Neutron Imaging - Science Drivers

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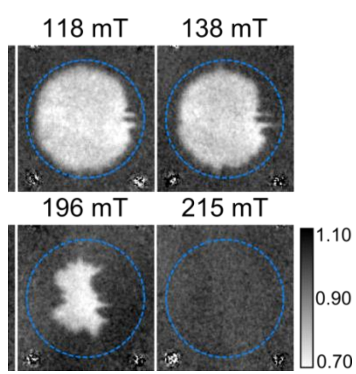
20km north: FRM II, Garching

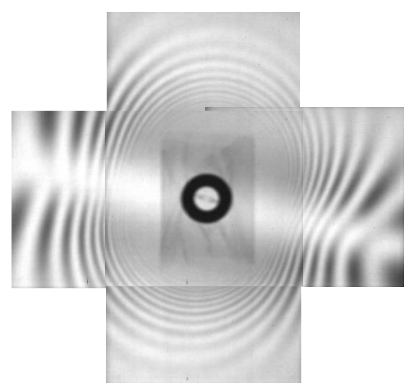
Munich Neutron Imaging Group (MUNIG)

Some words about myself...

- Physics Degree in Munich
- PhD in Munich on Imaging with polarized neutrons
- Postdoc as Instrument Scientist at ANTARES, Munich
- Since 2014 head of Neutron Imaging Group (MUNIG) at FRM II
- We operate 2 neutron imaging beam lines (ANTARES + NECTAR) + contribute to the design and construction of ODIN@ESS
- Personal (scientific) interests:
  - Novel and advanced instrumentation (interferometry, polarized neutrons)
  - Applications in magnetism and superconductivity

ANTARES
NEUTRON IMAGING



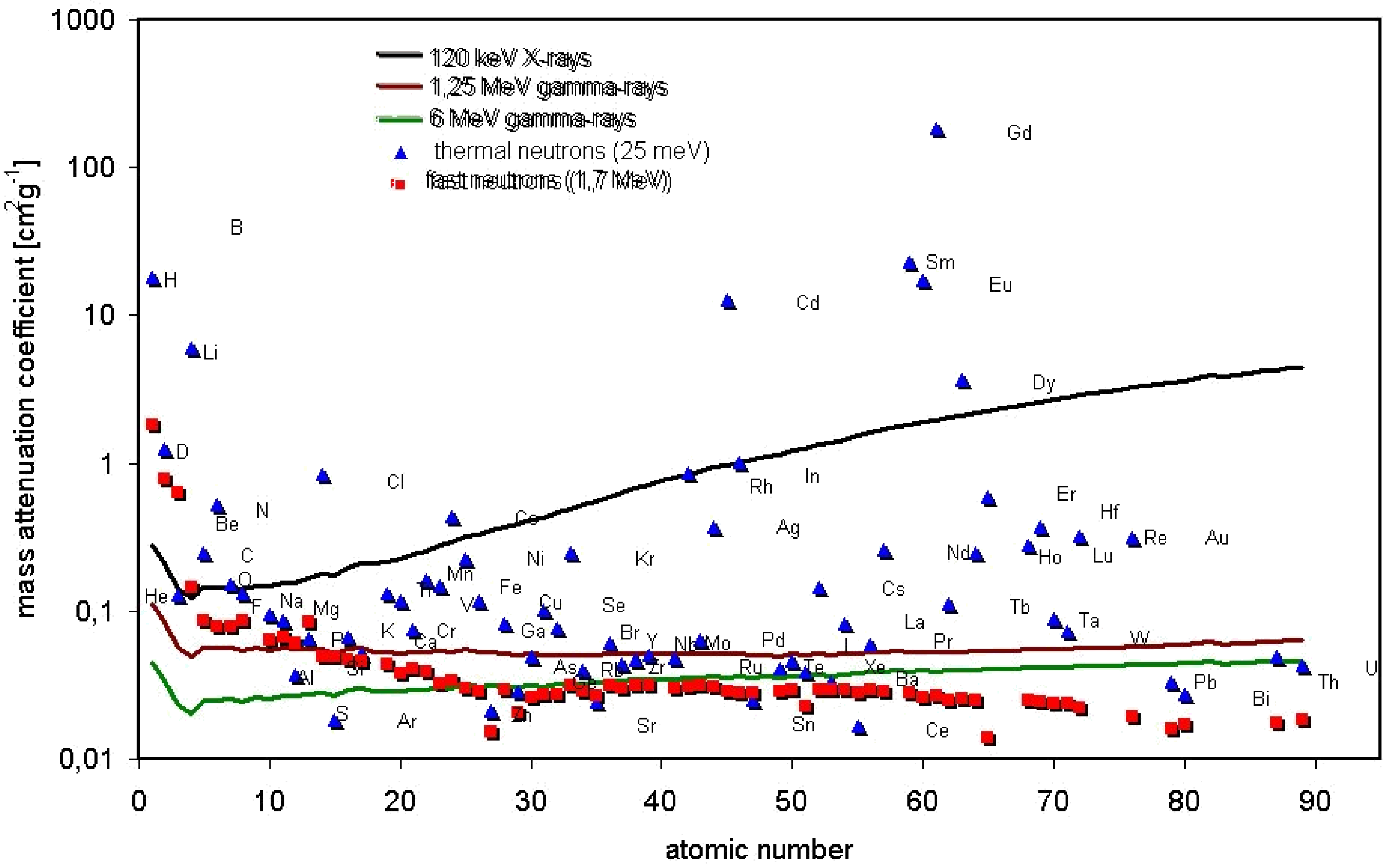


FAST NEUTRON IMAGING
NECTAR

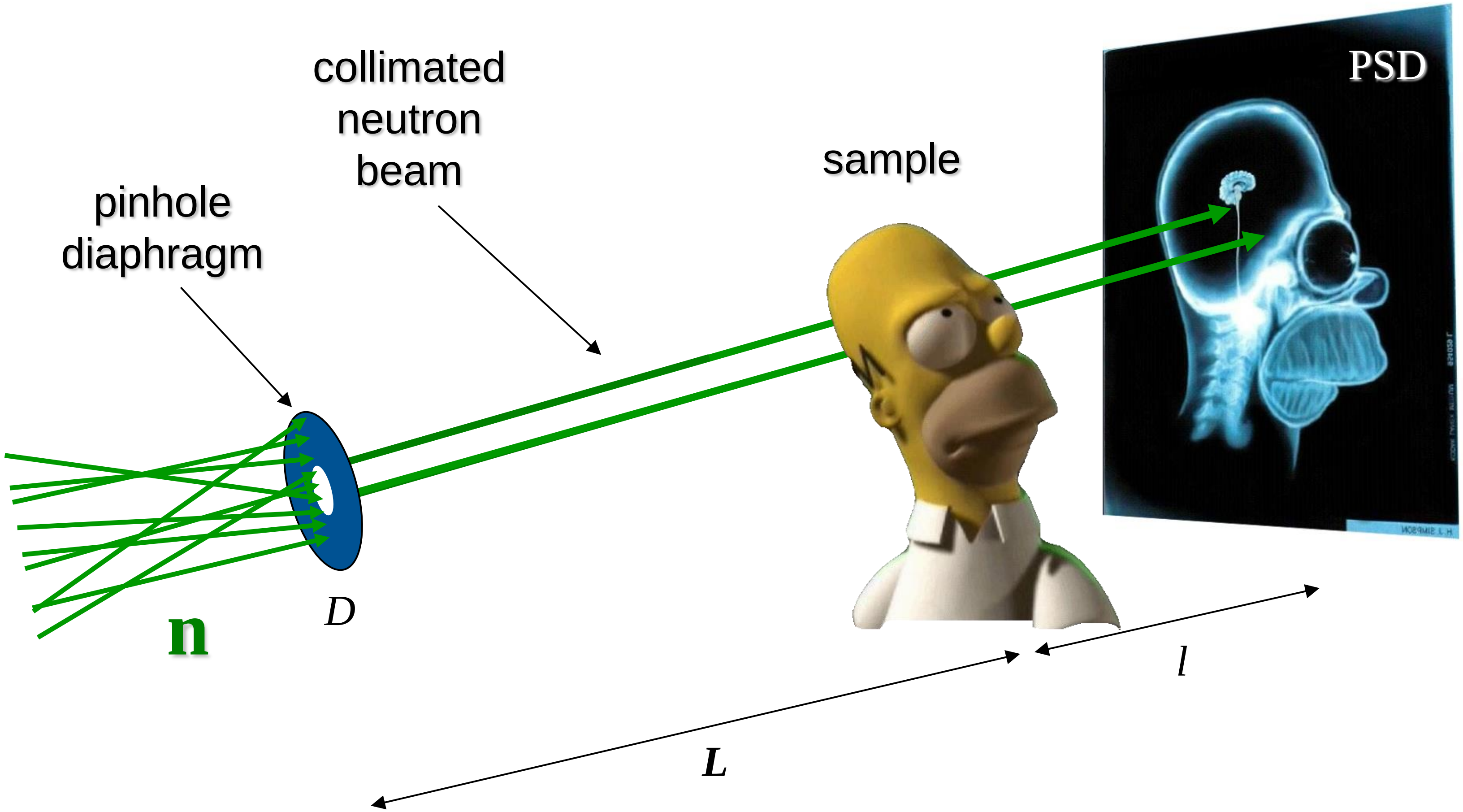
Outline

- Fundamental principles of neutron imaging
- Selected scientific applications
  - Archaeology
  - Polarized neutron imaging
  - Energy storage
  - Flux line lattices in superconductors
- Conclusion

Comparison neutrons & x-rays



The Principle of Neutron Imaging



Analytical description of the transmission process

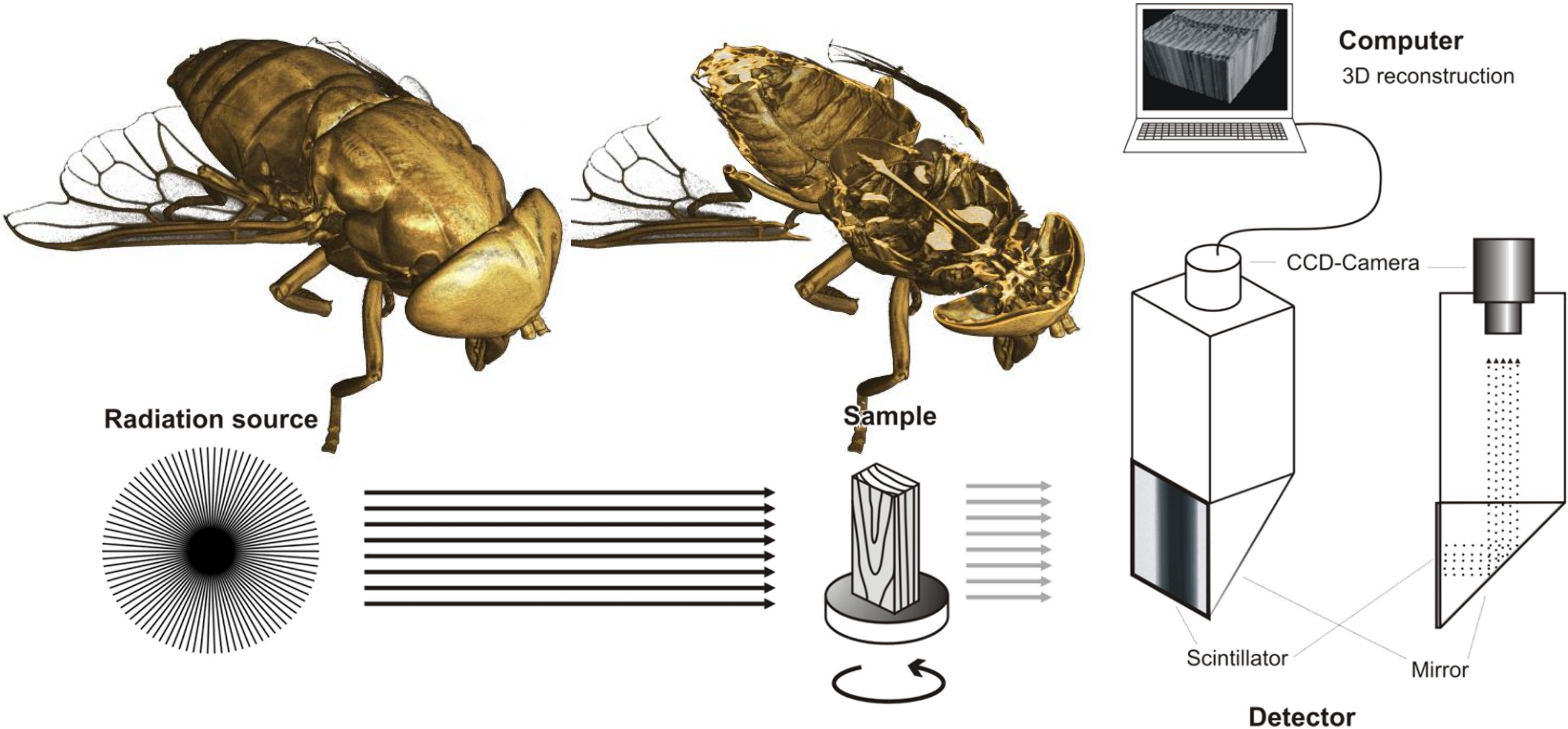
Transmission **Beer-Lambert law**

$$T = \frac{I}{I_0} = e^{-\Sigma \cdot d} = e^{-\sigma \cdot N \cdot d}$$

and inverted …

$$\Sigma \cdot d = \ln\left(\frac{I_0}{I}\right)$$

Neutron tomography →3D information



- Several hundred single projections are required
- A reconstruction algorithm delivers the 3D structural data
- A visualization tool delivers slices and views at arbitrary positions

Courtesy of E. Lehmann, PSI

Why / when neutron imaging

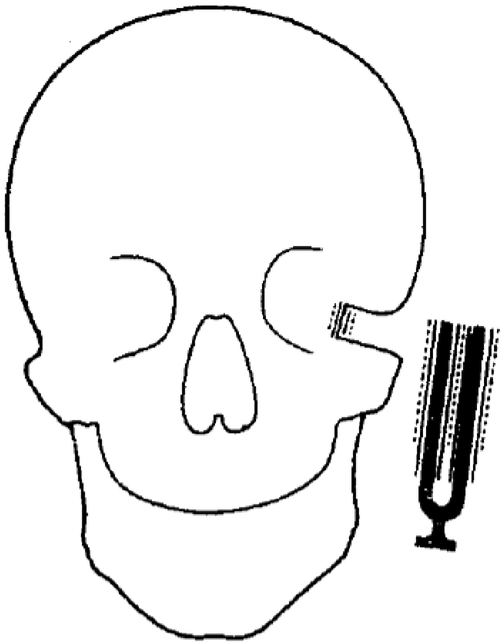
- Neutron Imaging is expensive!
- It will always be a niche application
- If there is another way to investigate your sample -> go for it!
- Not suitable for series inspection in fabrication processes
- Only some specimens can be inspected as representatives for a series

BUT:

In some case you can only see what you want with neutrons!

