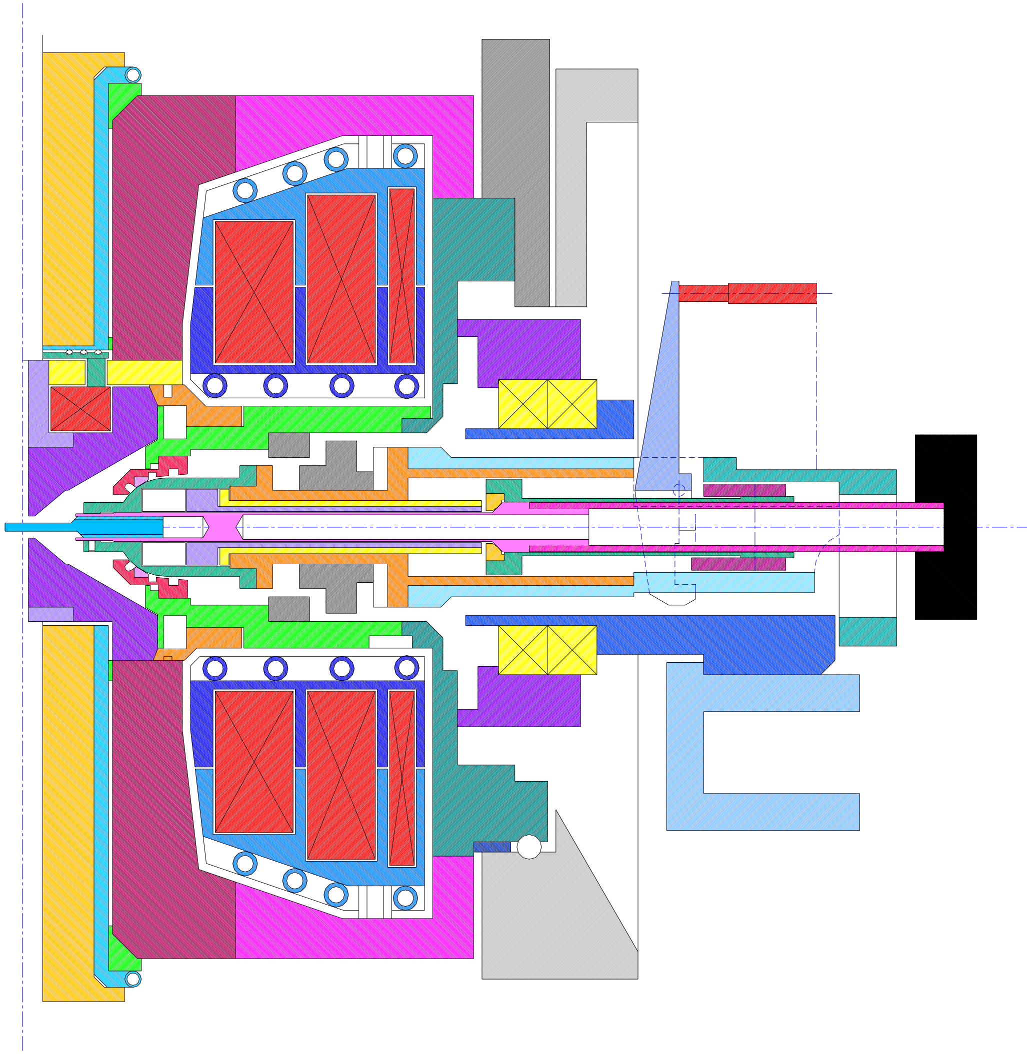
- Tilt axis for 3D reconstruction



TEM stages design

Example parameters

- XY range ±1.5mm
- Z range ± 0.375 mm
- Tilt ± 90°
- Specimen drift < 0.5nm/min
- Smallest steps in nanometres



Design principles

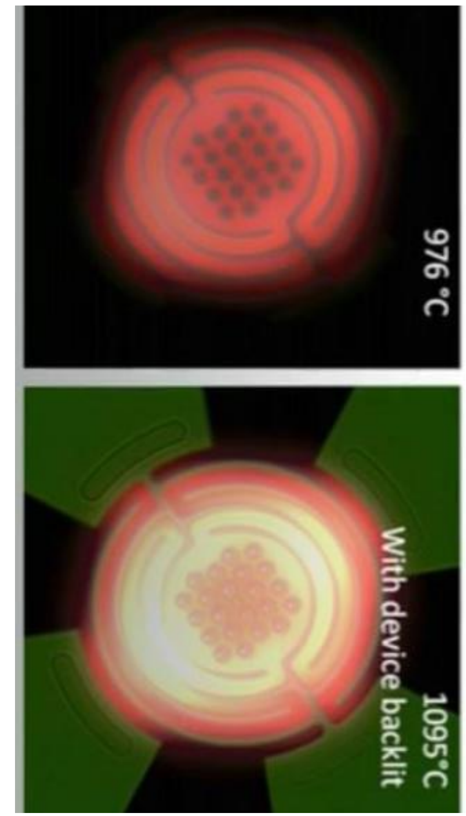
- Flexure design
- Paralelograms, lever mechanisms
- Pretension in gears
- Tuned mass damper
- Friction brakes
- DC or piezo driven

TEM stages – sample holders – extra functionality

Multisample

Double tilt

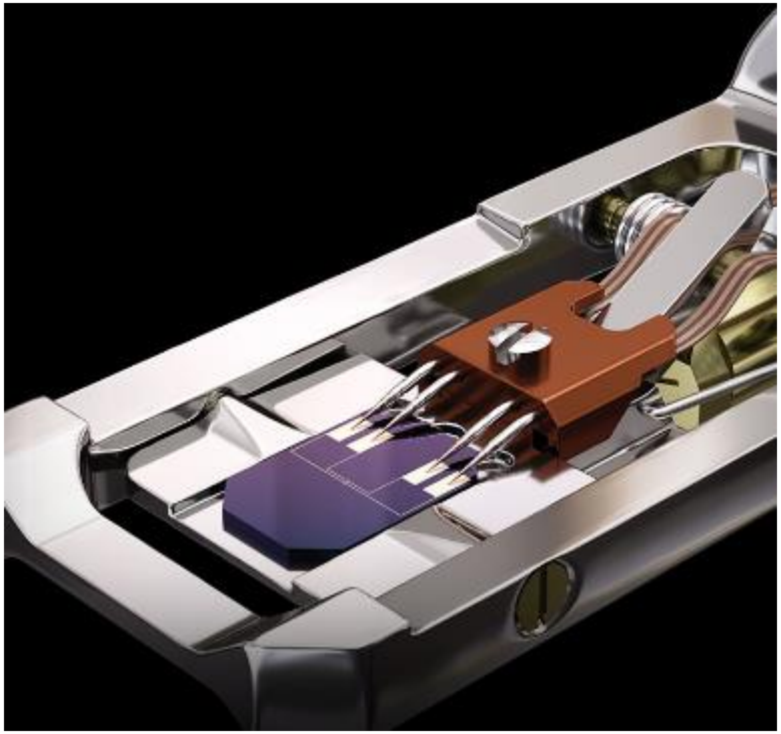
Hot



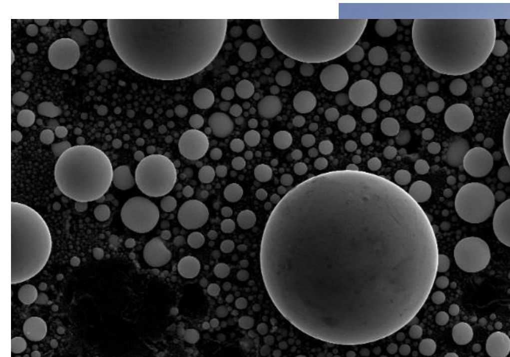
TEM stages – sample holders – extra functionality

