HZB Helmholtz Zentrum Berlin

FASEM 2019

Neutron and X-ray imaging

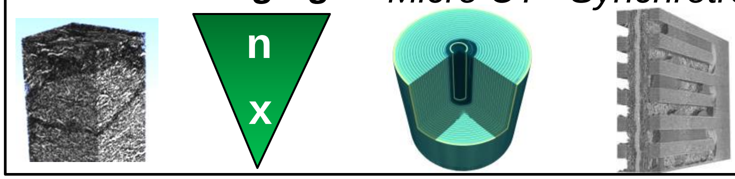
Nikolay Kardjilov

Introduction

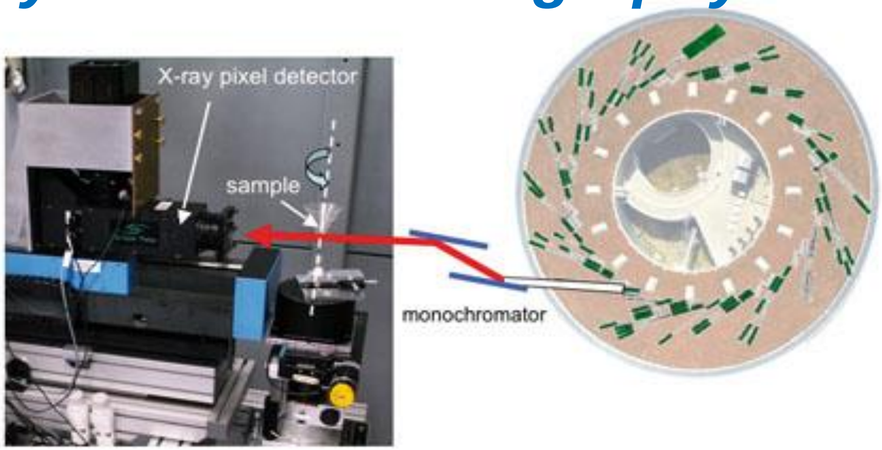
Introduction

Institute of Applied Materials

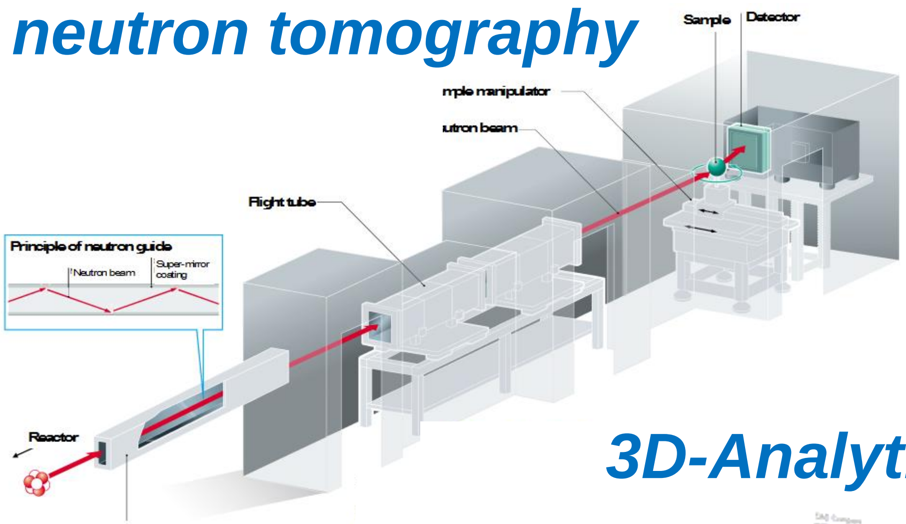
Neutron ***Imaging*** *Micro CT* *Synchrotron*