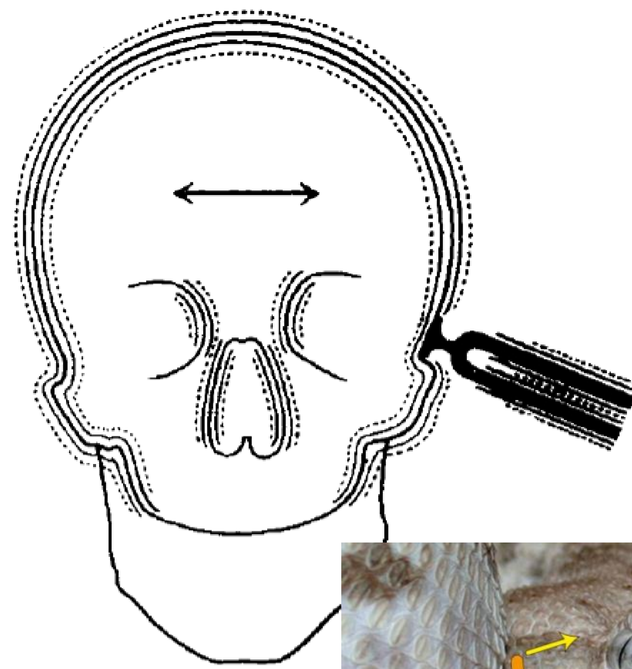
Tympanic Hearing and bone-conduction hearing

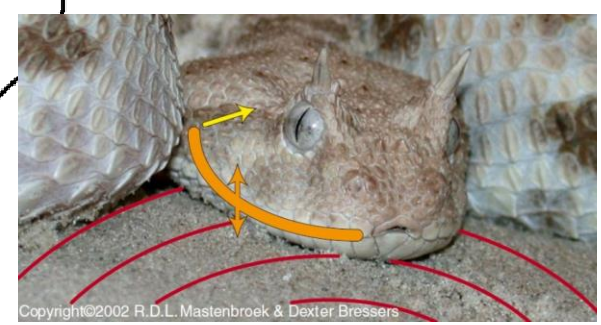
Hearing with tympanic membrane and middle ear bones



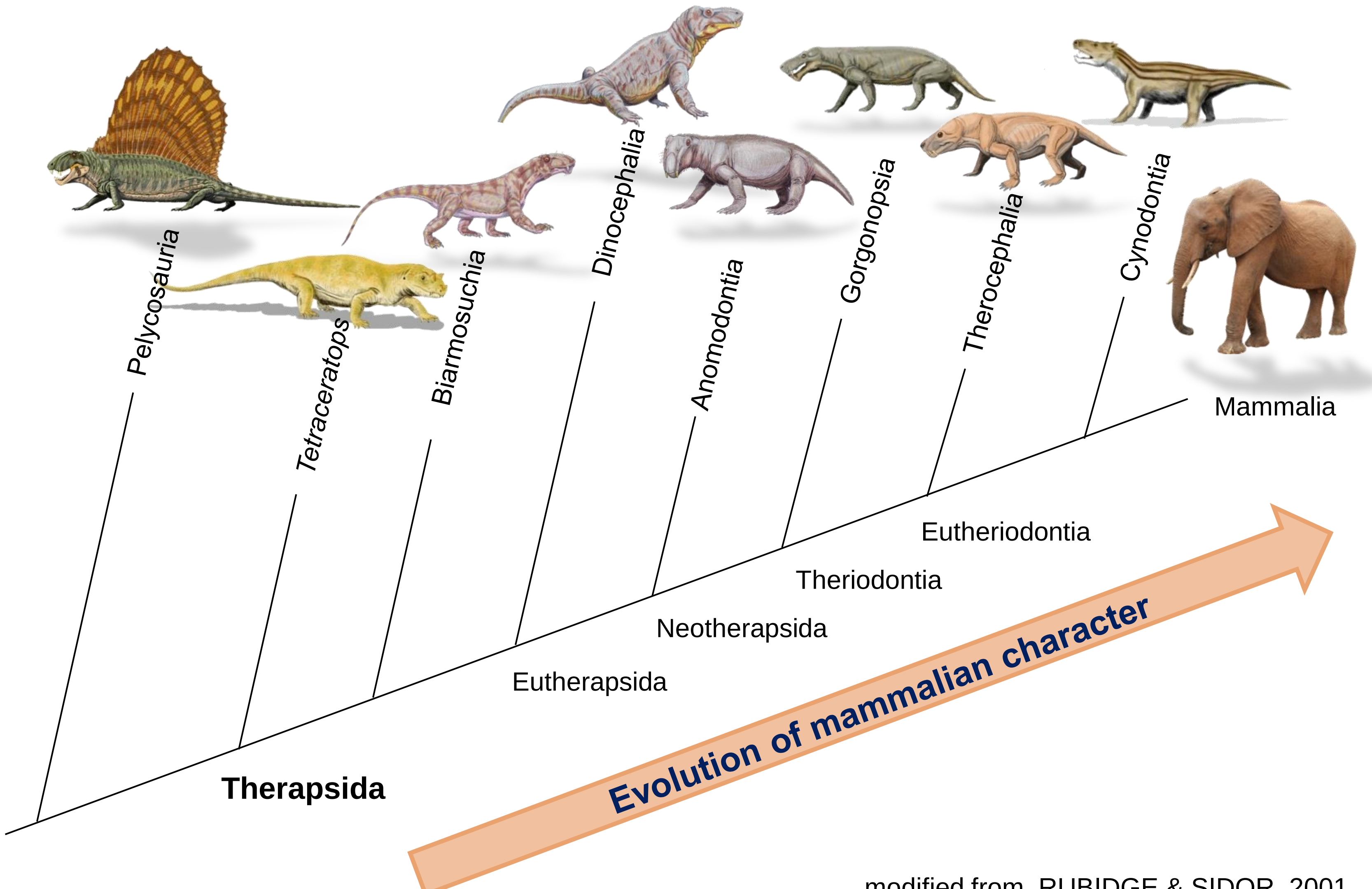
from Tumarkin 1968

Hearing by bone conduction



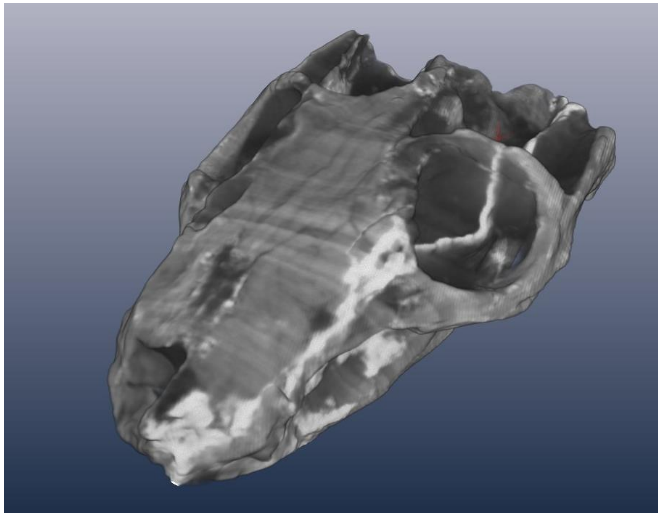


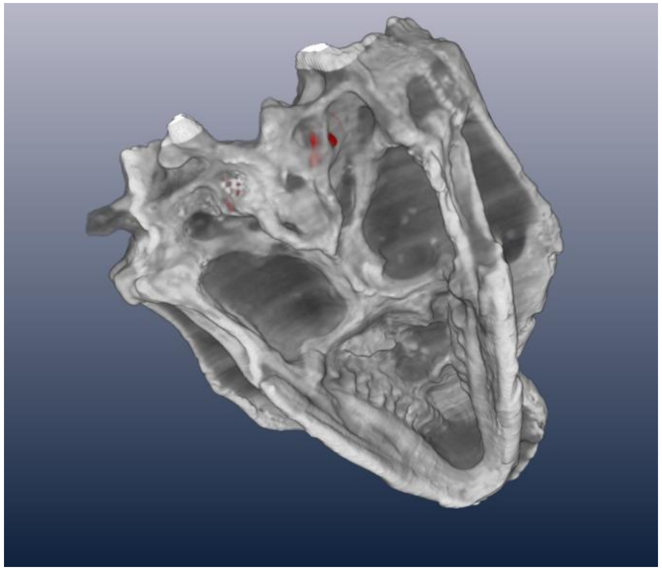
Synapsid evolution



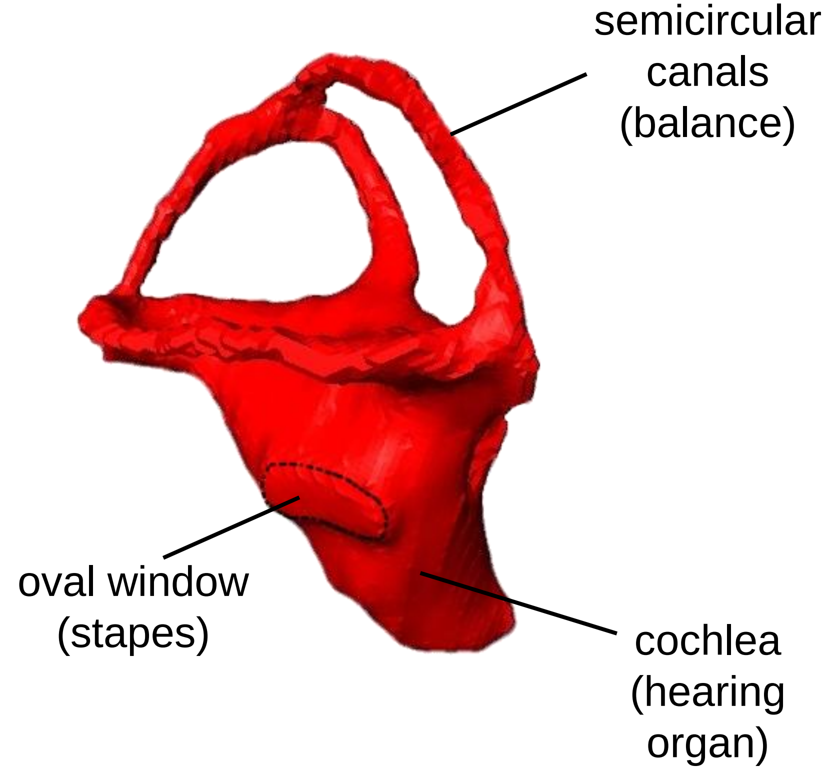
modified from RUBIDGE & SIDOR, 2001

The origin of tympanic hearing





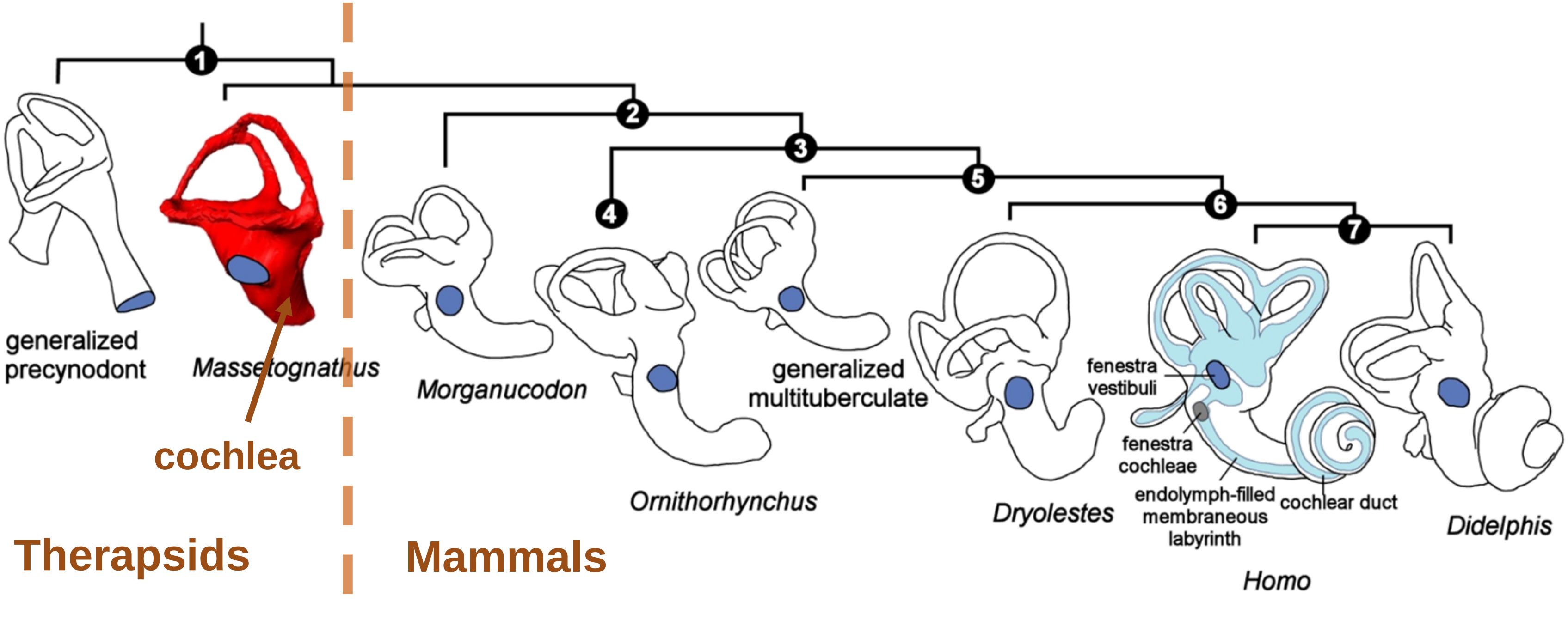
Massetognathus (Cynodontia), approx. 230 million years old