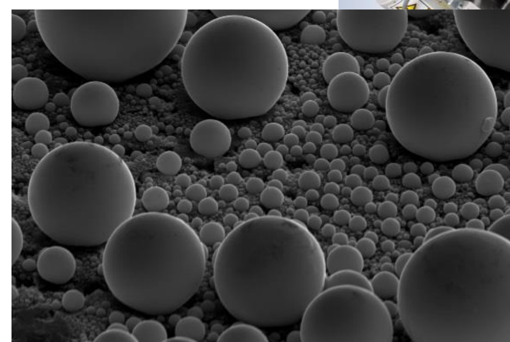
Ambient

Electrical



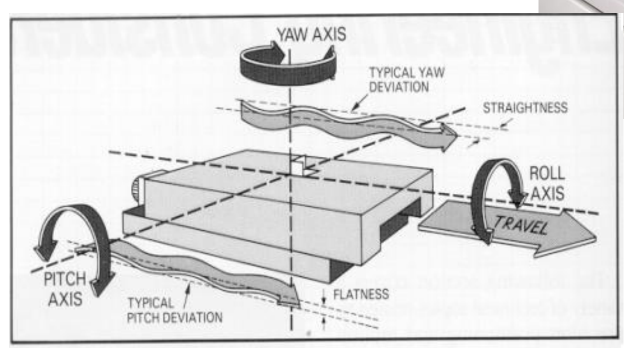
SEM stages





Operate inside a vacuum chamber
- On chamber door
- On bottom of chamber

High movement ranges
- 100x100mm XY
- Waffer inspection, mineralogy
- Accessories operation – load lock, STEM detectors…
- Influence of yaw, pitch, roll errors in linear guides

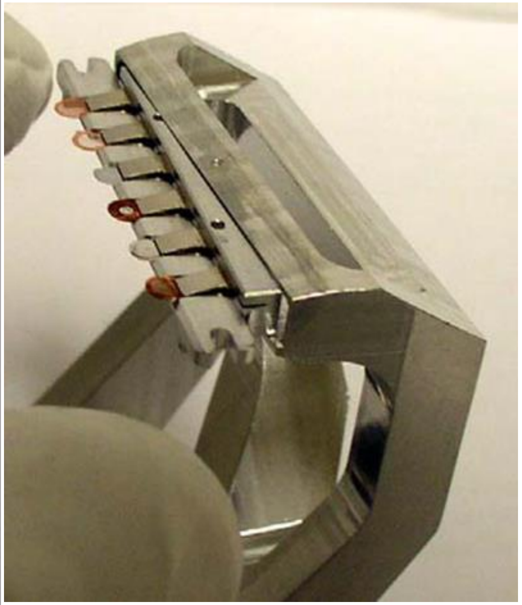


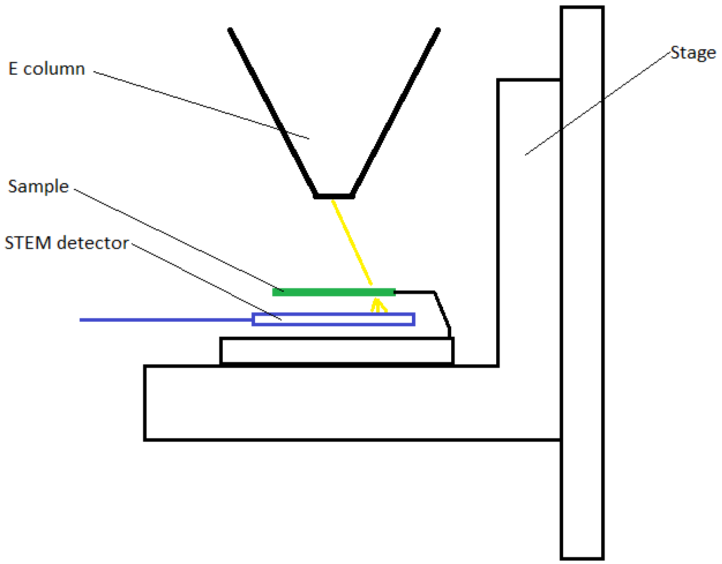
Tilt axis
- SEM and FIB beam planes
- Topography information