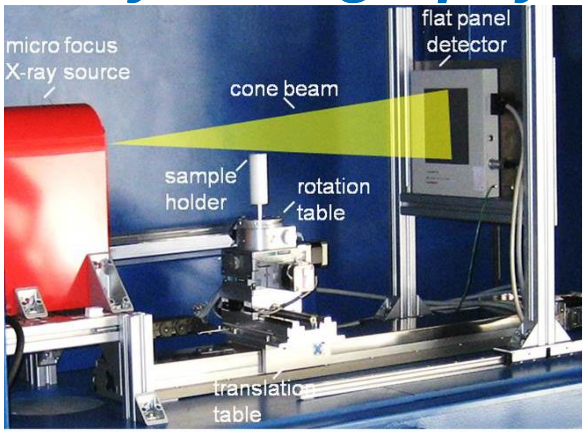
BAM-Line @ BESSY
Synchrotron tomography



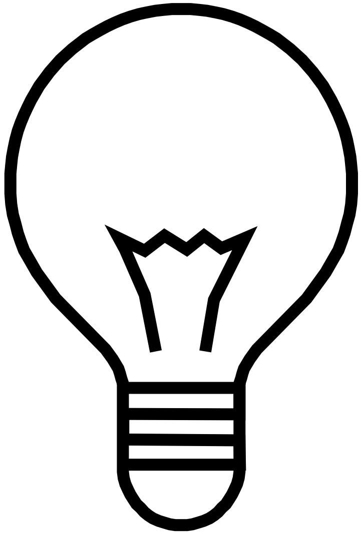
CONRAD-2
neutron tomography

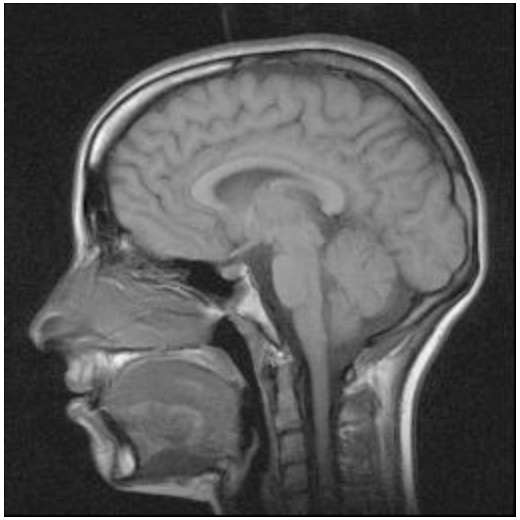


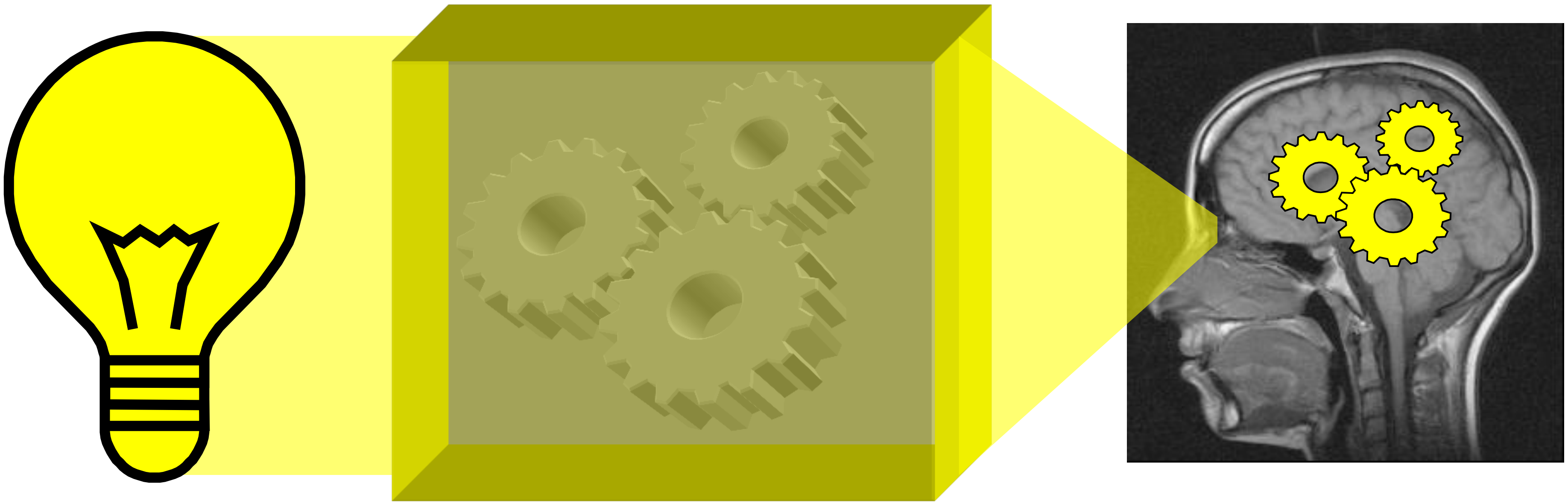
MicroCT Lab
X-ray tomography