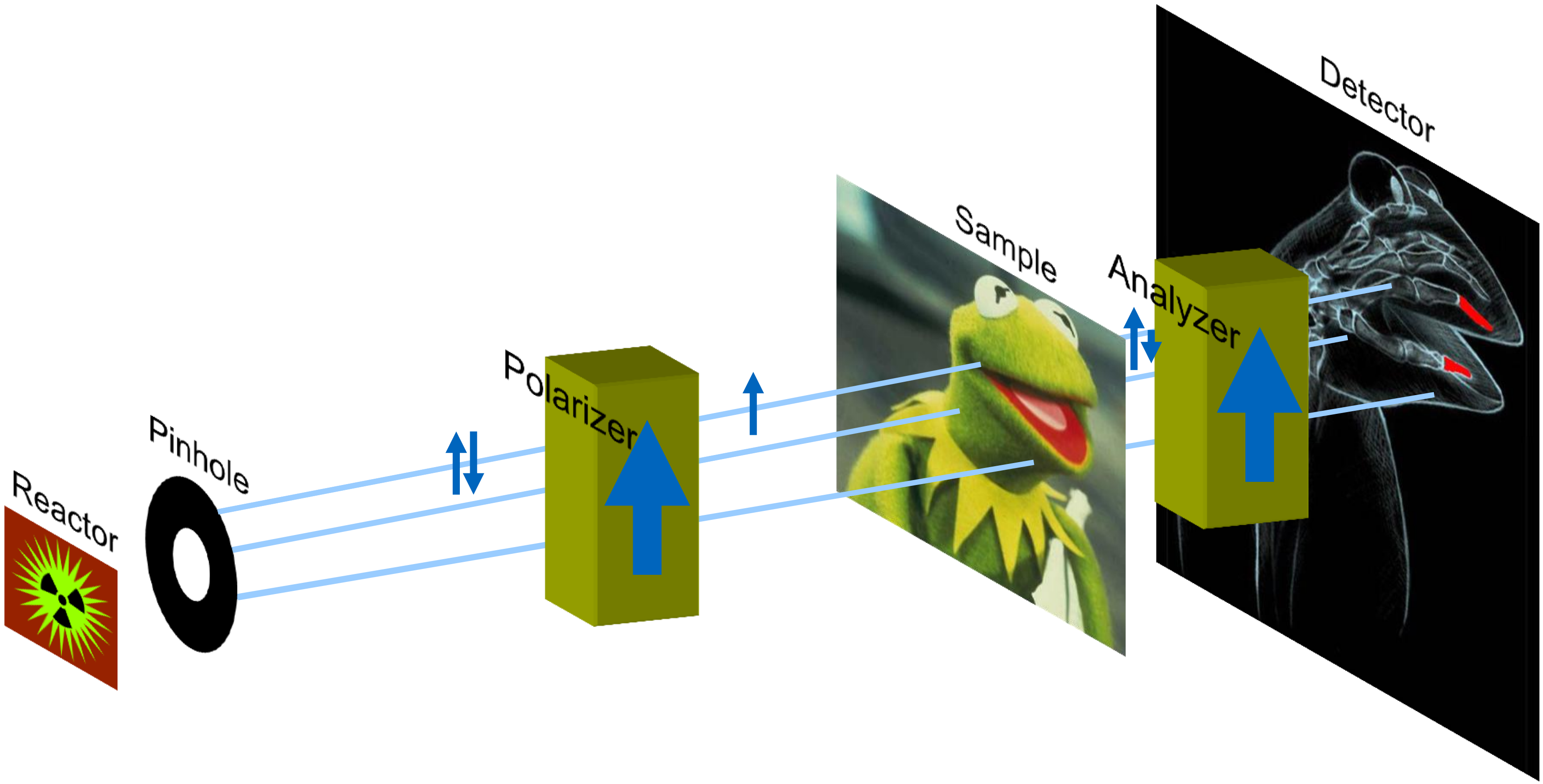
inner ear of *Massetognathus*

The origin of tympanic hearing

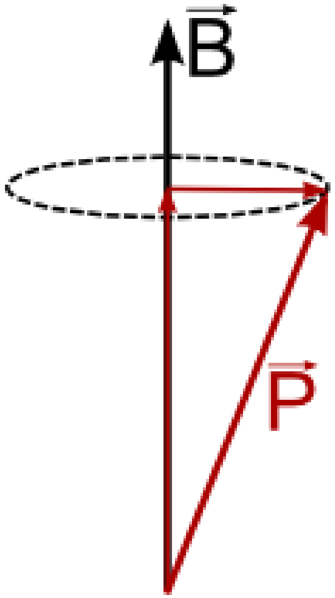
- short, tube-like cochlea in the cynodont therapsid *Massetognathus*
- 3,9 mm long
- enhanced sensitivity to high-frequency air-borne sound
- small stapedial footplate area (1,69 mm^2)