SEM stages sample holders

- For single or multiple samples
- Stiff
- Conductive - grounding

- Can bring extra functionality - STEM



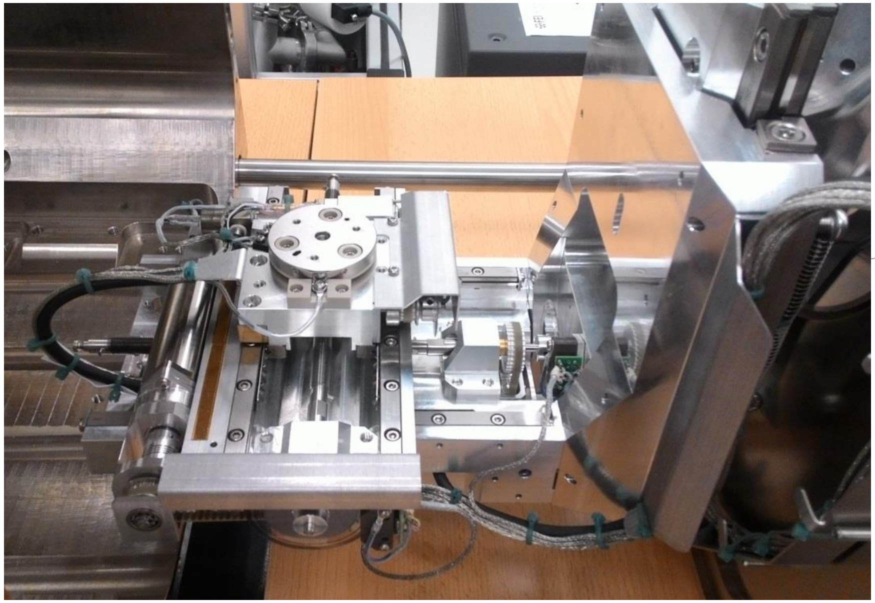


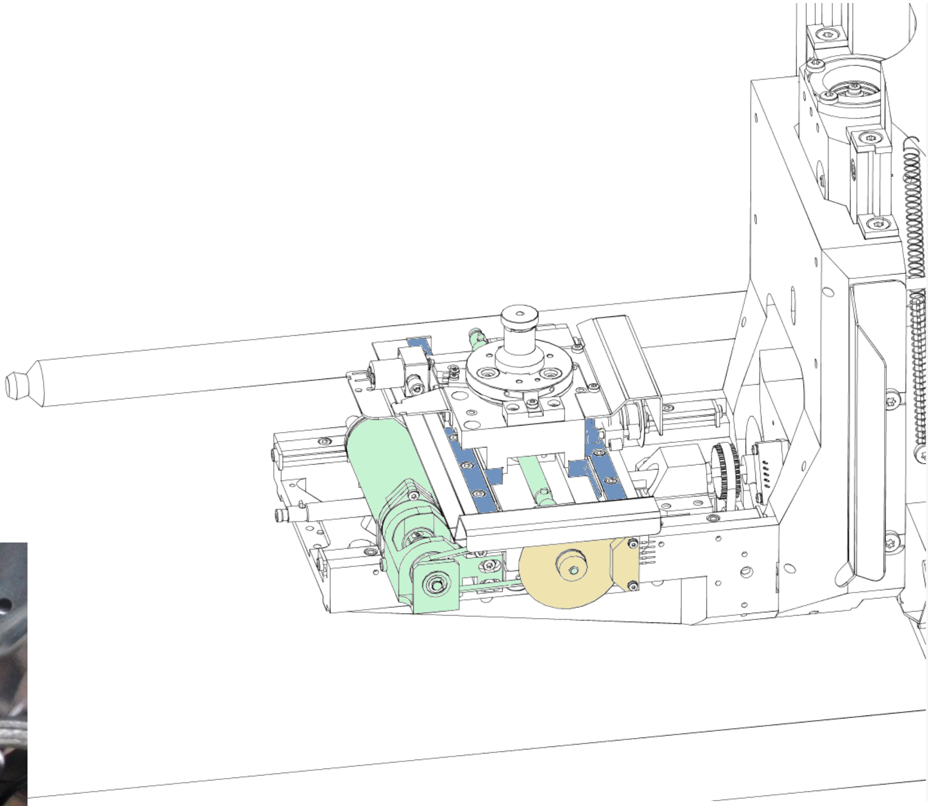
SEM stages spindle drive

Spindle & nut, rotary motor

- Accuracy <5um
- Repeatability <2um
- Drift <20nm/min

- Simple
- **Not so expensive**





SEM stages spindle driven

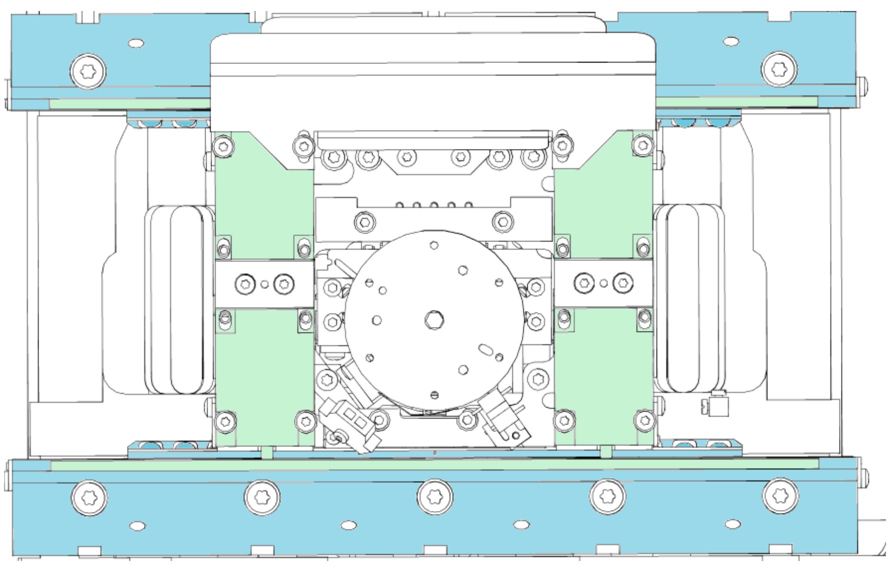
But there are design chalenges

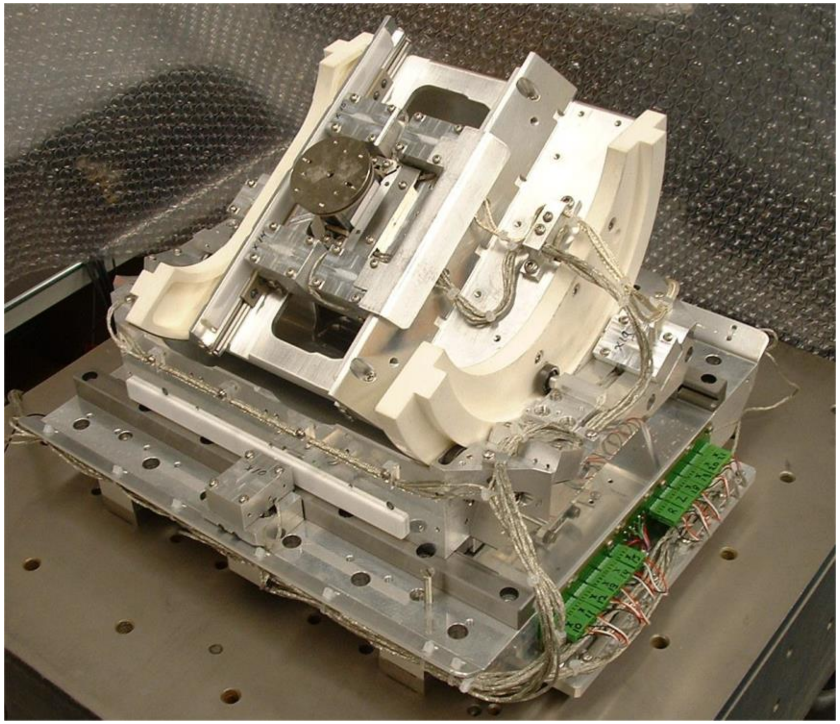
- Relatively lower stiffness
- Lower axis acceleration and speed
- Motor shielding
- Remove play in gears

SEM stages piezo driven

Direct piezo drive

- Accuracy <0.5um
- Repeatability at smallest step level (with linear encoders)
- Drift <8nm/min
- **For highend systems**
- Great stiffness and dynamics
- No play
- Easy to be non magnetic
- Pricey



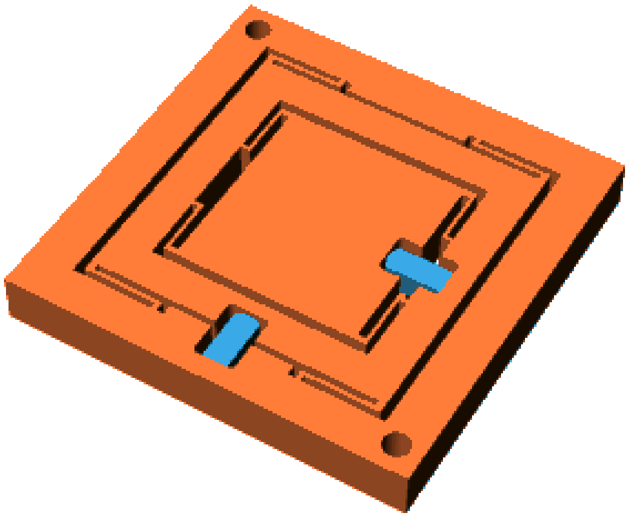


SEM stages substages

Piezo driven flexures

- Flexible joints only – no friction at all
- Piezo stacks
- Subnanometer steps
- Substage or for AFM

http://www.physikinstrumente.com/products/xyz-nanopositioning-stages.html



http://www.piezostage.net/

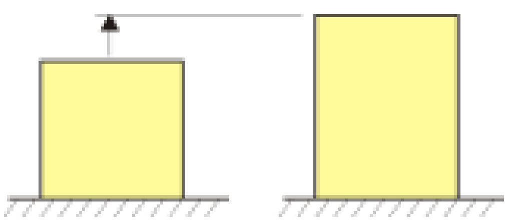
Piezo motor principles

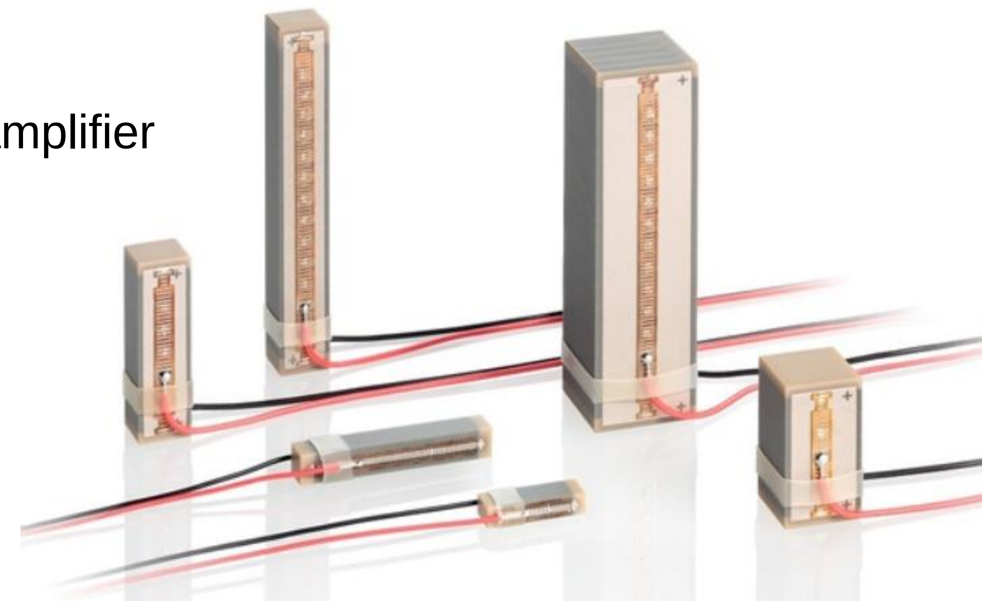
Indirect piezoelectric effect – change of shape of a crystal by voltage applied to it