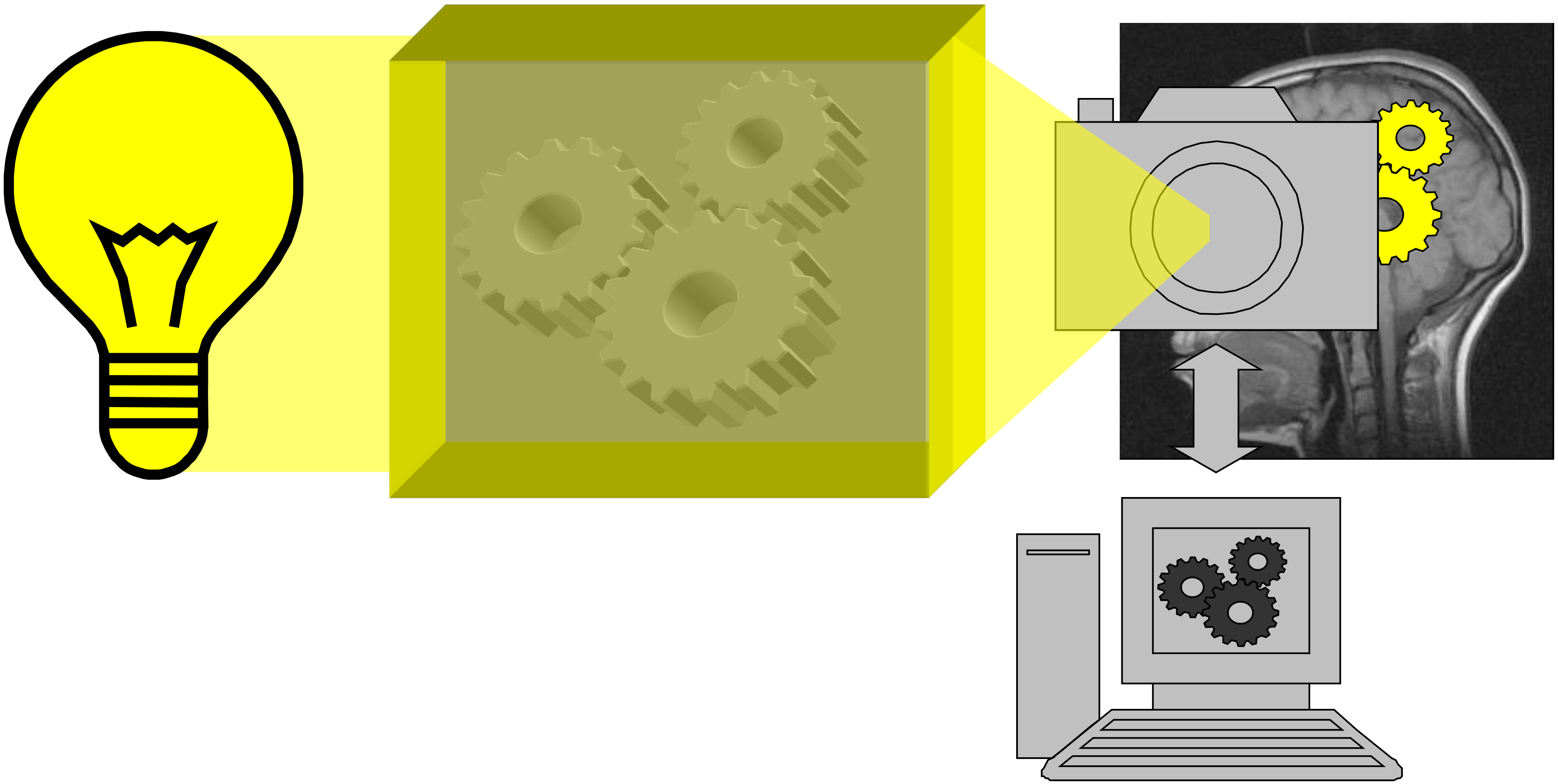
3D-Analytics Lab



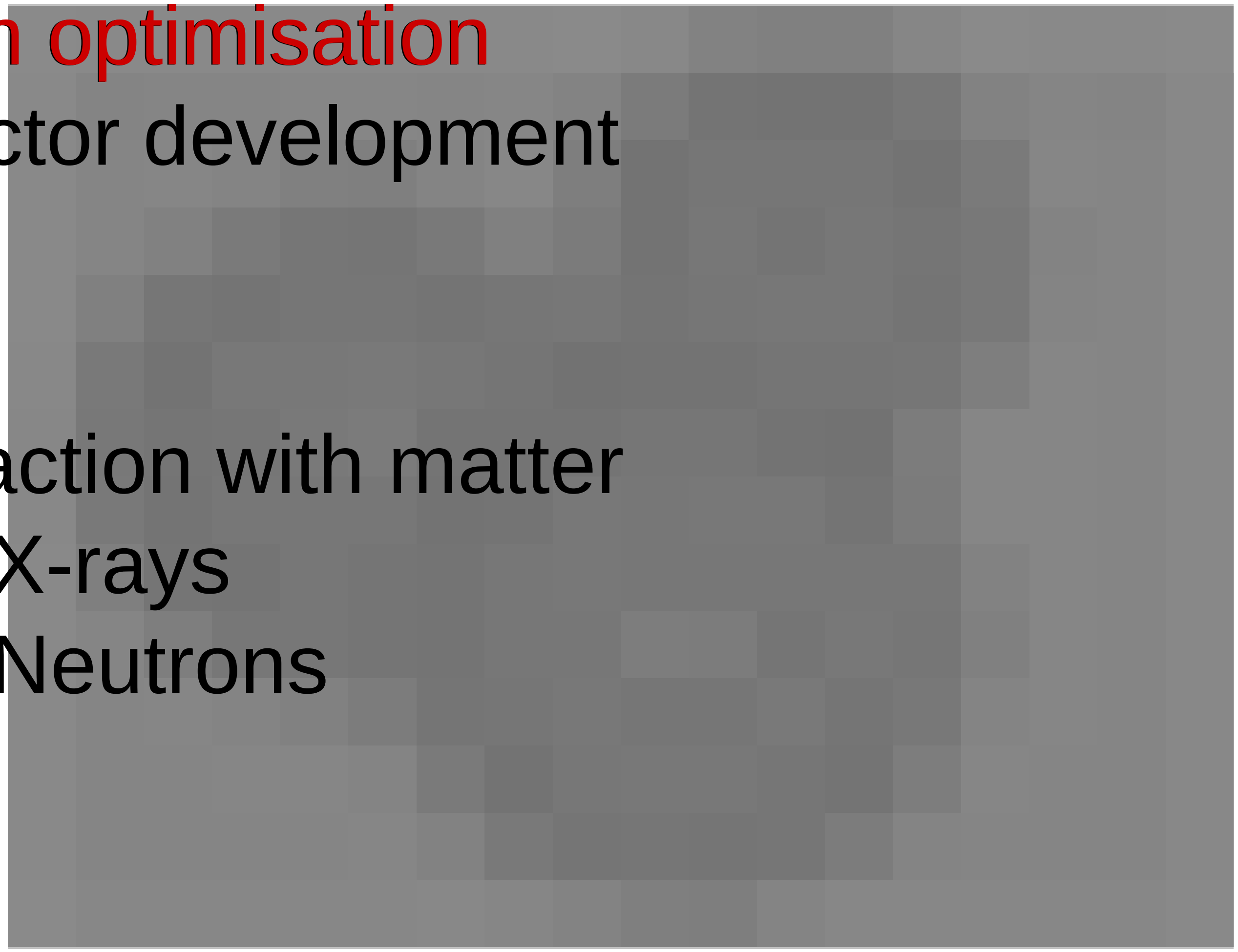




Source | Sample | Detector

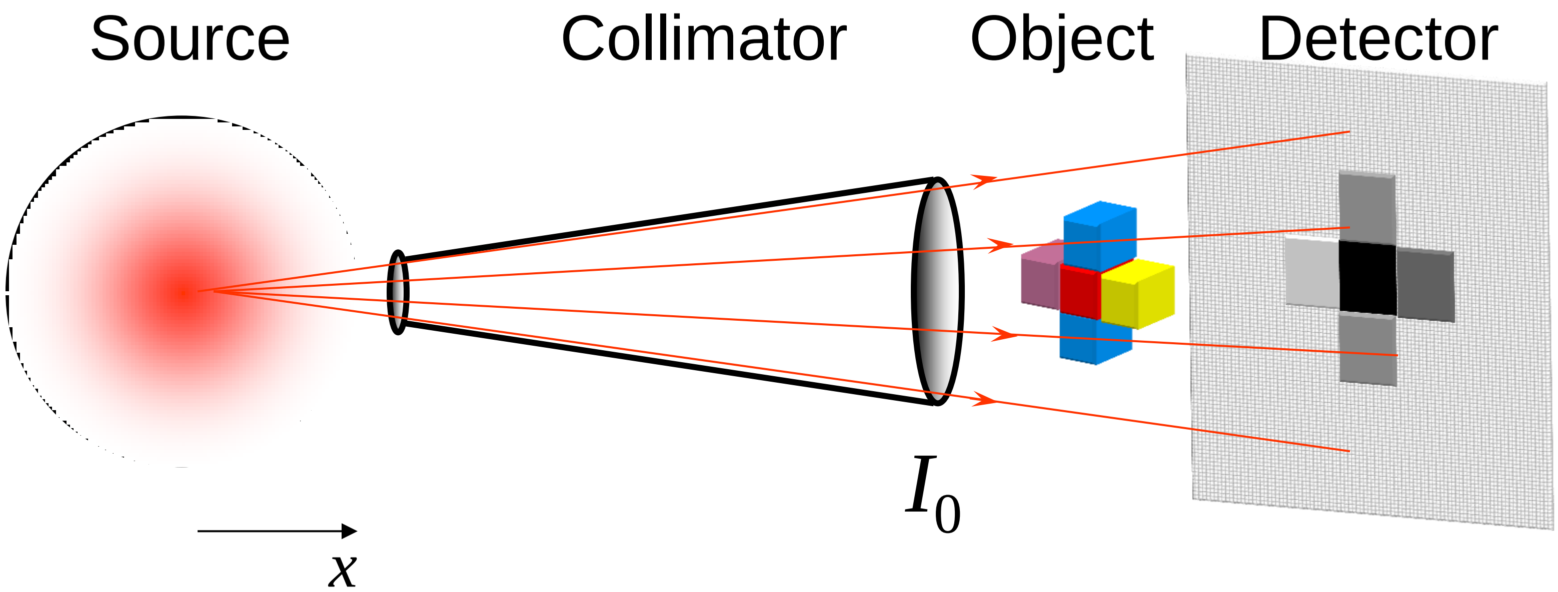


Contrast Resolution



- Beam optimisation
- Detector development
- Interaction with matter
  - X-rays
  - Neutrons