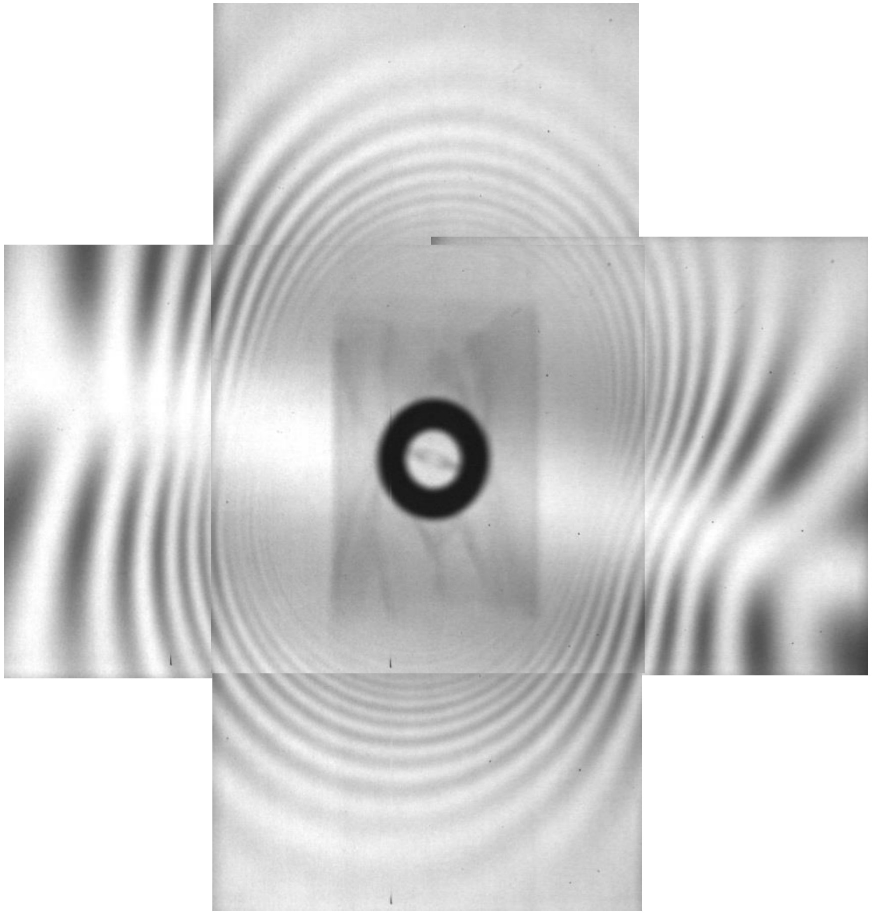


More complex: Polarized Neutron Imaging

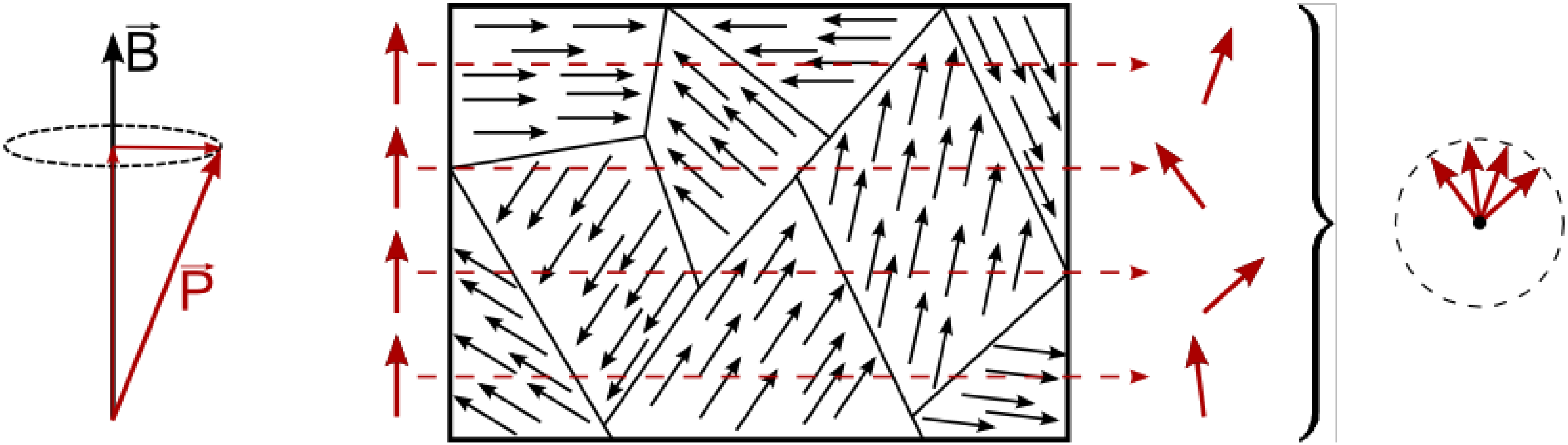


Stray Field of Ring Magnet



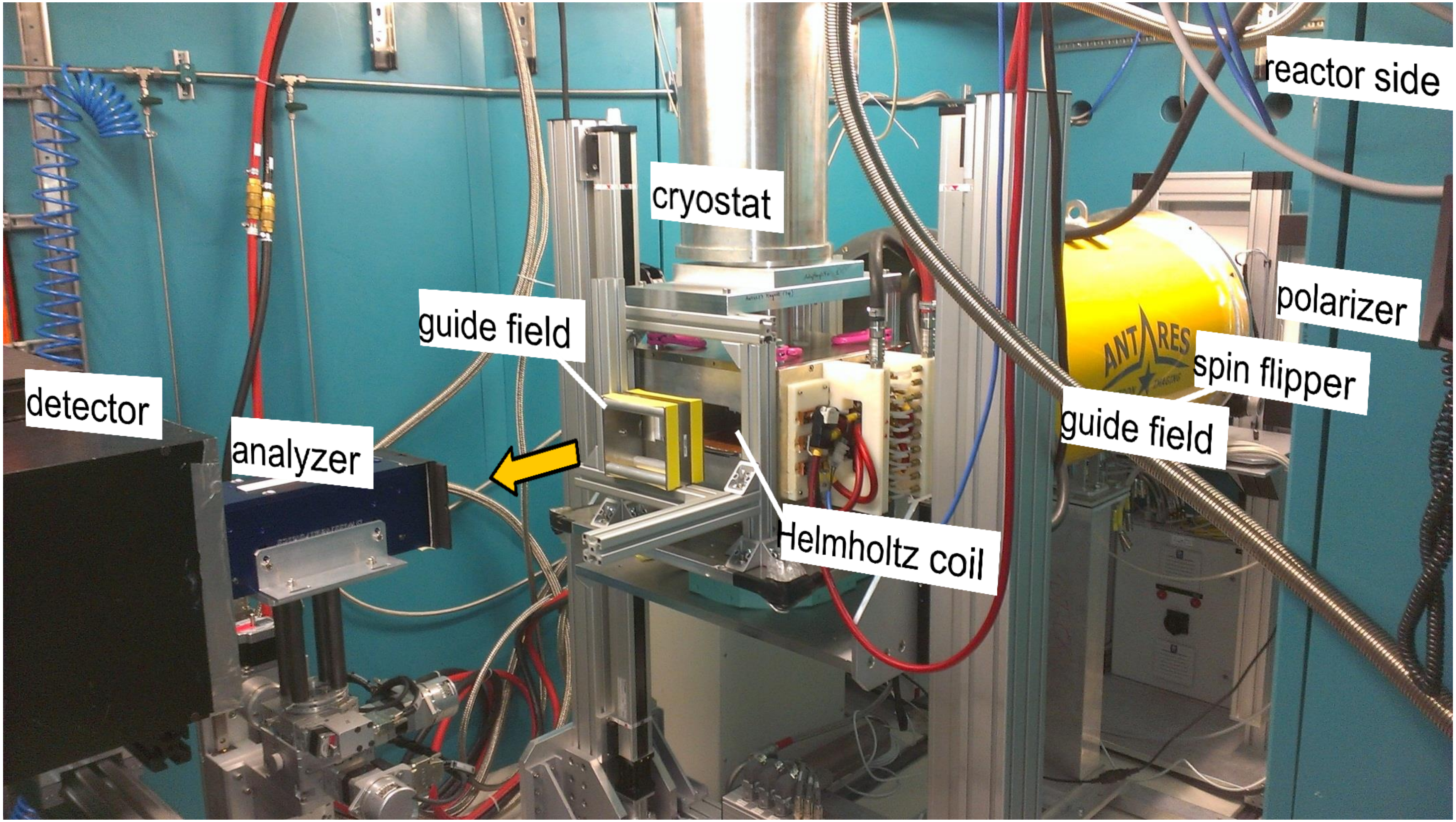


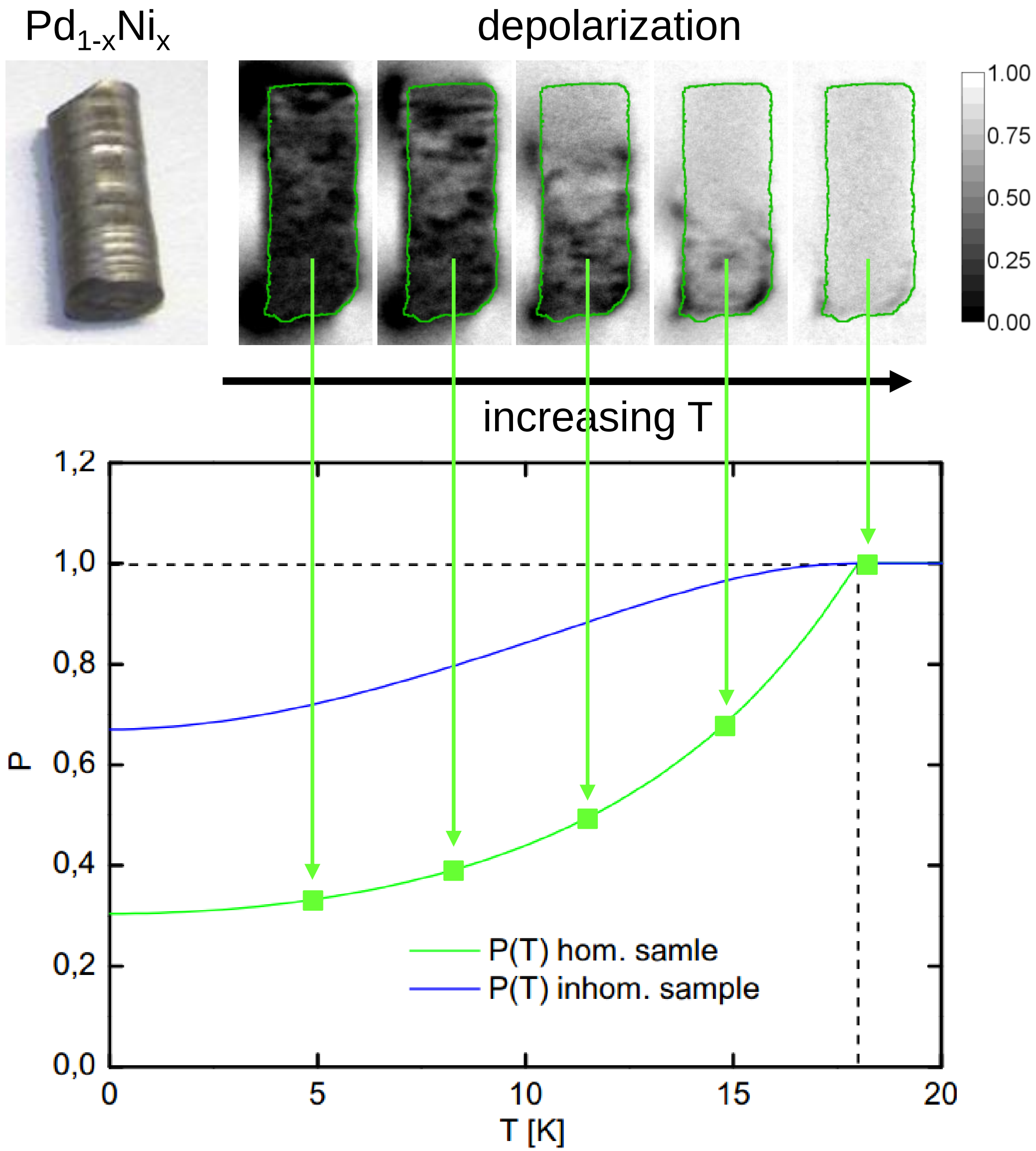
Neutron Depolarization in Ferromagnets

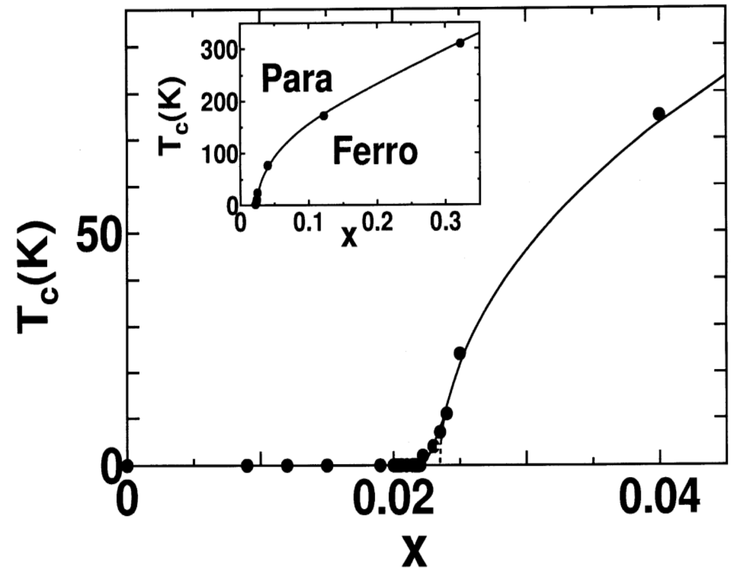


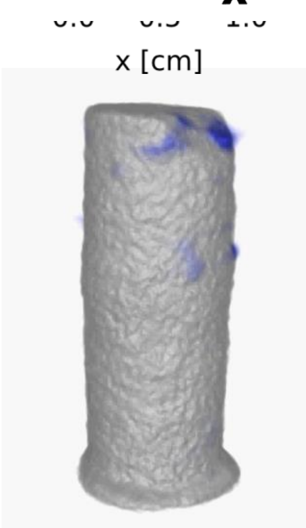
$$P = P_0 \exp\left(-\frac{1}{3}\gamma^2 \mu_0^2 M^2 \frac{d\delta}{v^2}\right)$$

Setup for Depolarisation Imaging









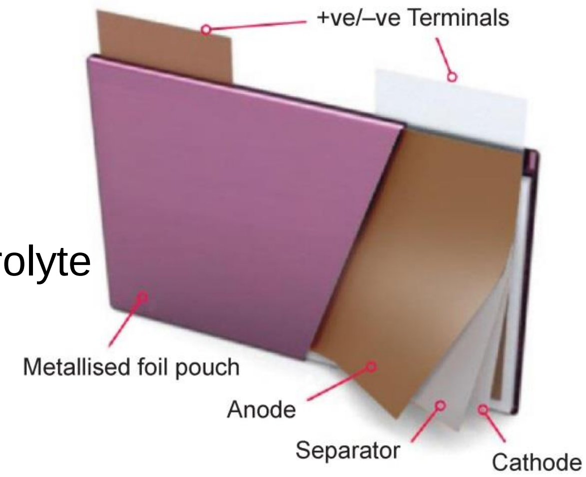
T = 8K

M. Schulz, *et.al*.
J.Phys. Conf., **211** (2010) 012025

In-situ filling of Li-ion Pouch Batteries

Pouch Batteries

- High potential for electro mobility and stationary energy storage
- Electrolyte filling is a key process in cell production
- So far only limited knowledge about the process
- Phenomenological: pressure cycles to optimize wetting with electrolyte



Why Neutron Imaging?

- Cell housing optically intransparent
- Other approaches not successful
- Neutrons offer high contrast due to H-content in electrolyte

Goals

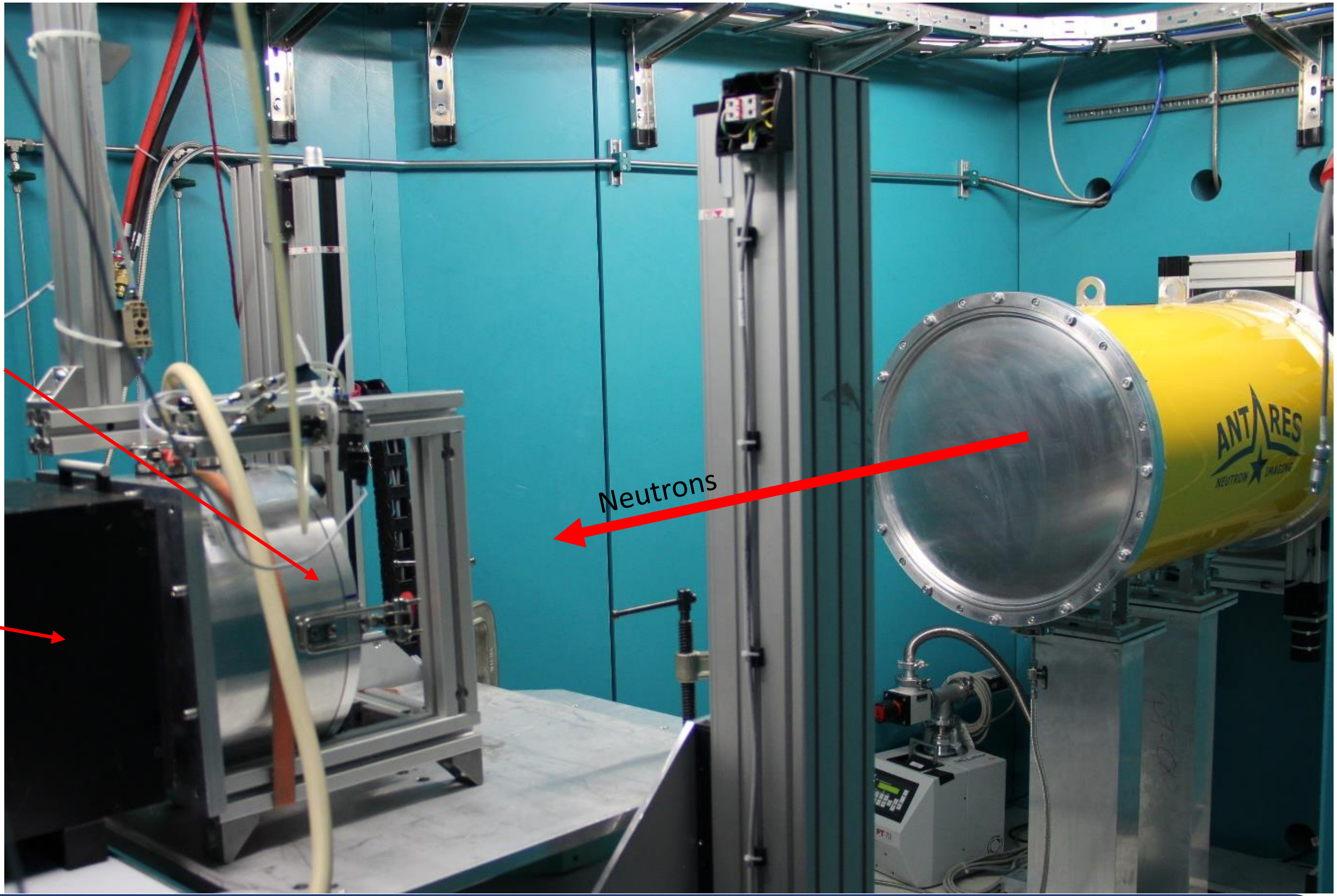
- In-situ visualization of the wetting process
- Study and optimize influence of process parameters