Piezo stack

- Stacked piezo crystals – direct actuation
- Most accurate
  - DC voltage
  - Depends on quality of drive amplifier
- Micrometer ranges

$$\Delta L = n * d_{33} * V$$



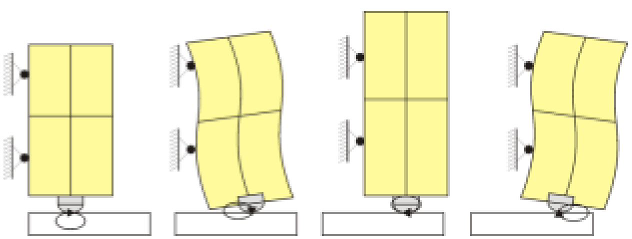


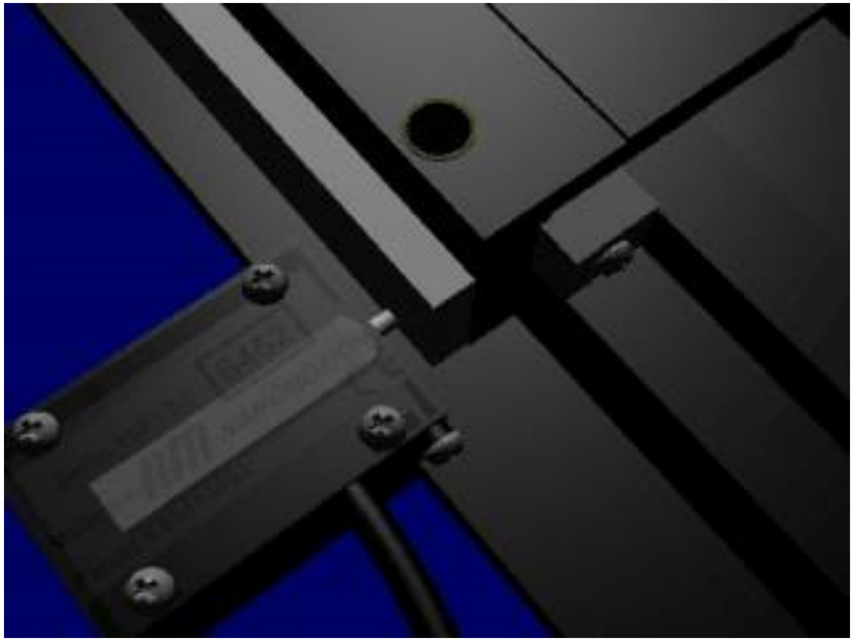
https://www.physikinstrumente.com/en/products/piezoceramic-actuators/linear-actuators/p-882-p-888-picma-stack-multilayer-piezo-actuators-100810/

Piezo motor principles

Ultrasonic

- 40kHz AC excitation, 300V_{RMS}
- High speed - meters per second
- Discrete steps with voltage period ±100nm





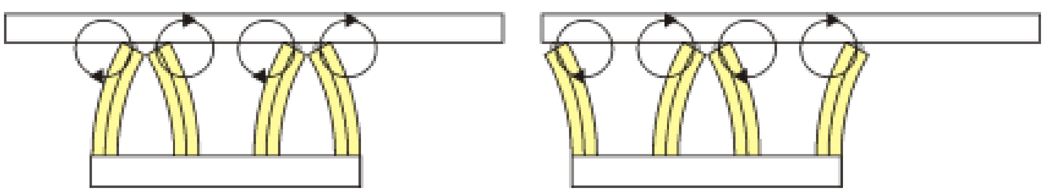
http://www.nanomotion.com/support-downloads/product-videos/

Piezo motor principles

Walking leg

- Equiwalent of a stanfard stepper – including microstepping ±1nm
- Lower speeds
- Low wear