Beam optimisation



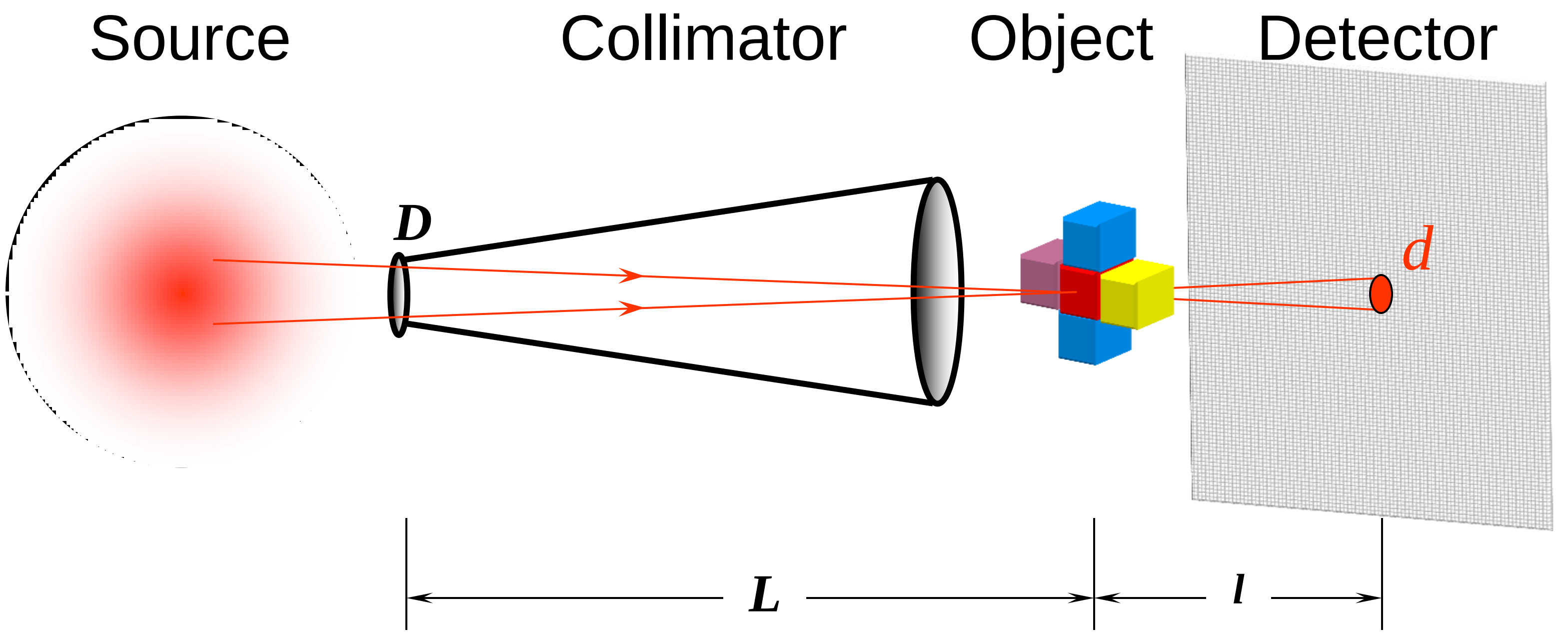
$$\sim I_0 e^{-\int \Sigma(x)dx}$$

x – propagation direction

I_0 – primary beam

$\Sigma(x)$ – attenuation coefficient

Beam optimisation

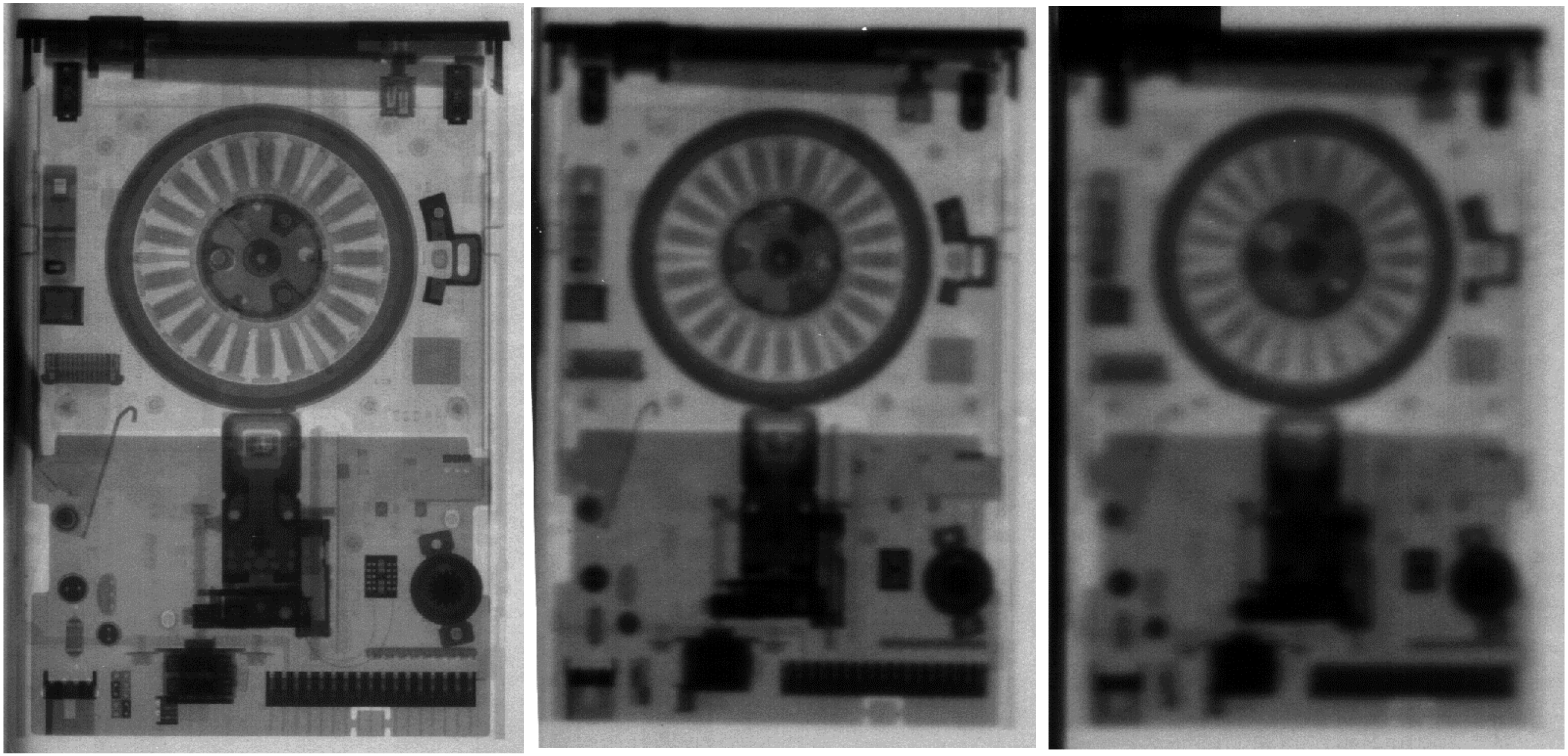


D – Collimator aperture

L – Distance Collimator-Object

l – Distance Object-Detector

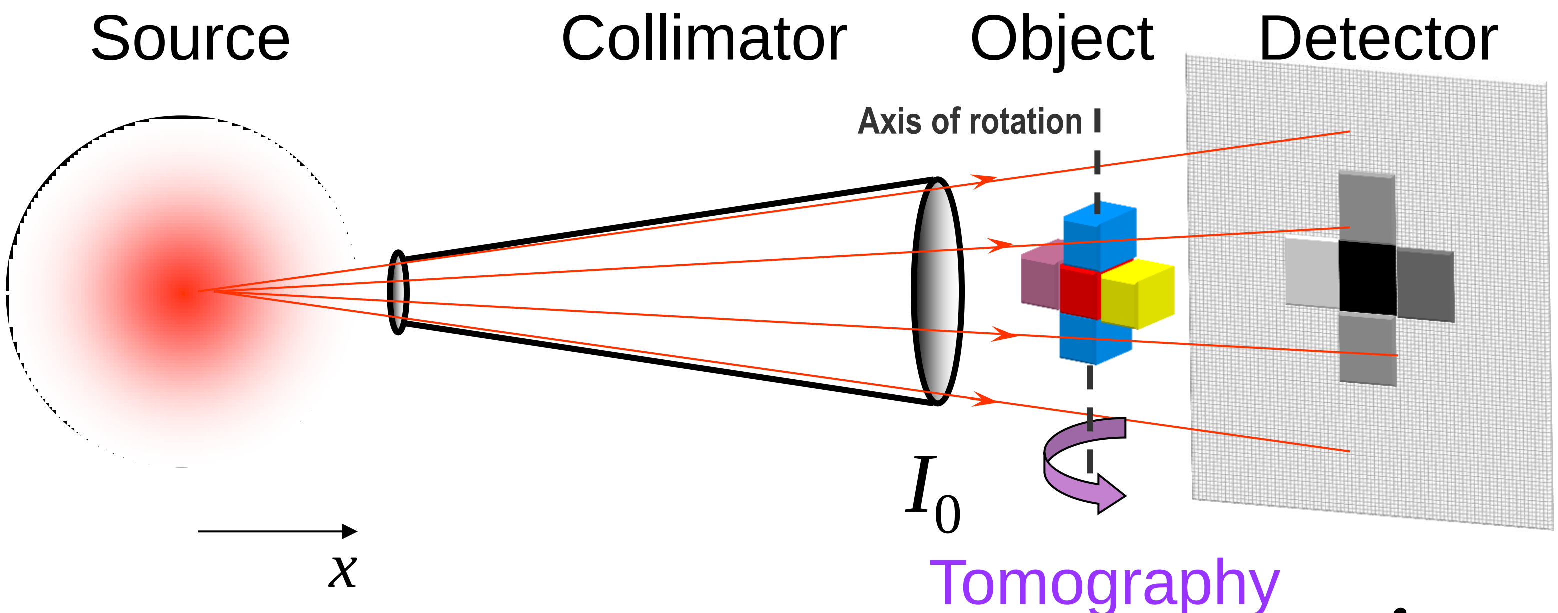
$$d = \frac{l}{L/D}$$



Radiographs of a 3,5'' floppy drive in 0 cm , 10 cm and 20 cm distance from a film + Gd sandwich taken at a cold neutron guide with *L/D*=71.