Technique

Setup

Vacuum chamber

Detector



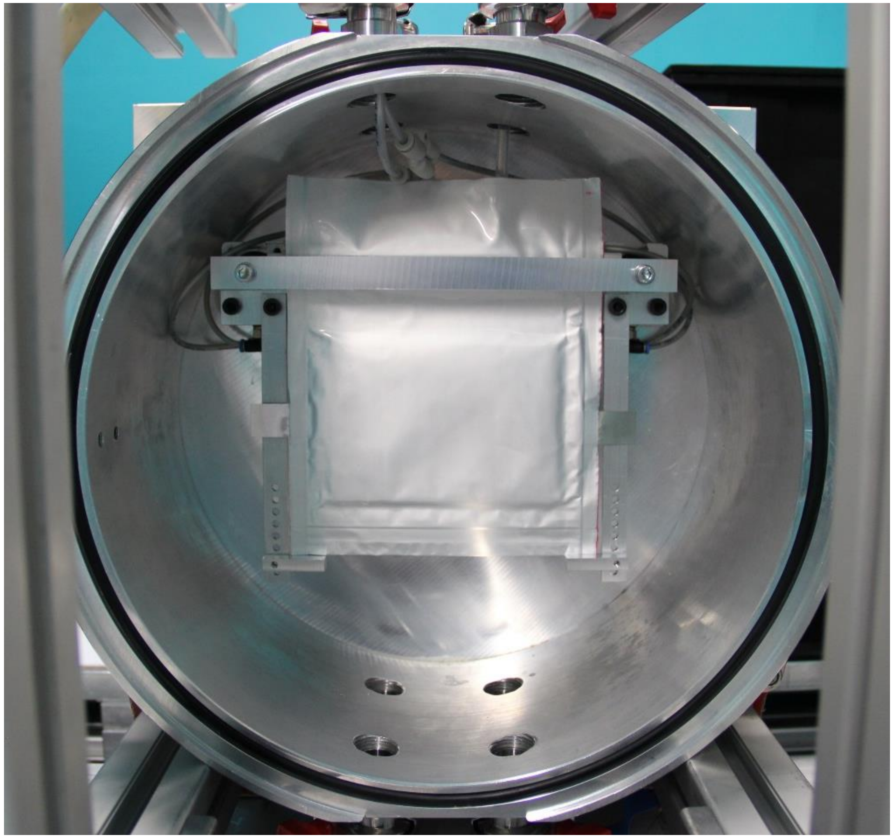
Technique

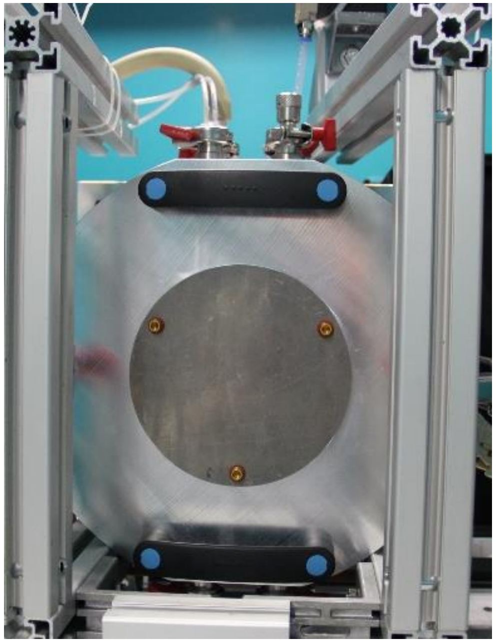
Setup with cell

Materials

Cell
- 5 Anodes,
- 4 Cathodes,
- z-folded
- ExZellTUM-format

Elektrolyte
- EC:EMC 3:7
- No LiPF6,
- No VC

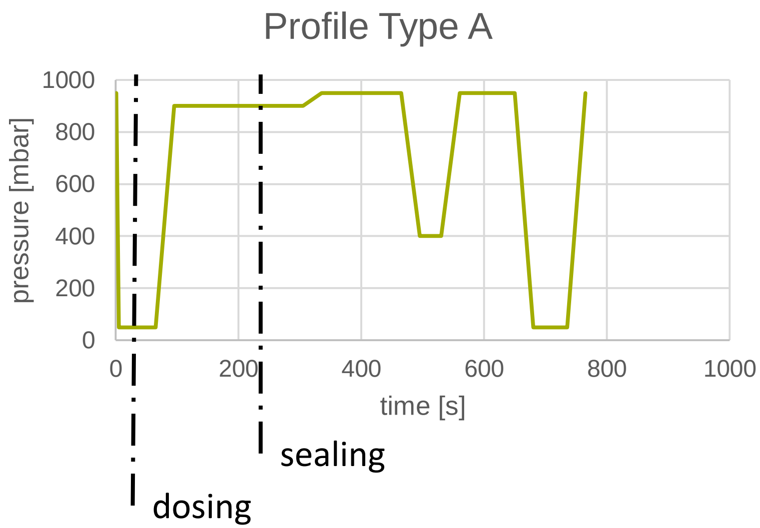


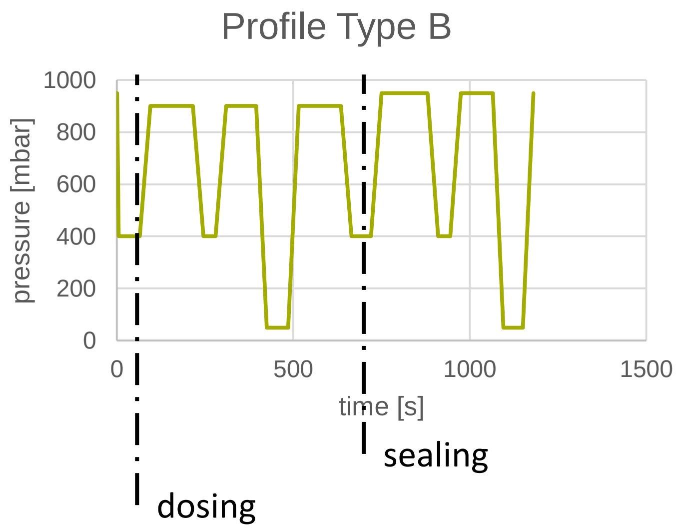


Technique

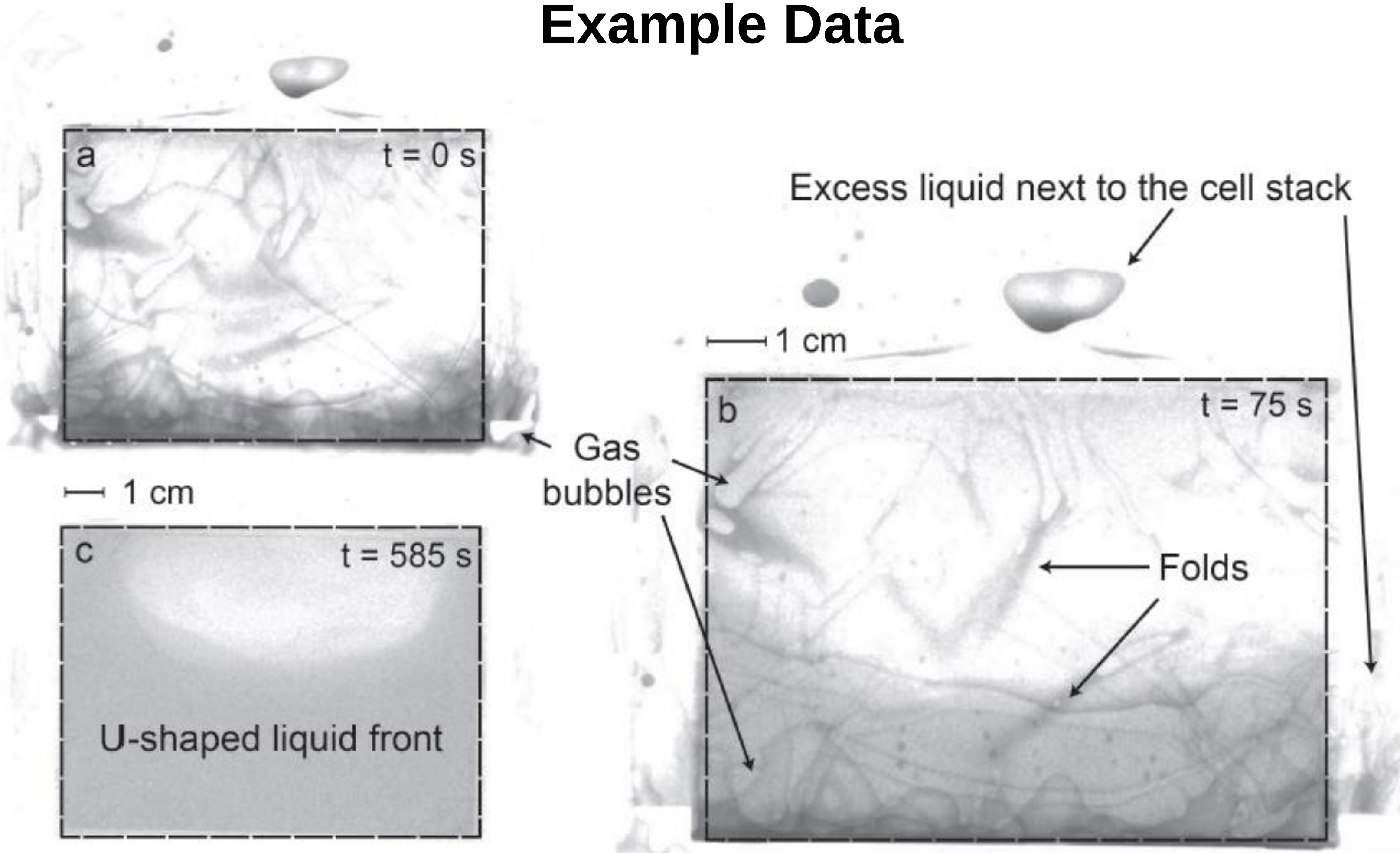
Experiments

- 25 pre-defined pressure profiles
  - Two fundamental types: w/ and w/o wetting cycles before sealing
  - Reference experiments w/o pressure variation
  - Variation of pressure levels
  - No variation of of timing for pressure levels, filling and sealing within one profile
- Image acquisition every 15s
- Control of injection and sealing by the instrument control software





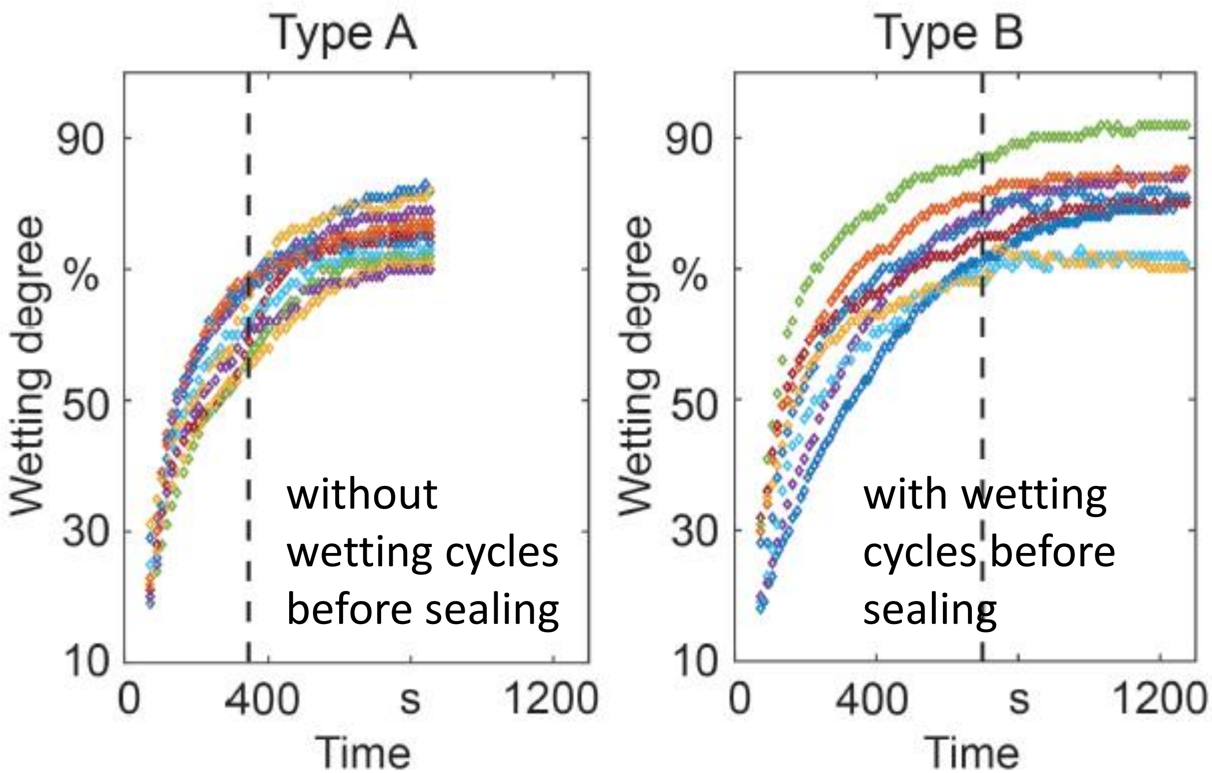
Example Data



Homogenous distribution of liquid

Heterogenous distribution of liquid

Quantitative Evaluation



- Higher wetting degree (t=850s) when applying wetting cycles
- No significant influence of wetting cycles after sealing
- Initial wetting degree depends on dosing pressure

Hydrogen storage

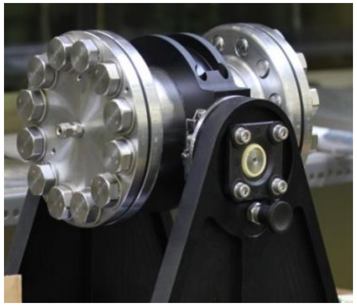
H_2 production	H_2 storage	Conversion to power
		
high selectivity + efficiency	low weight + volume	low cost + high efficiency

www.linde-le.com

www.toyota.de

Lab scale
[mg…g]

ANTARES



Scale-up

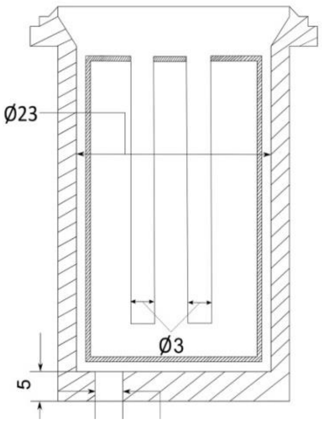
Storage tank [kg]