https://www.youtube.com/watch?v=Z45PWSGpc8A

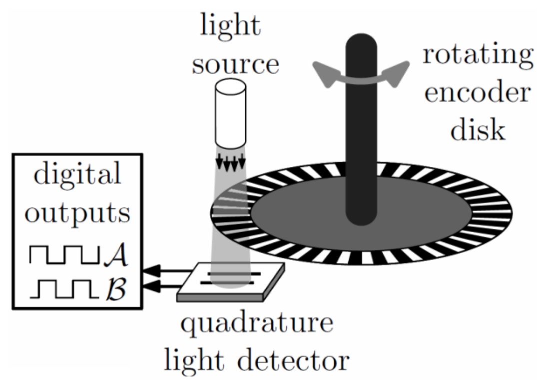


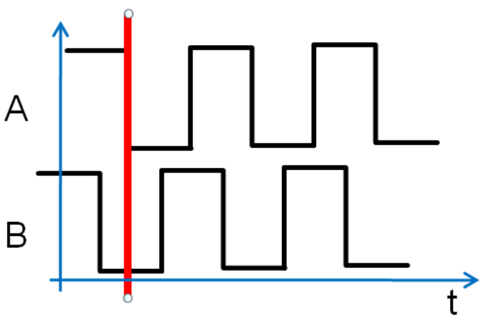
Position feedback - encoders

Optical encoders

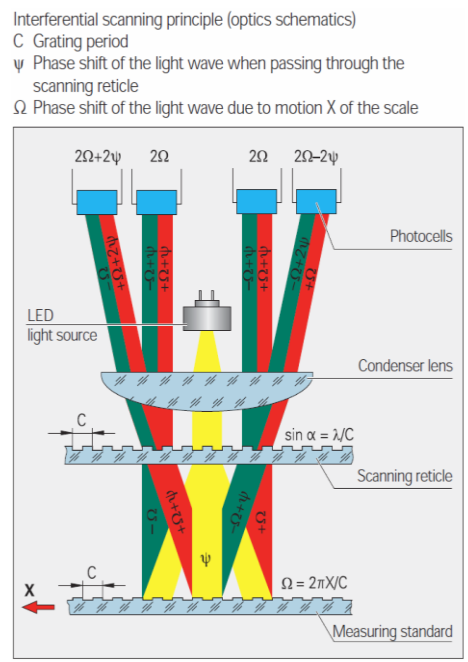
Incremental or absolute

Rottary



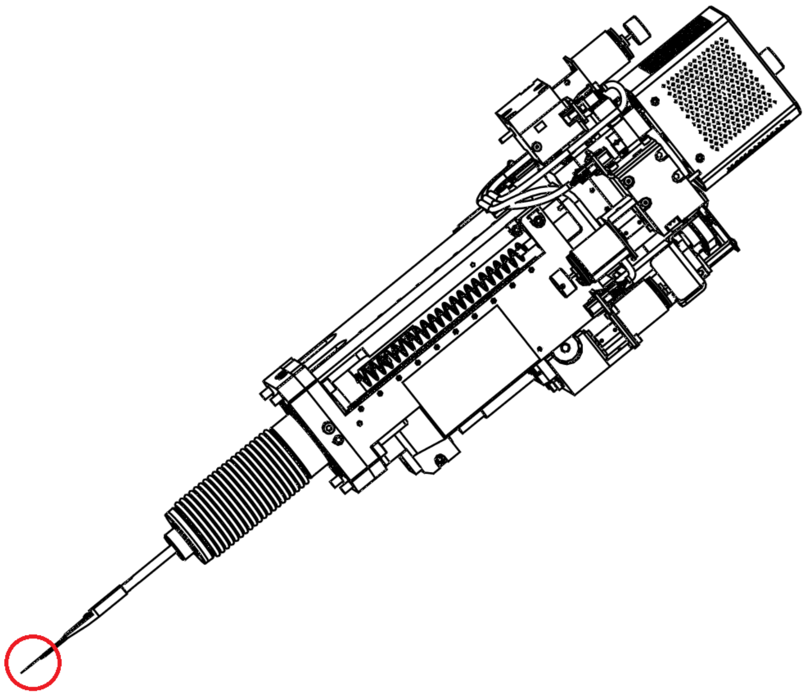


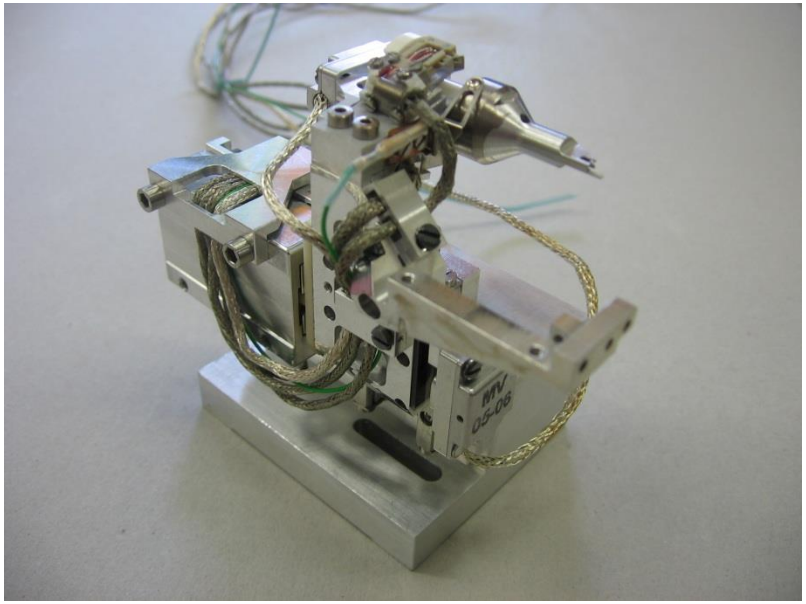
Linear - interferometric



TEM sample preparation

Extraction of a lamella from a bulk sample using SEM/FIB





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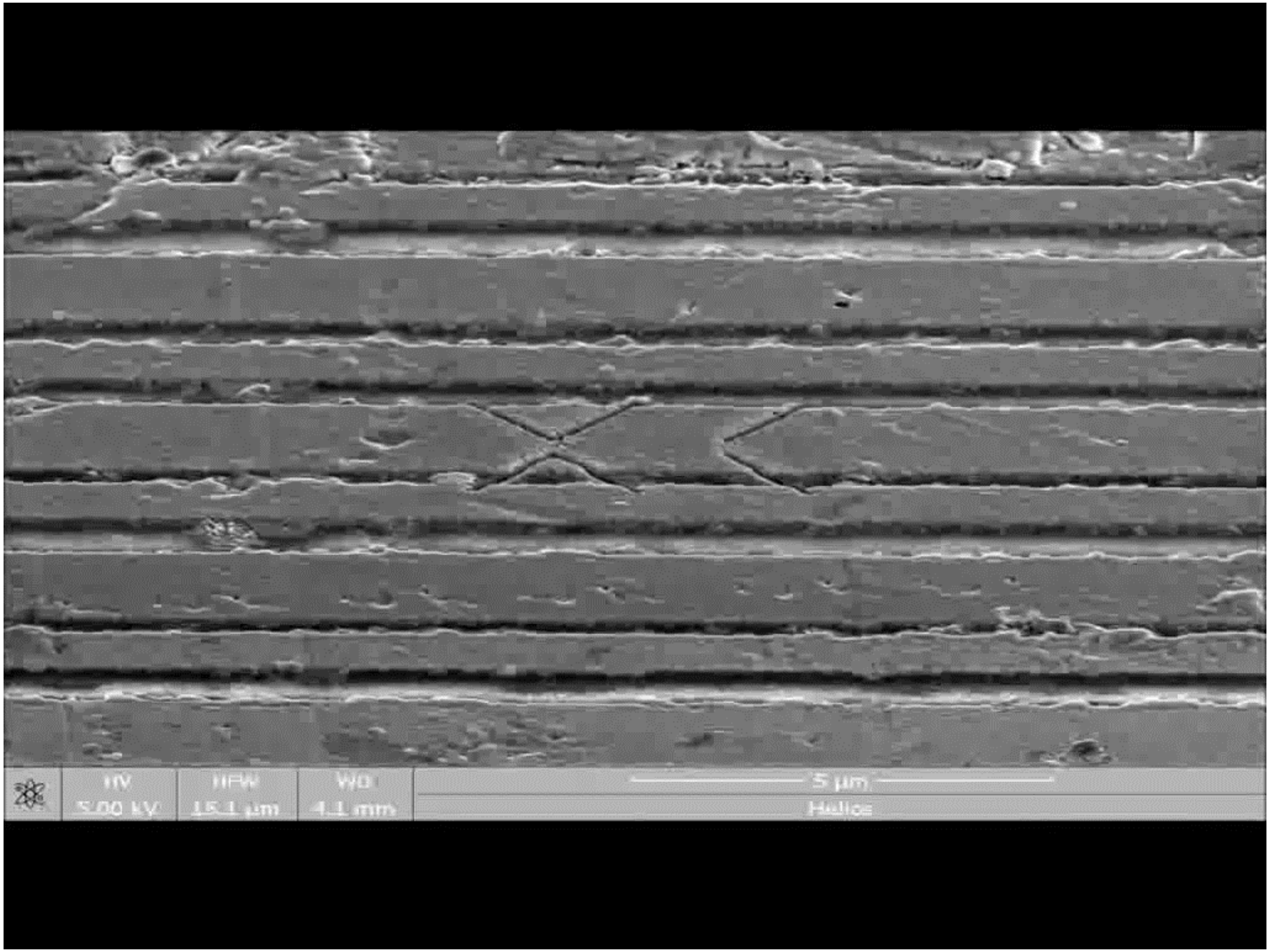
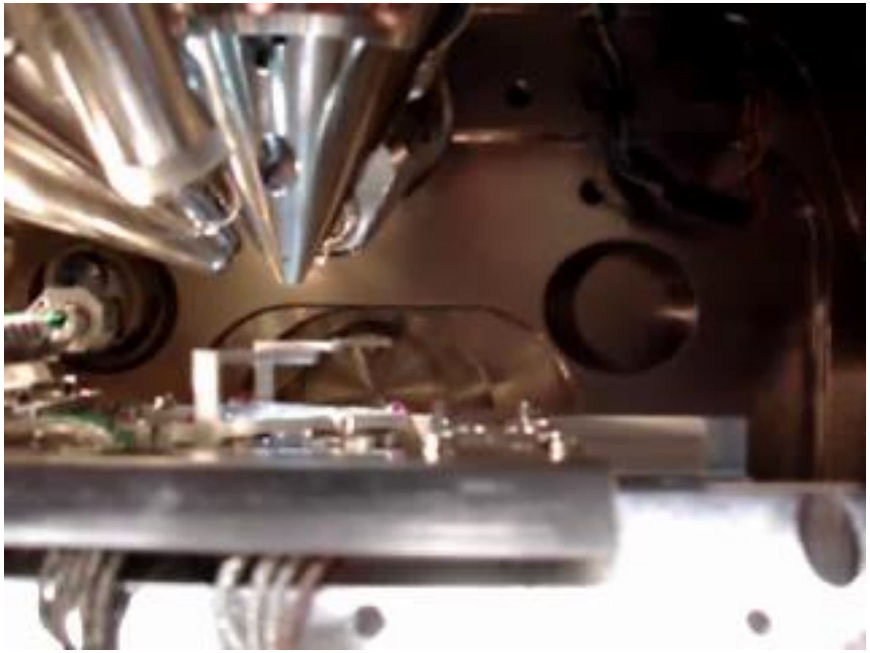
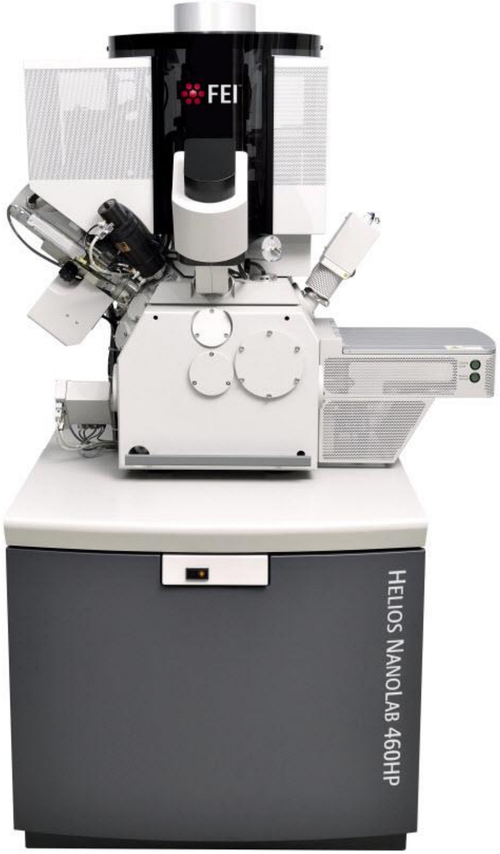