The measurements are performed at FRM-I, TU-München by B. Schillinger

Absorption tomography



$$\sim I_0 e^{-\int \Sigma(x)dx}$$

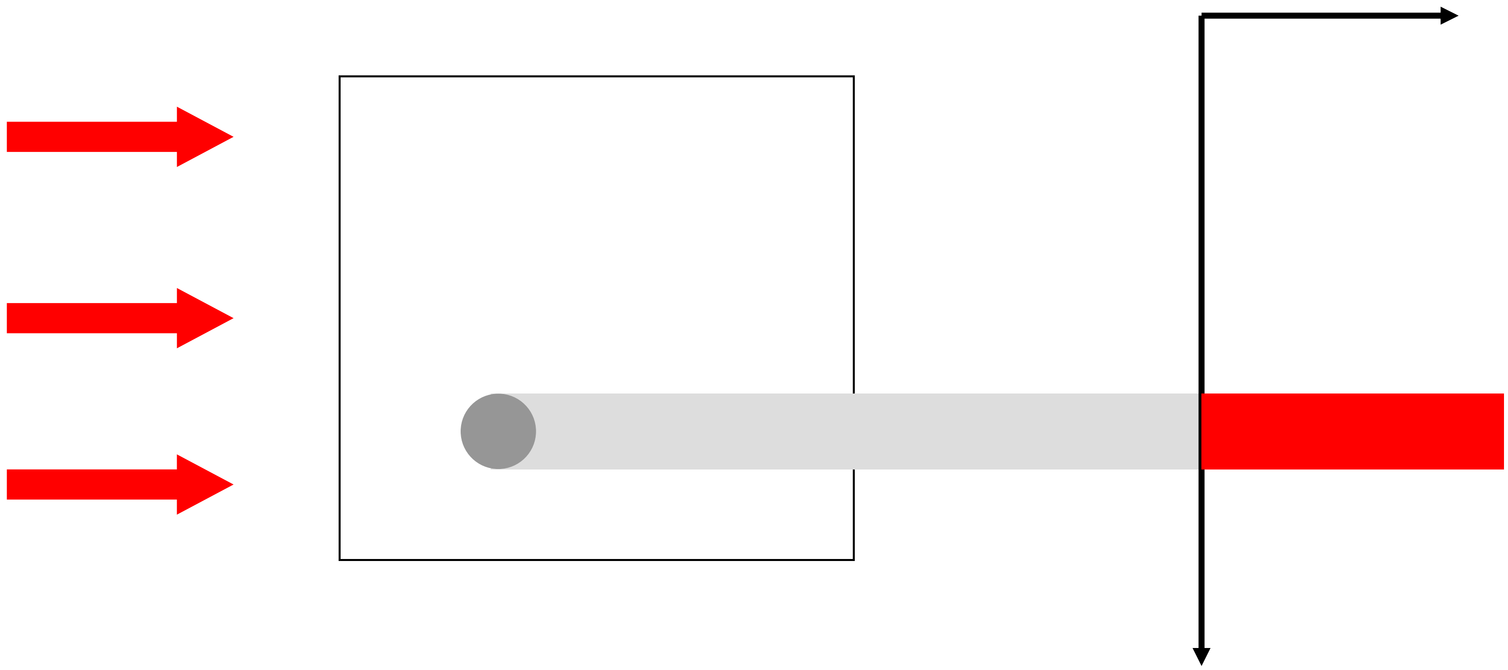
x – propagation direction

I_0 – primary beam

$\Sigma(x)$ – attenuation coefficient

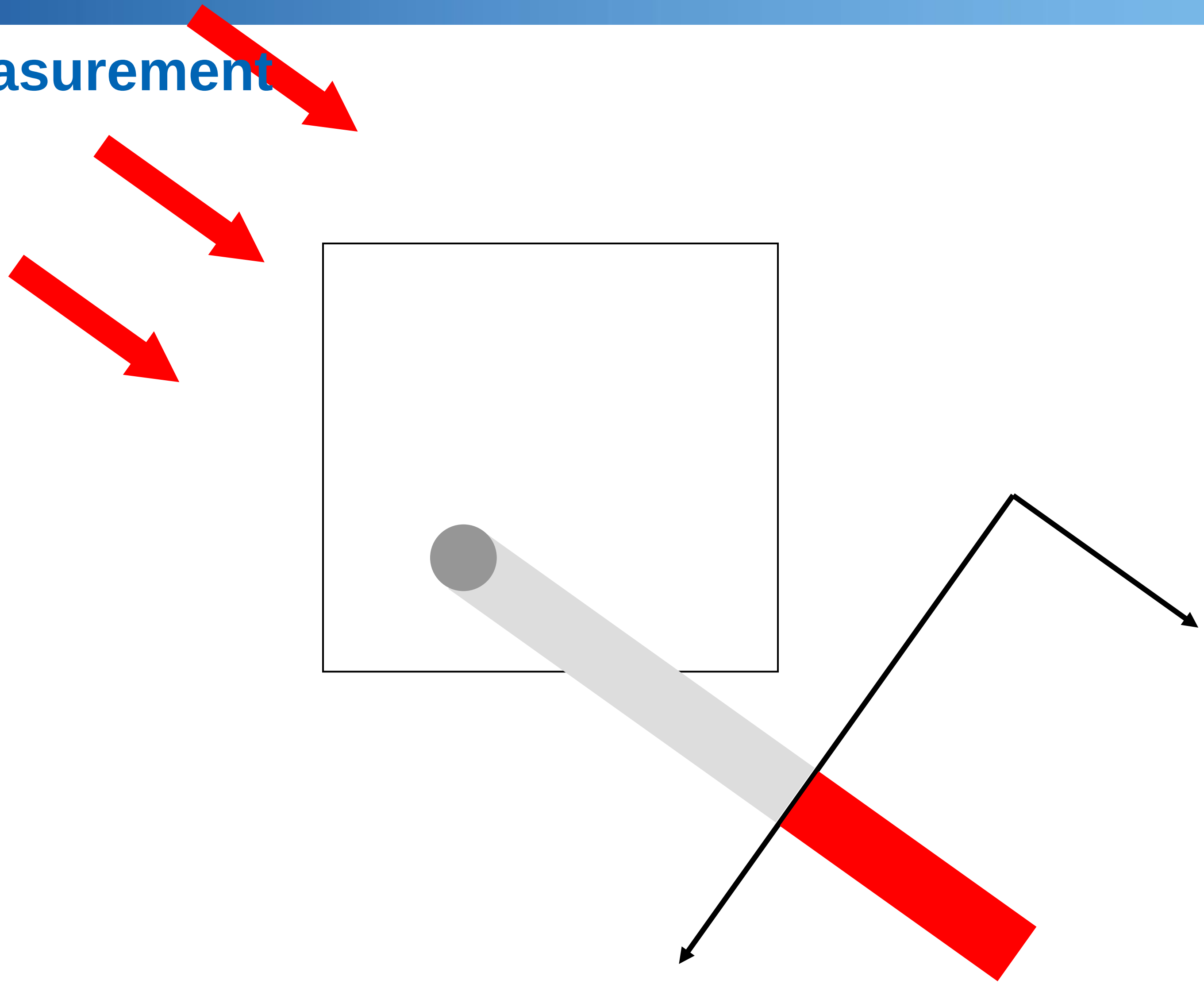
Principle of tomographic reconstruction

Measurement



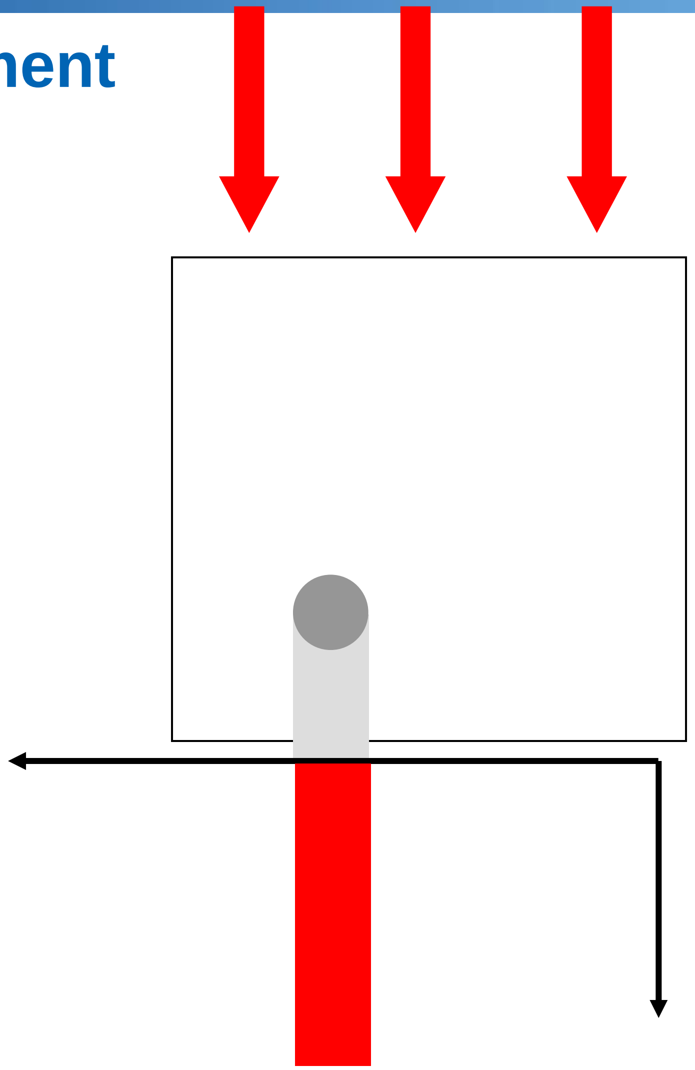
Principle of tomographic reconstruction

Measurement

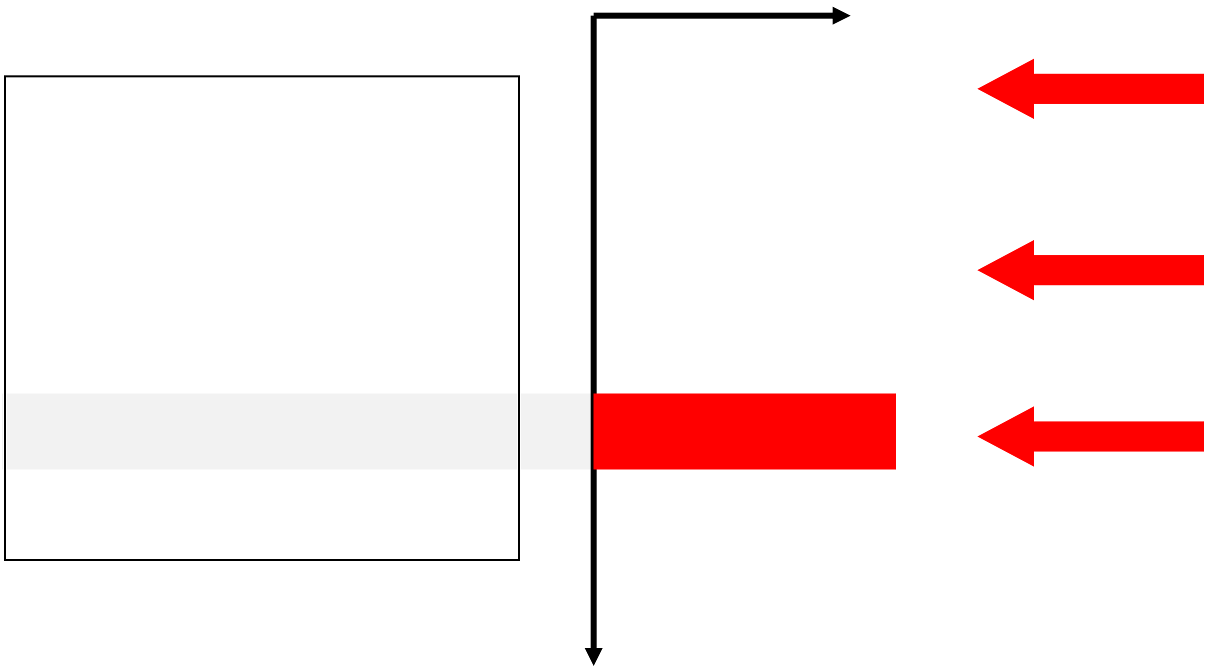


Principle of tomographic reconstruction