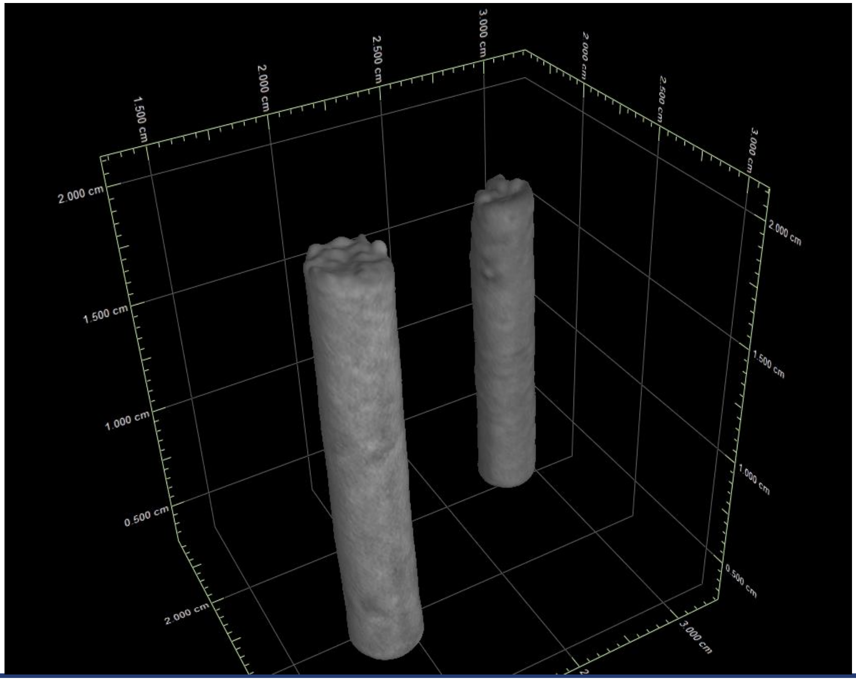
NECTAR

H_2 storage @ ANTARES: high resolution

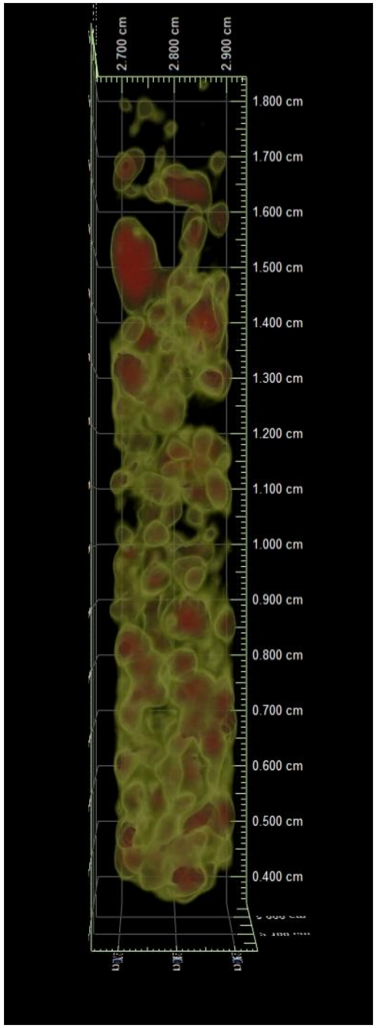
Neutron Imaging study of promising class of Reactive Hydride Composite Materials

- Mixture of two different hydrides to reduce overall enthalpy of formation
- Liquid phase of $LiBH_4$ (melting point 275° C) at operating conditions
- pressure (100 bar) & temperature (400° C) resistant cell made of 1.4401 steel
- powder samples in 3 mm boreholes of aluminum sample inlet (2 x ~75 mg)
- in situ monitoring of temperature, H_2 flow and pressure



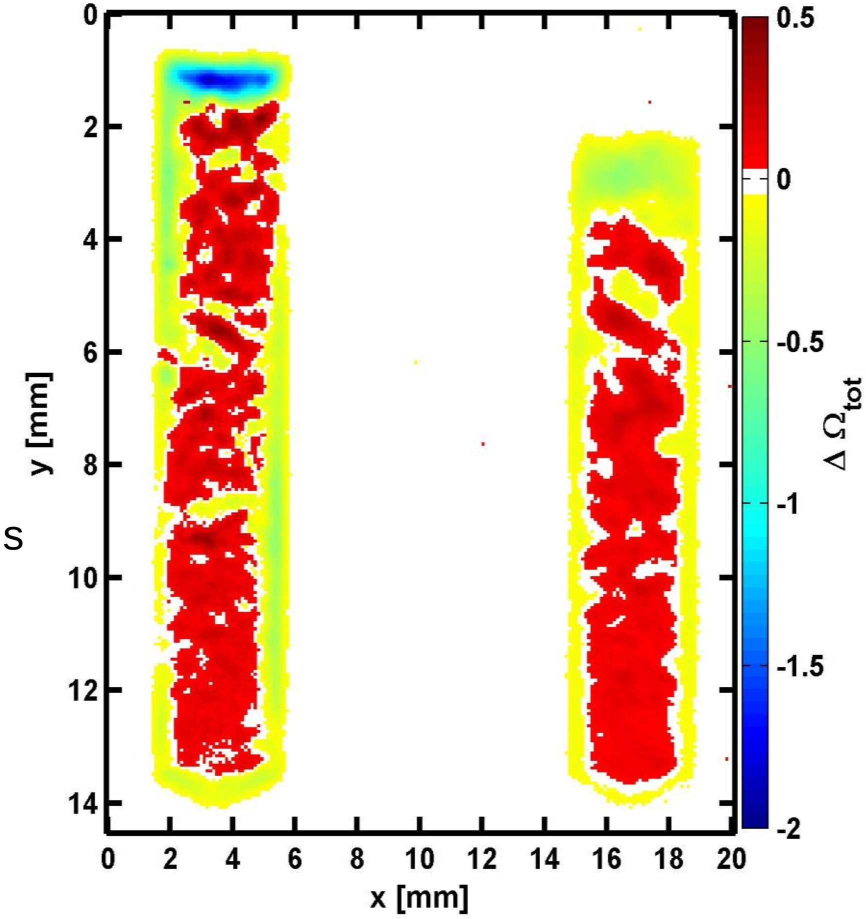


H_2 storage @ ANTARES: high resolution



Tomography analysis of heated (355 ° C) and pressurized (15 bar) sample after induction of phase transition (melting of $LiBH_4$):

- densification / sintering
- counteracts homogeneous material distribution and therewith absorption process

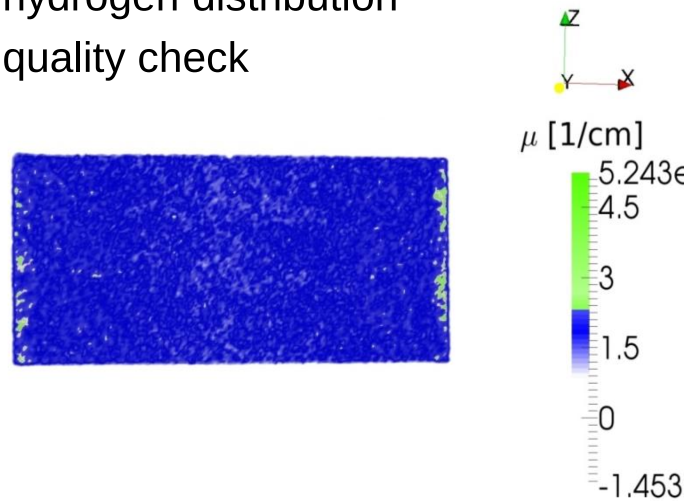


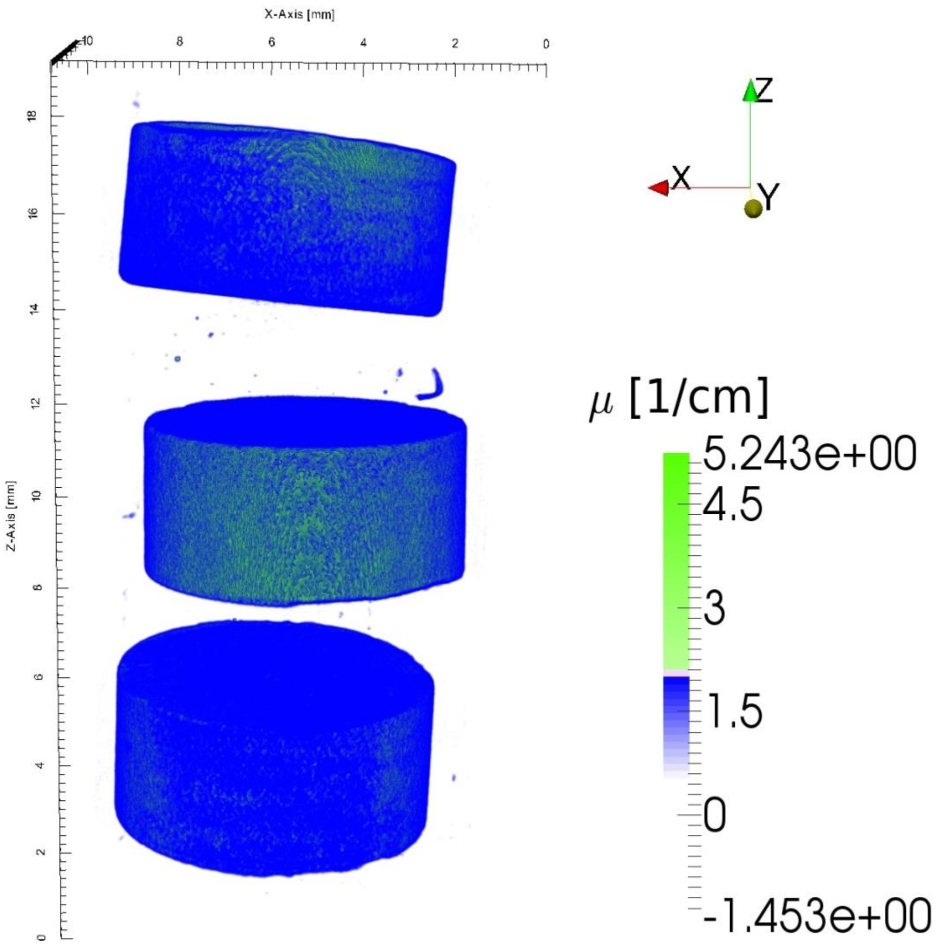
In situ Neutron Radiography study of sintering process: Difference data set, fast sintering (Δt < 200 s) – heavily influencing material structure

H_2 storage @ ANTARES: high resolution

Neutron Tomography of MgH_2 pellets:

- effects of cycling on material structure
- effects of material processing conditions
- hydrogen distribution
- quality check

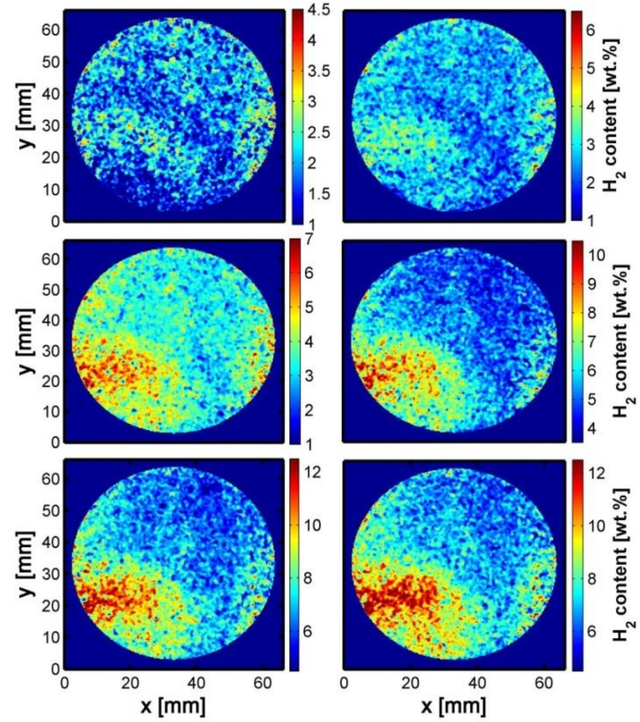


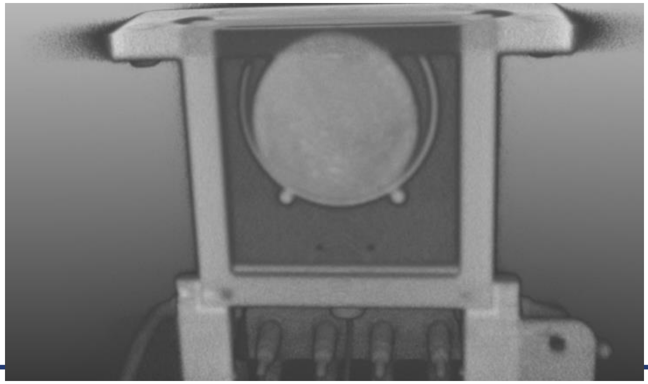


- Optimized processing parameters: homogeneous hydrogen distribution

H_2 storage @ NECTAR: large sample scales

- Investigation of scaled-up & pilot plant hydrogen storage tanks
- In situ Neutron Radiography & ex situ Neutron Tomography studies
- t_{exp} ≥ 120 s @ pixel size 293 µm
- sample thickness up to 200 mm (100 mm steel)
- Investigation of macroscopic material structures, driving forces

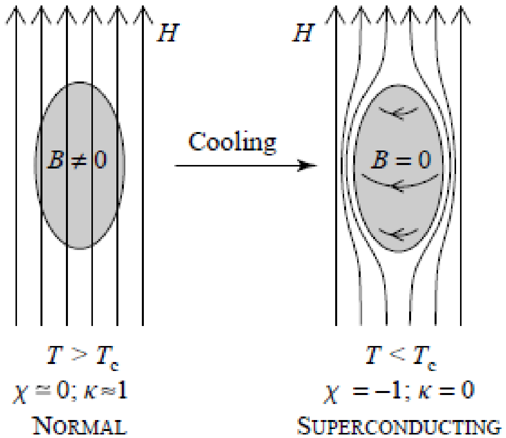


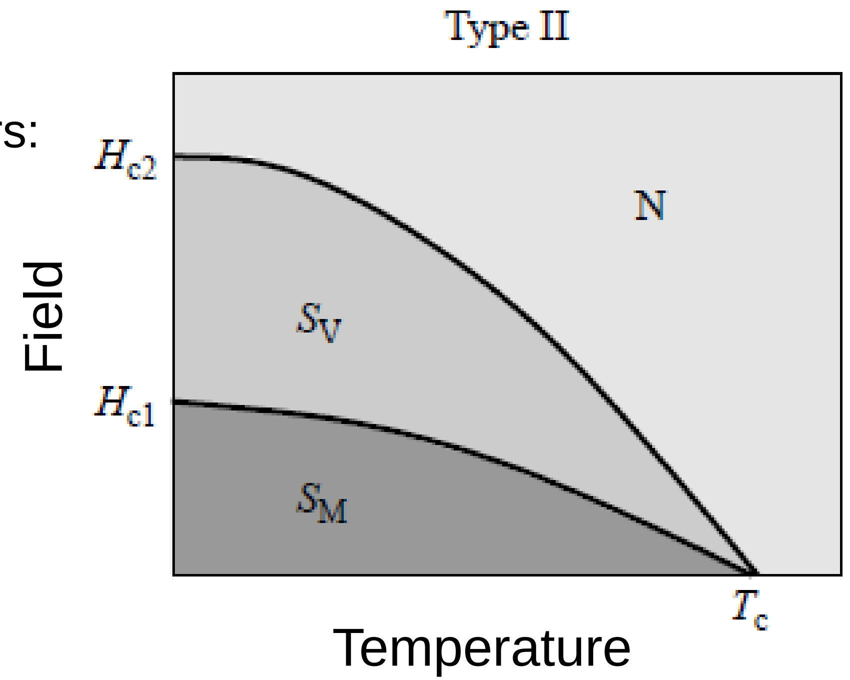


What is Vortex matter?