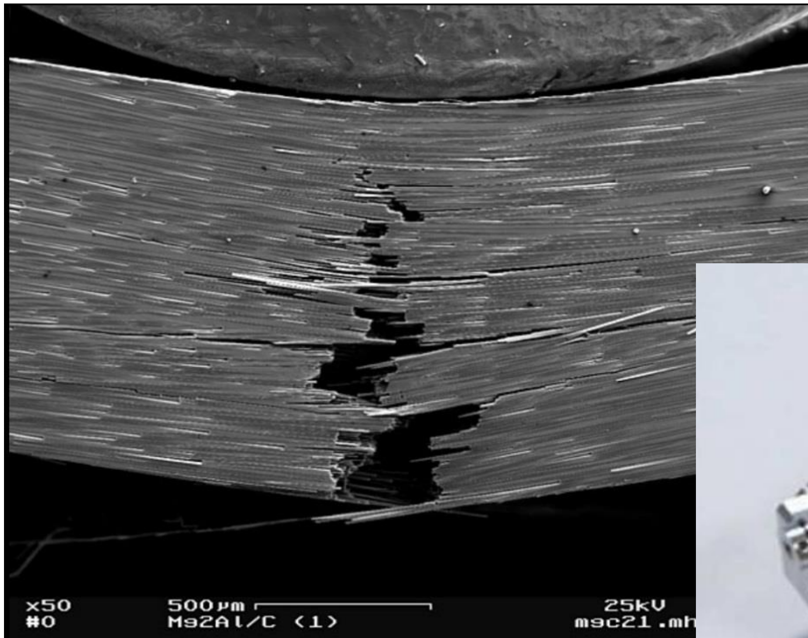
Additional equipment – loadlock

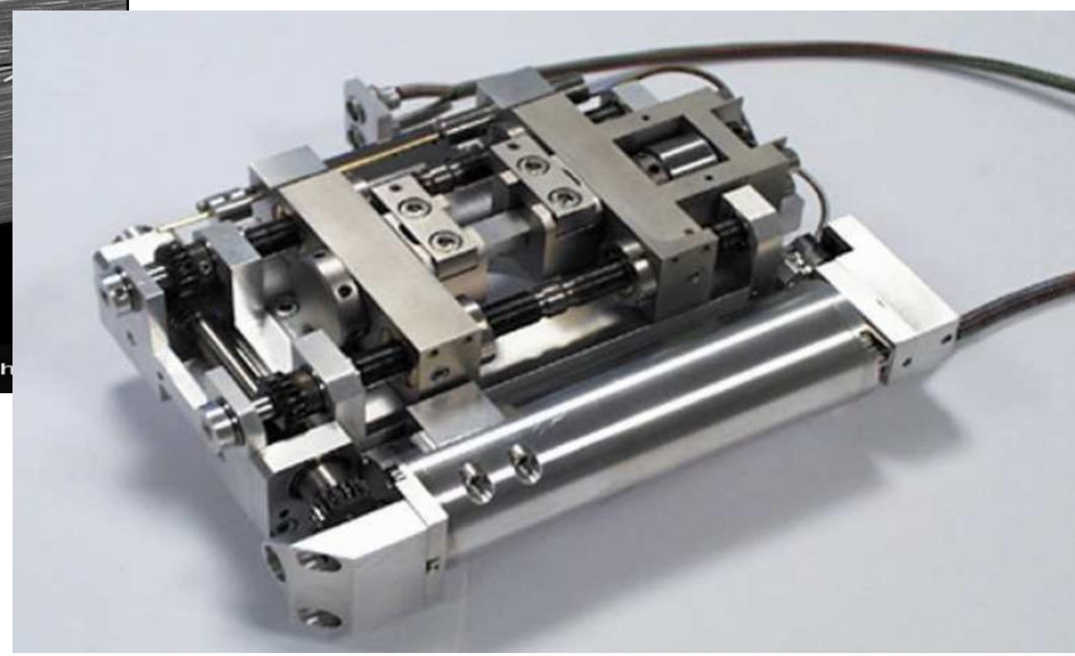
- Shortening pumping cycle
- Reducing vacuum contamination