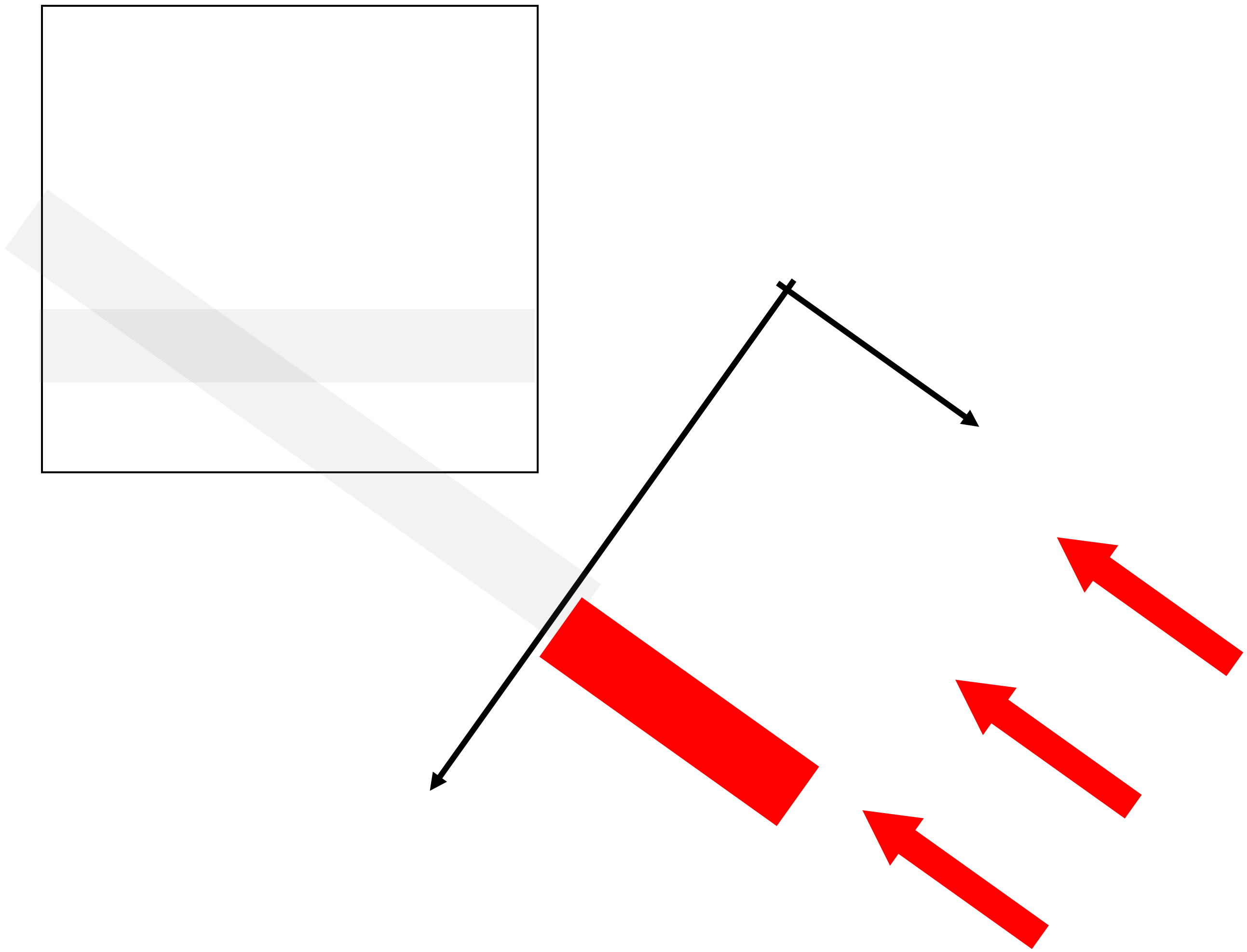
Measurement



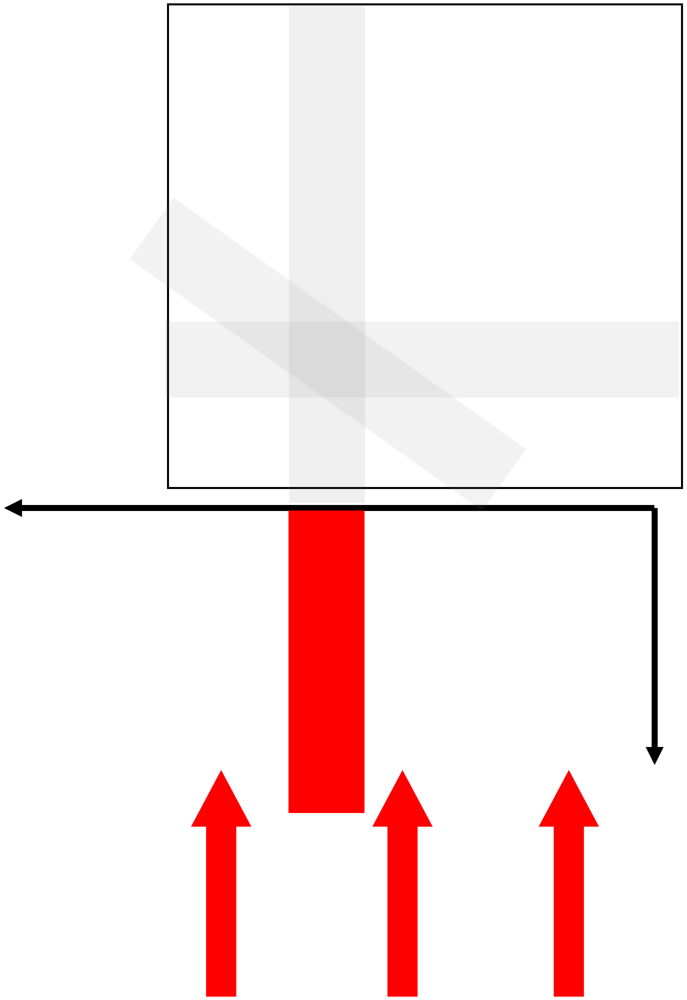
Back projection



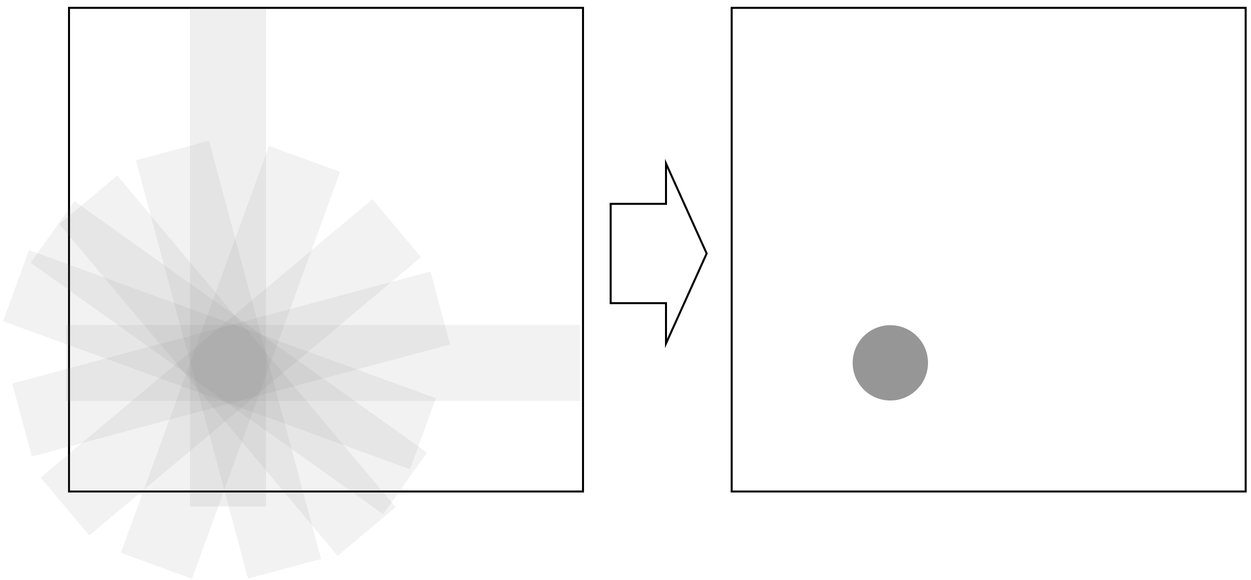
Back projection



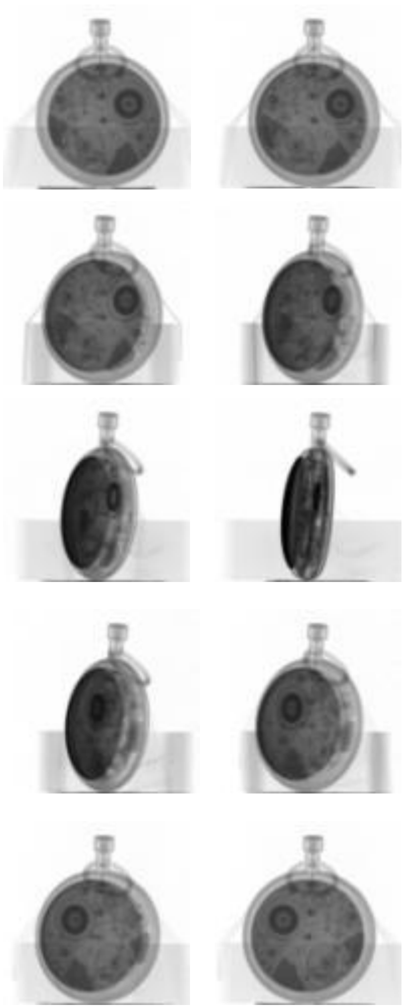
Back projection



Back projection



Absorption tomography



Resolution

- Beam optimisation
- Detector development

Contrast

- Interaction with matter
  - X-rays (sources, interaction, detectors)
  - Neutrons

Introduction

First experiments with a new kind of radiation were performed by Konrad Röntgen in 1895 during investigations with cathode-ray tubes.