- Phase diagram of type-II superconductors:

- N … Normal conducting phase
- S_M… Meissner Phase:



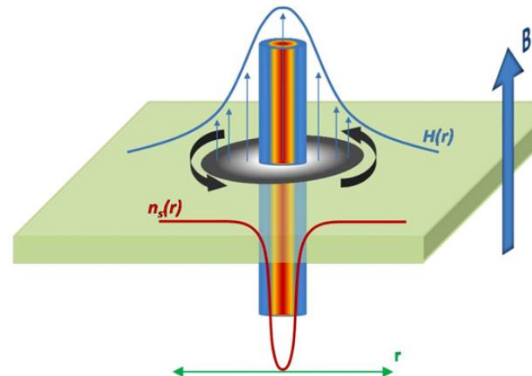


K. Fossheim, A. Sudbo,
Superconductivity: Physics and Applications (2004)

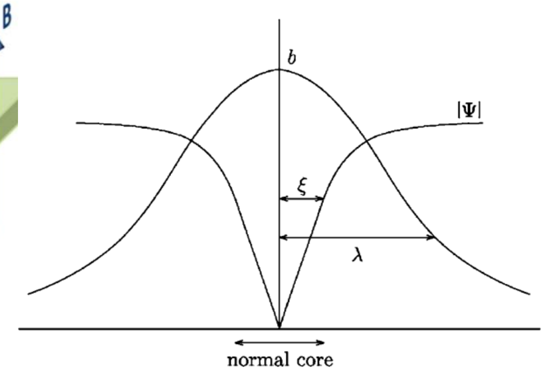
- S_V… Vortex phase → Flux can penetrate the still superconducting sample as **vortex lines**, which carry **one flux quantum**

What is Vortex matter?

- A single vortex line:
  - stable and particle like

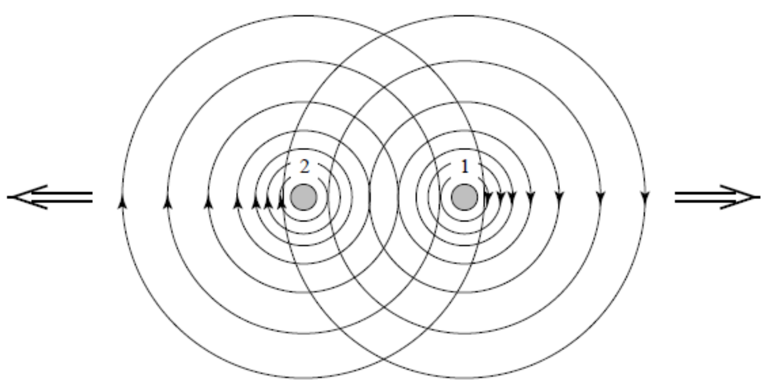


H. Suderow, et al.,
Sup. Sci. Tech. **27,** 063001 (2014)

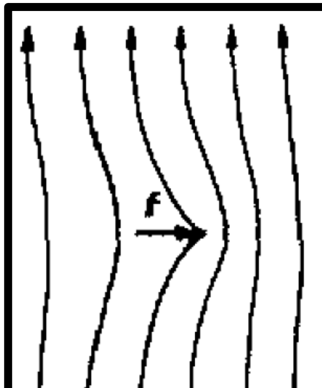


K. Fossheim, A. Sudbo,

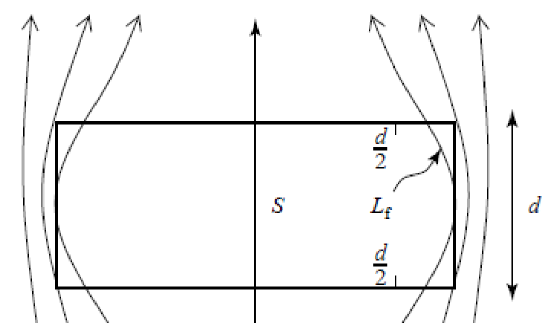
- Interactions (just a few):



Vortex-vortex interactions



pinning

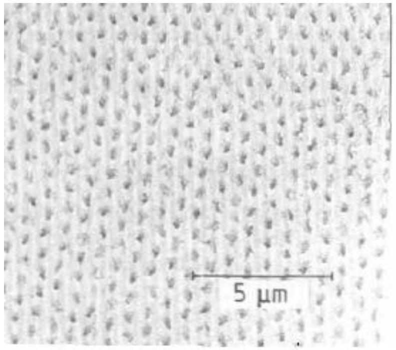


Surface & geometrical barriers

Complex interplay of interactions → **multiple phases**

How to observe Vortex matter?

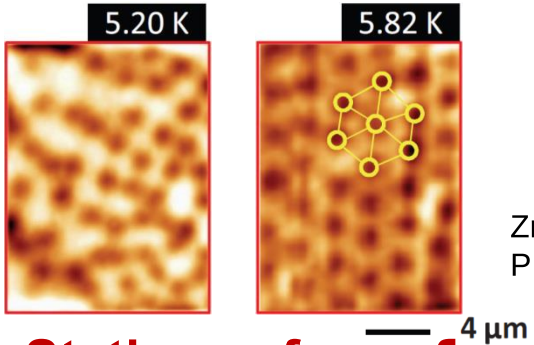
- Decoration:



Brandt,
Rep. Prog. Phys. **58,**
1465 (1995)

Static, influenced surface : λ

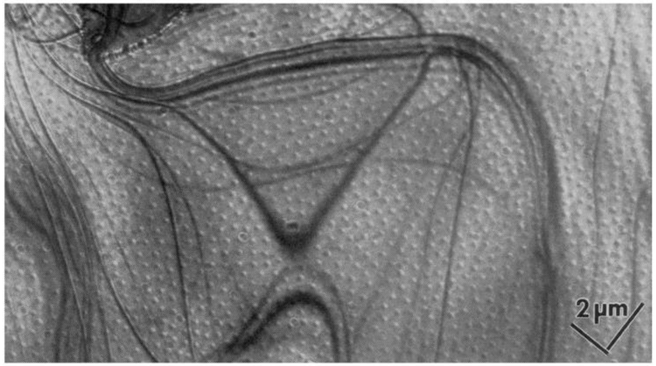
- Scanning tunneling microscopy:



ZrB_{12}: J. Ge, et al.,
PRB **90**, 184511 (2014)

Static, surface : ξ

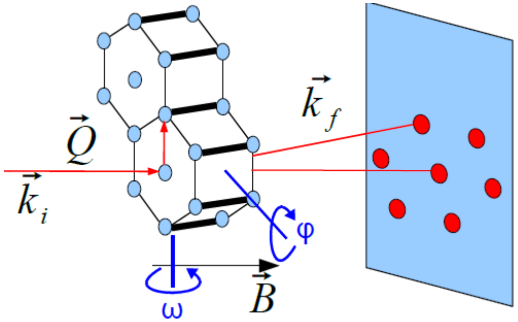
- Lorentz microscopy:

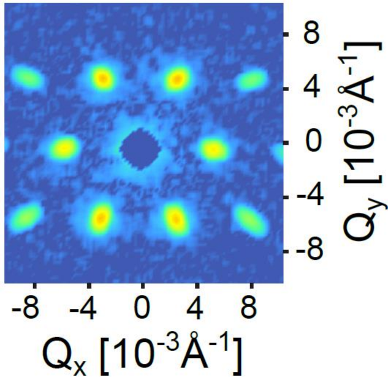


Real time, thin film: λ

K.Harada, et al., Nature **360**, 51 (1992)

- Small angle neutron scattering (SANS):

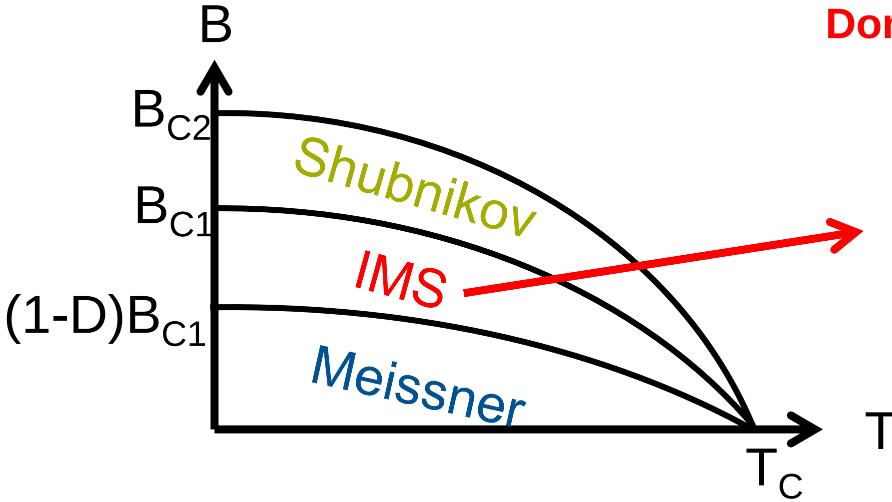




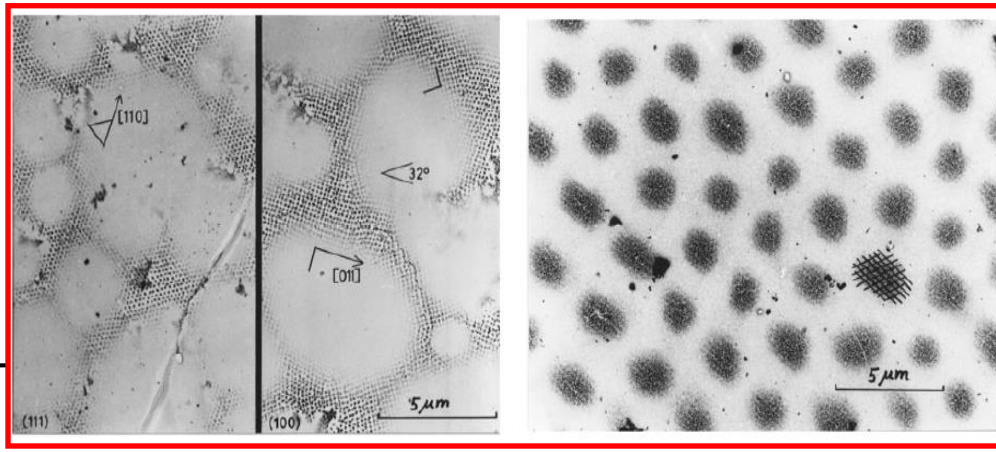
Static, averaged bulk : λ

The intermediate mixed state (IMS) in Niobium

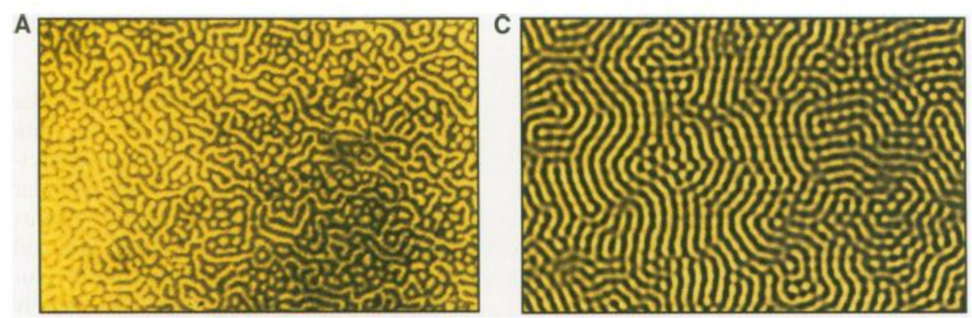
- In Nb: λ≈ξ → Normal cores of flux lines overlap → VL attraction in the IMS



Domain structure of Meissner and Shubnikov phase:

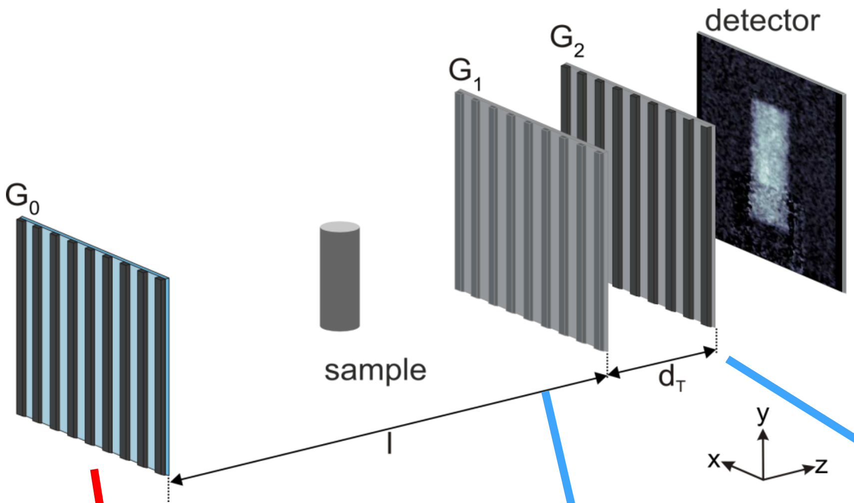


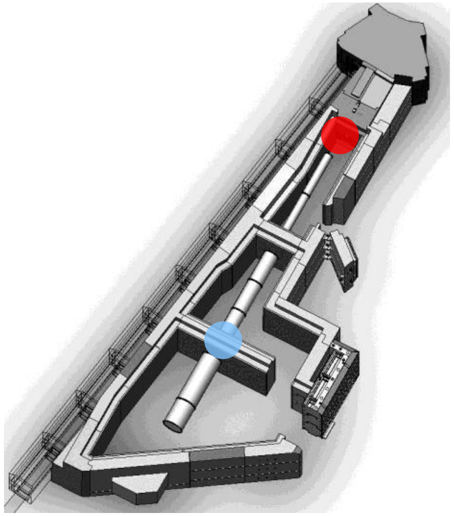
- **Connected problem:** Domain nucleation under fixed boundary conditions
- So far no bulk information

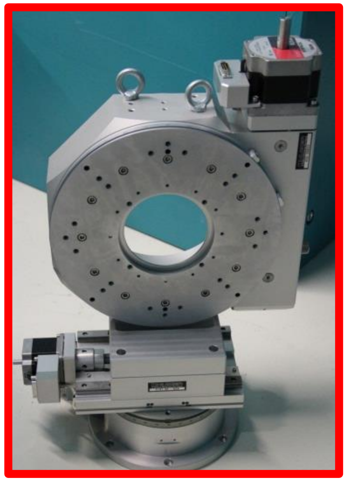


Top: Brandt, Rep. Prog. Phys. **58,** 1465 (1995)
Right: M. Seul, D. Andelman, Science **267,** 476 (1995)

The IMS in Niobium: neutron grating interferometry (nGI)



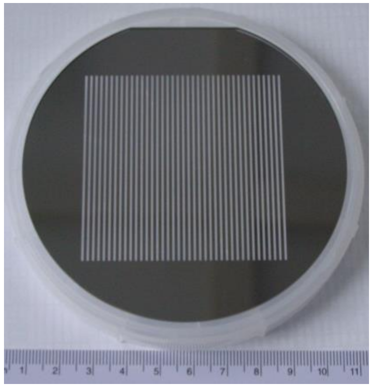


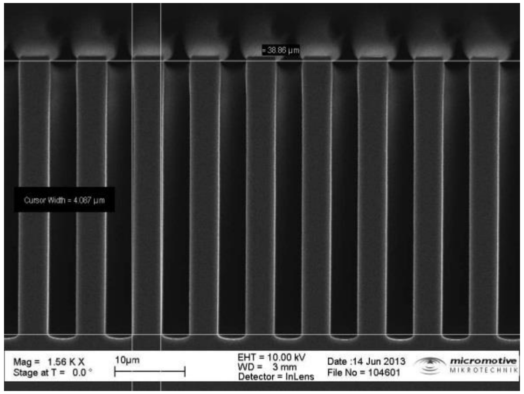


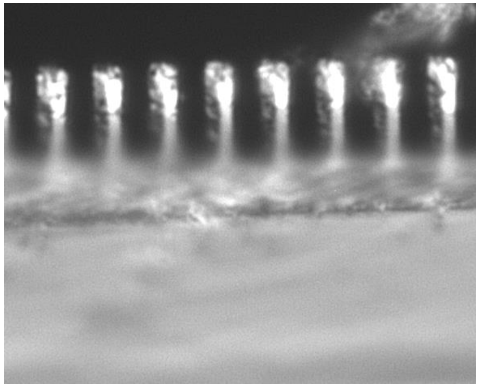
p_0=1.6 mm

p_1=7.98 µm

p_2=4.0 µm

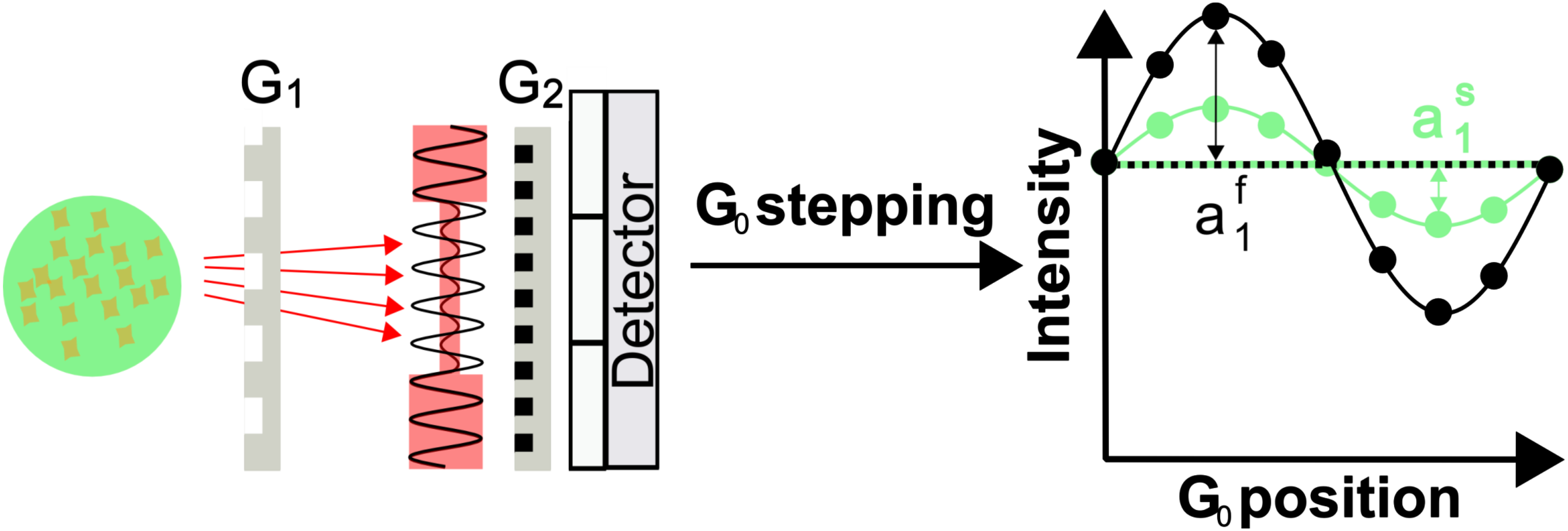






The IMS in Niobium: neutron grating interferometry (nGI)

- Setup generates neutron interference pattern at detector:

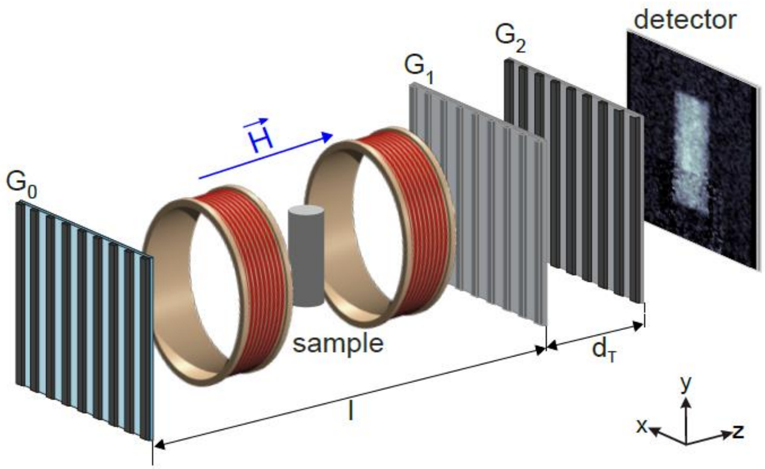


- Scattering at µm structures locally degrades interference pattern
- Degradation of interference pattern mapped in the DFI

→ DFI = spatially resolved USANS scattering map

The IMS in Niobium: neutron grating interferometry (nGI)

- Short reminder: IMS = µm domains of VL:
  → well suited for nGI
- Nb **ultra-pure** single crystal rod:
  → Where does the IMS nucleate in the absence of pinning?

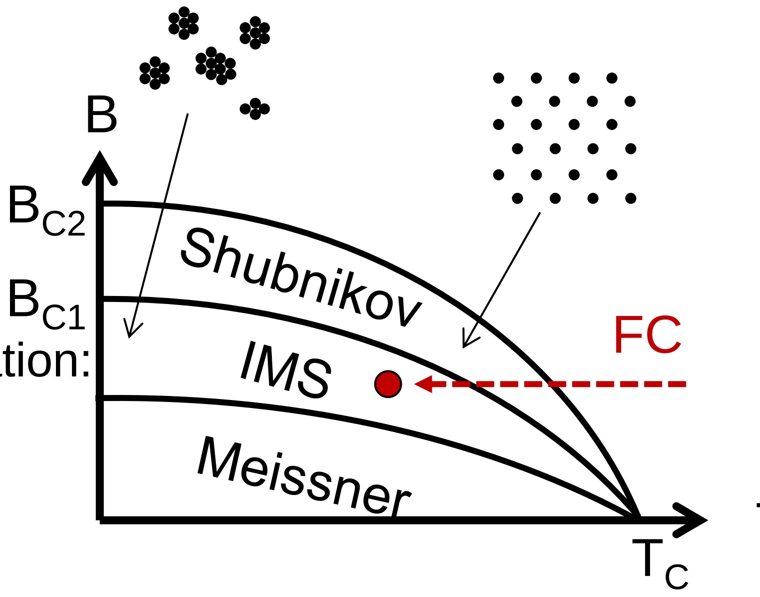


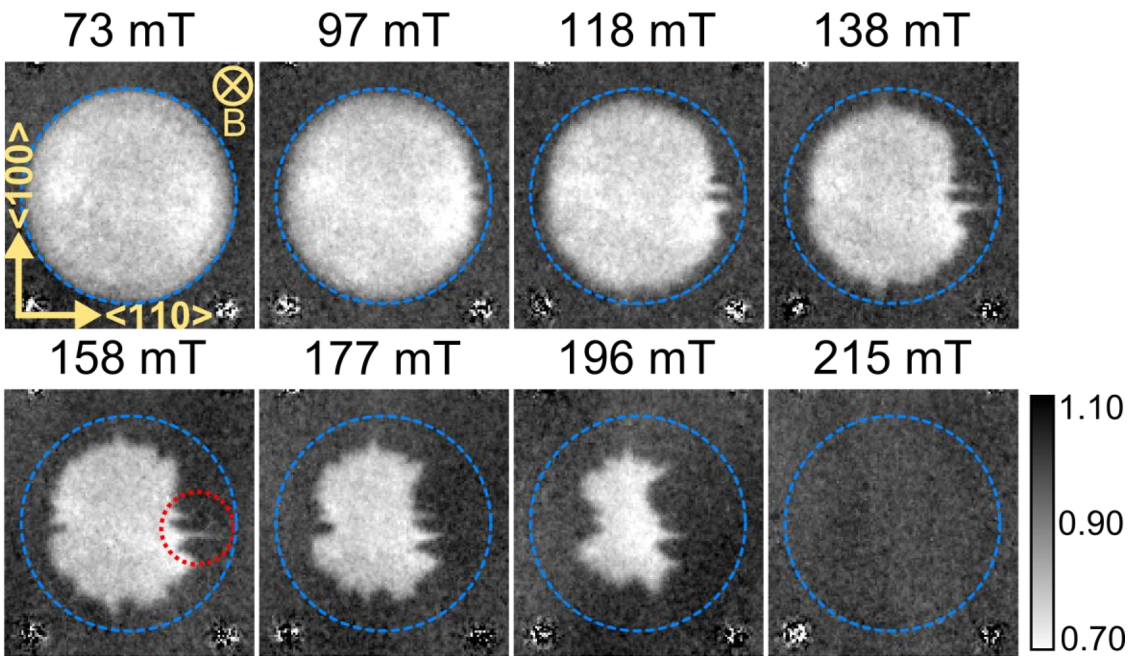
- Nucleation in the center→ growing to the edge of the rod

T. Reimann et al., Nature Communications, (2015)

The IMS in Niobium: neutron grating interferometry (nGI)

- Nb single crystal disc with **significant** pinning:
  → ZFC: No domain structure detected
  → FC: domain structure
  Pinning prevents IMS nucleation
- IMS as **contrast agent** to observe field penetration:





- IMS shrinks due to further field penetration
- Pinning centers and anisotropies can be identified
- **nGI probes domain distribution**

Conclusions

- Neutron imaging has applications in many fields of science and technology

- Complementarity to X-rays justifies higher effort

- Particularly the high sensitivity for hydrogen and the good penetration of many metals define the fields of applications

- Neutron imaging can not only probe absorption but also USANS scattering (nGI) and magnetism (polarized imaging)

Thank you!