Additional equipment – Mechanical material testing





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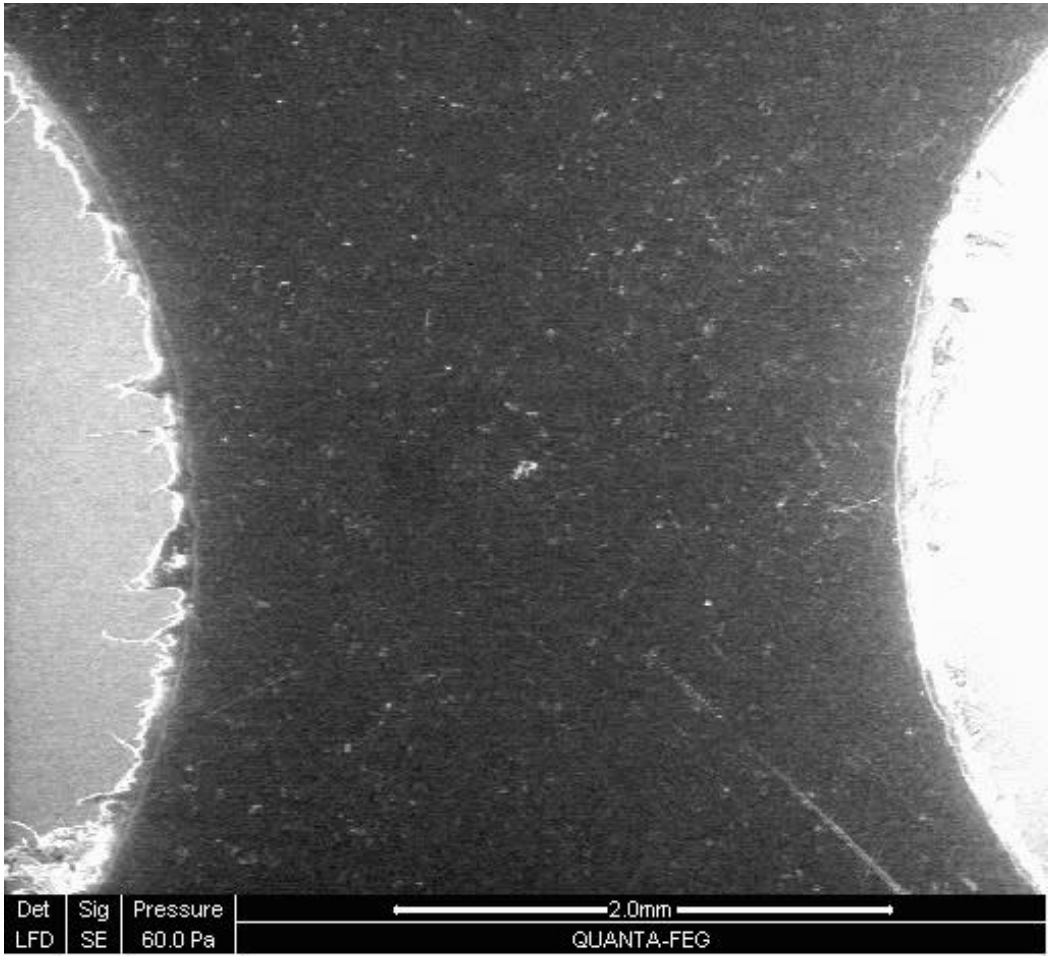
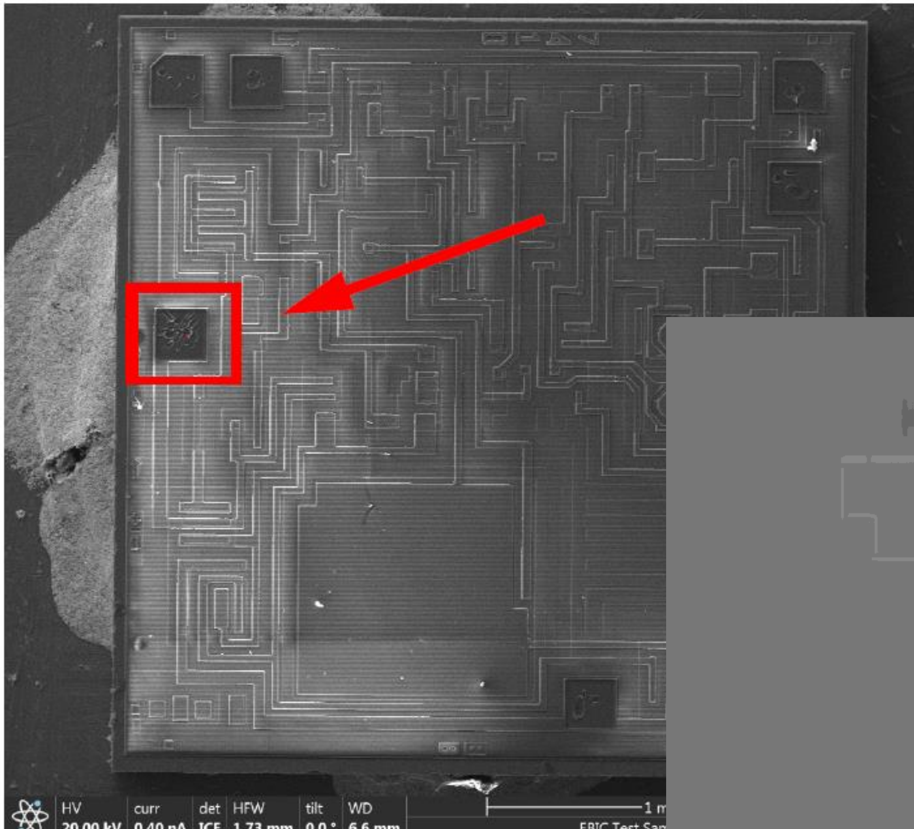
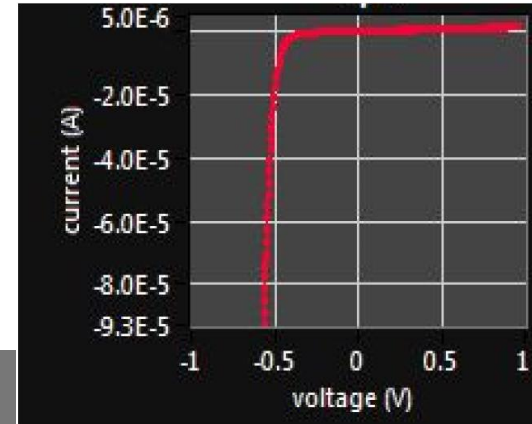


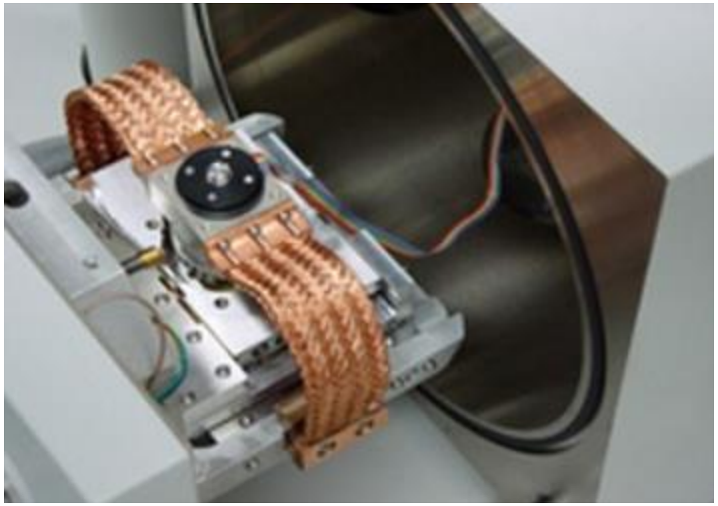
Additional equipment – Electrical failure analysis