He found the new ray could pass through most substances casting shadows of solid objects.

In conjunction with a photographic plate, a picture of interior body parts can be obtained when human tissue will be investigated.

Neutron imaging

Introduction

One of the first experiments late in 1895 was a film of a hand of his wife.

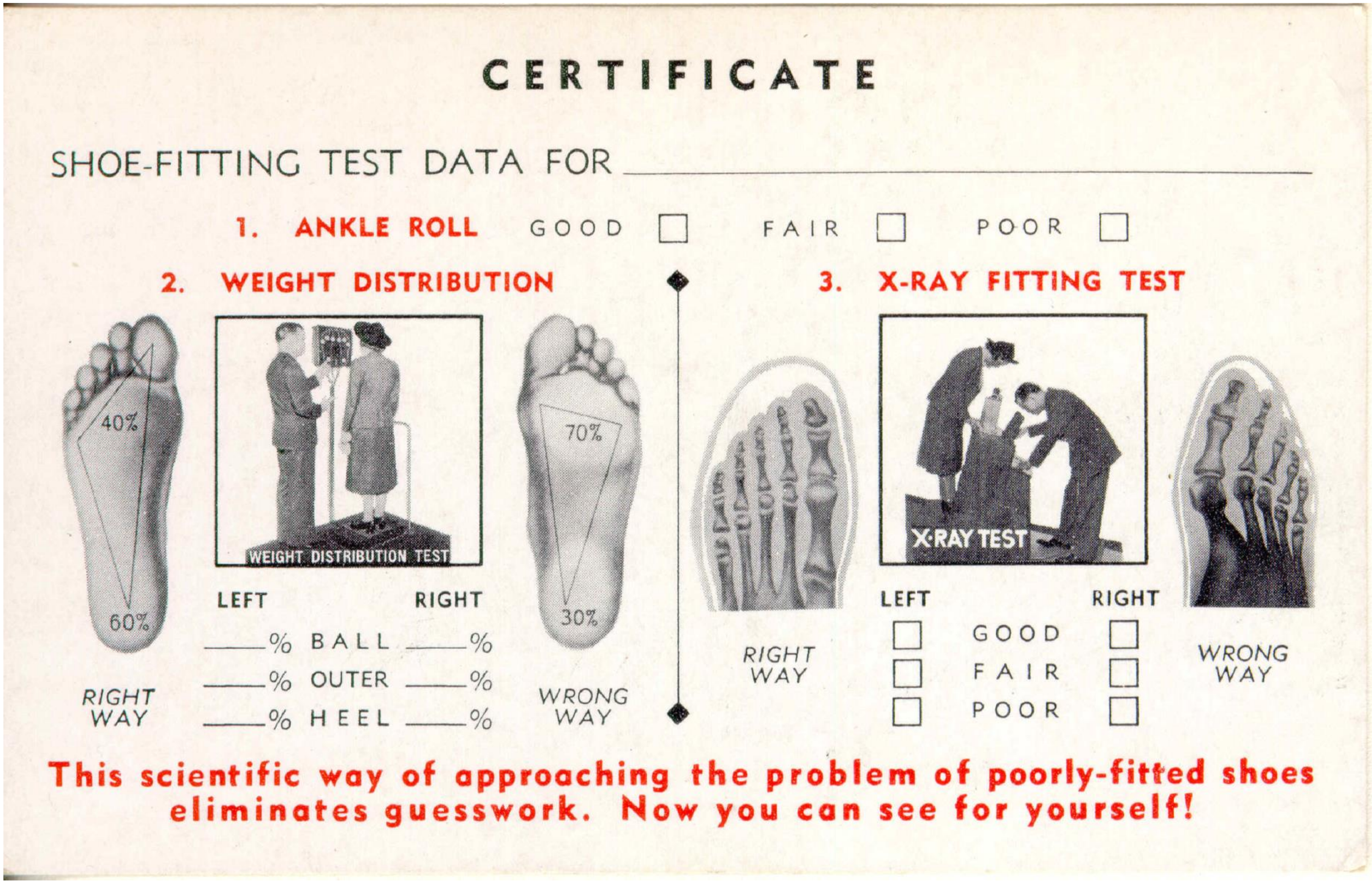
The bones and also finger rings deliver much higher contrast than the soft tissue.

Introduction

Some exotic applications of X-ray transmission in the earlier period of use.