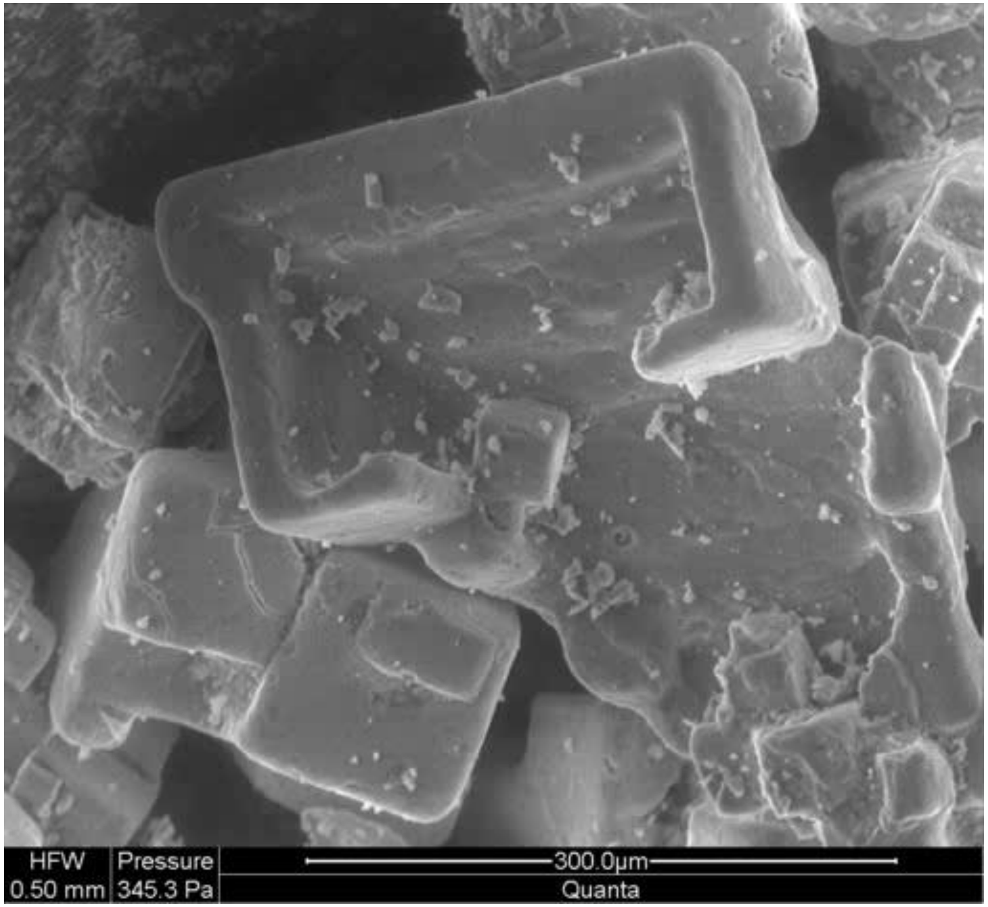




Additional equipment – cold stage

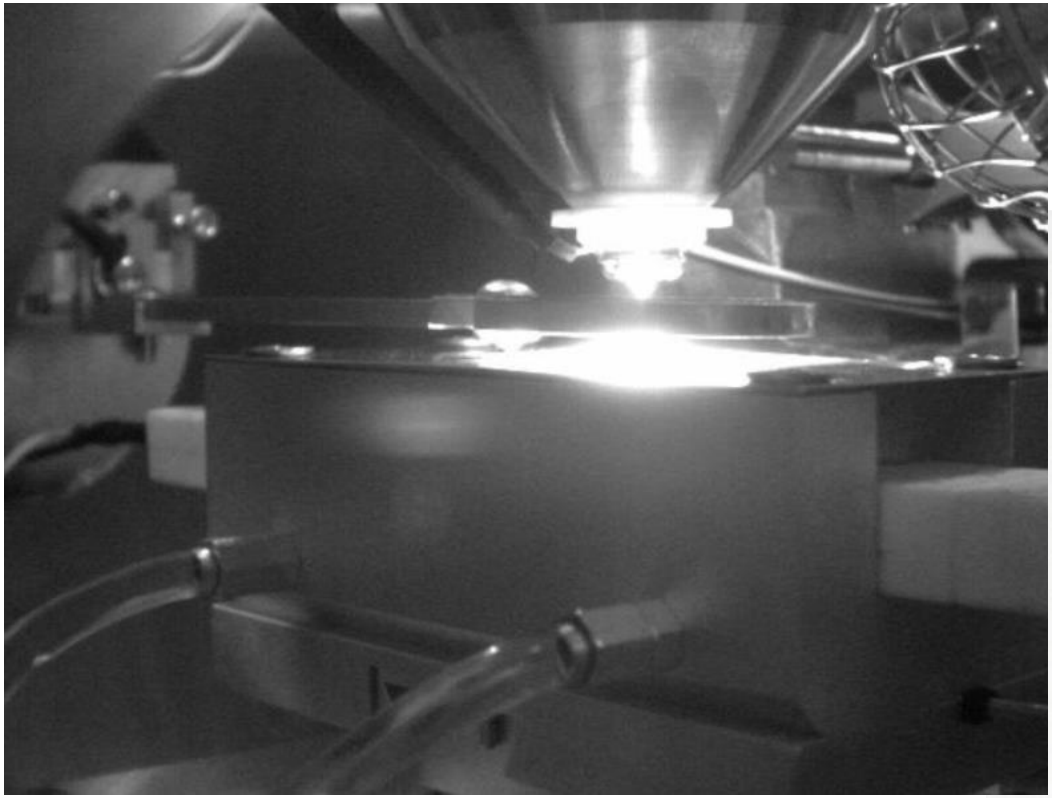
- In-situ observation of thermal processes
- Peltier stage > -100°C
- Cryo N2 >-190°C – biology

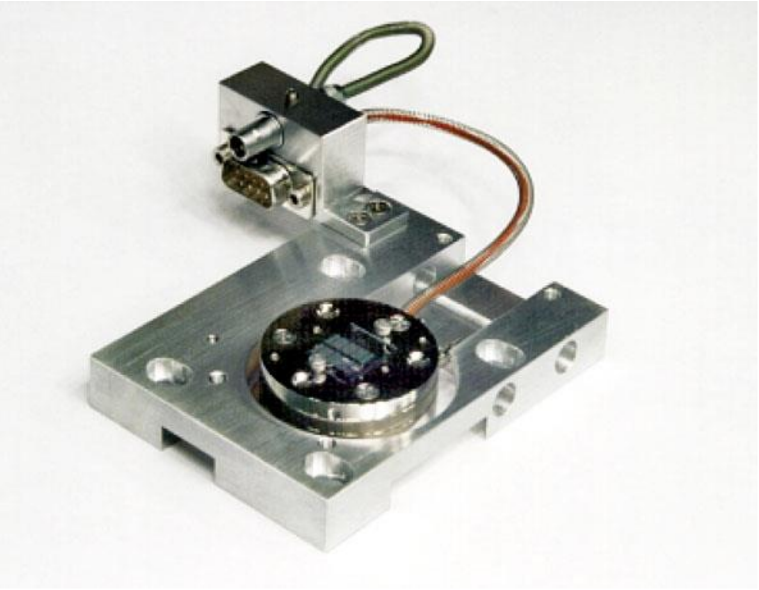




Additional equipment – hot stage

- Low temperature < 400°C
- Mid temperature <1000°C – shielding needed from thermal electrons
- High temperature <1400°C – special material requirements, lifetime in hours





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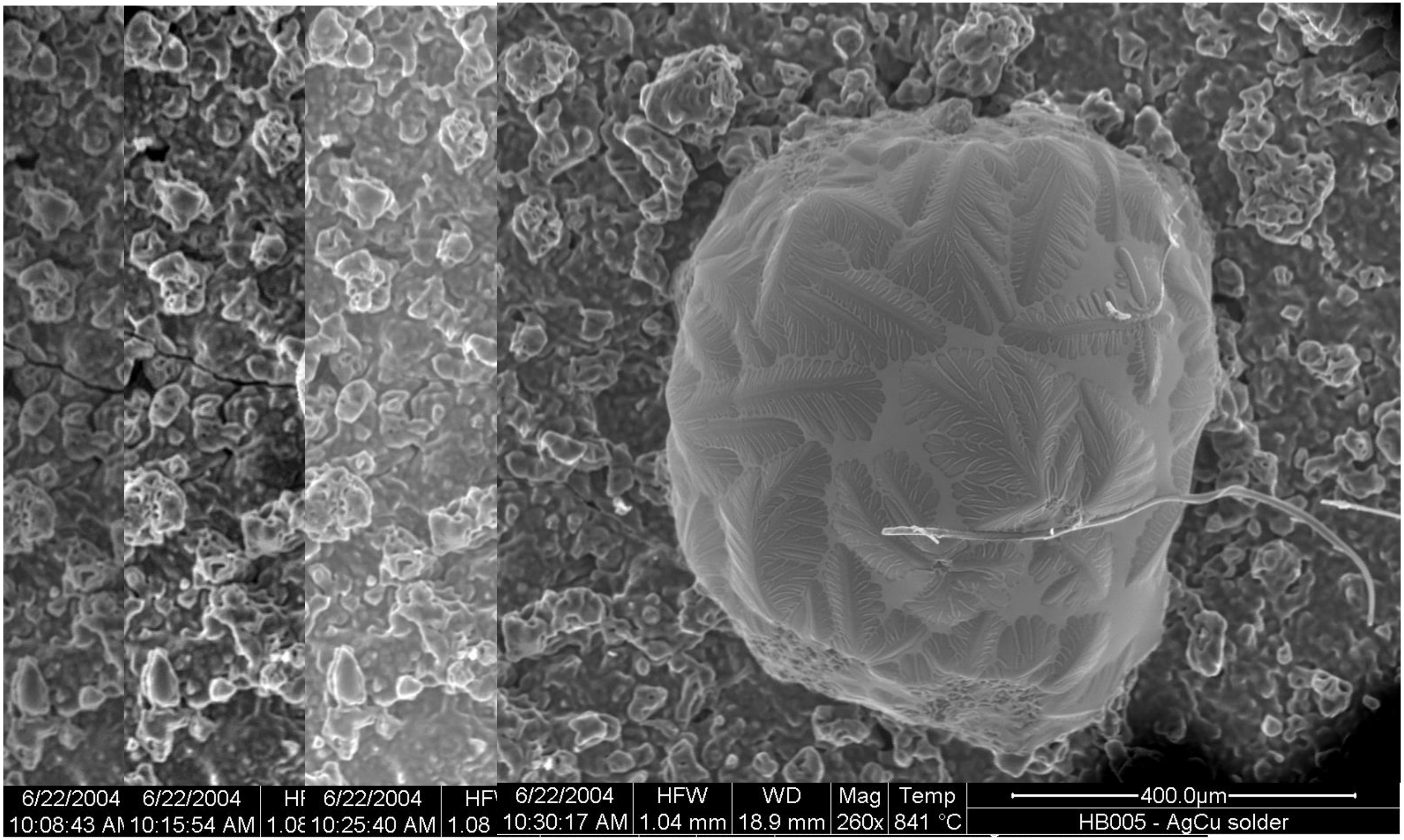

Melting and recrystallization of AgCu solder