http://www.orau.org/ptp/collection/shoefittingfluor/shoe.htm

Introduction



Introduction

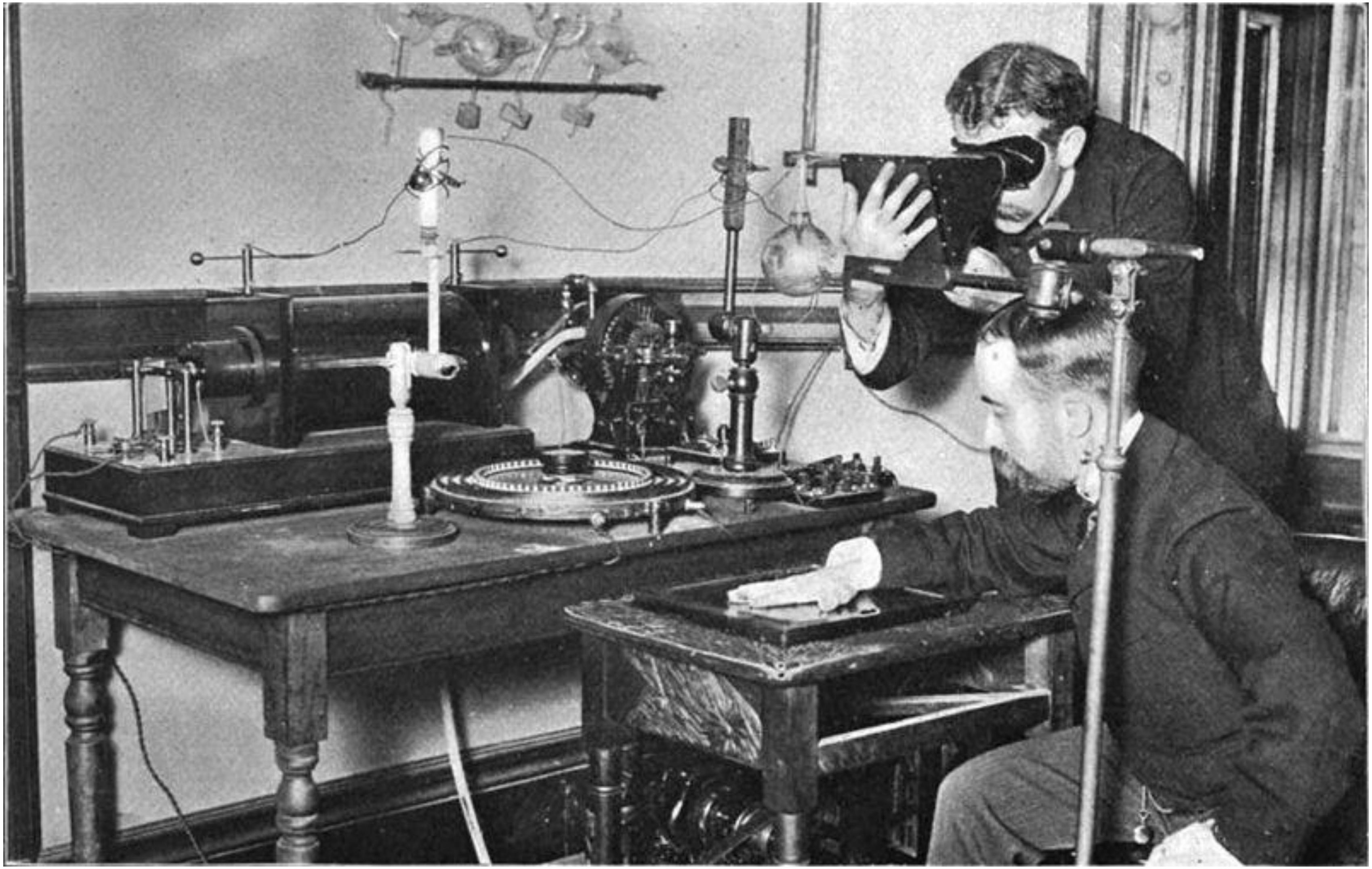
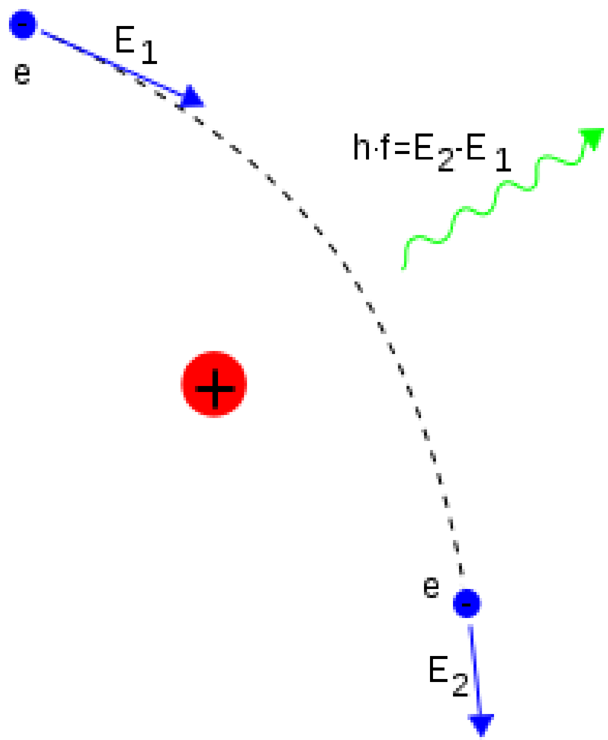
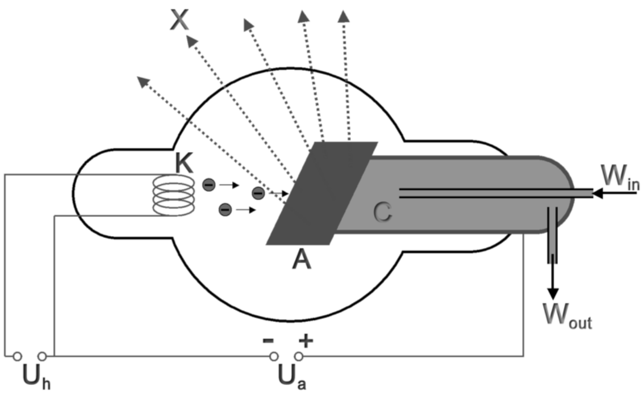


Photo of experimenters taking an X-ray with an early Crookes tube apparatus, from the late 1800s.

Bremsstrahlung





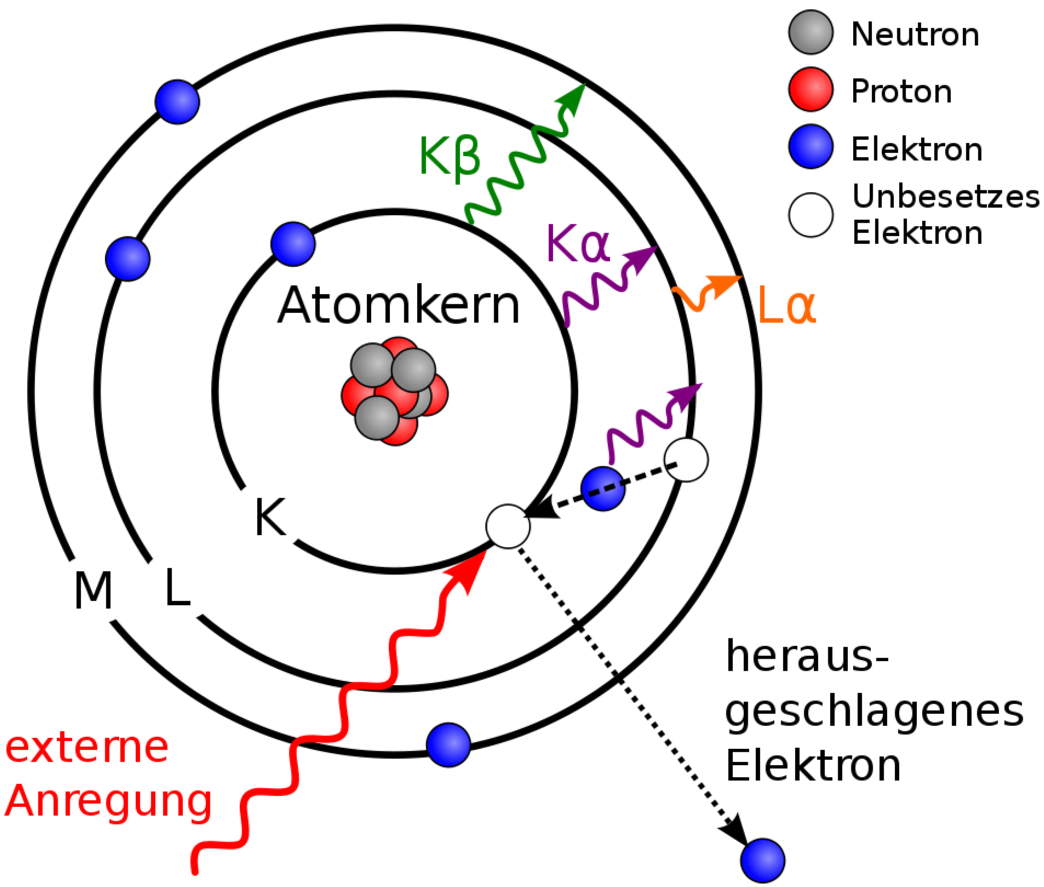
$$E_{\text{Photon}} = \hbar\omega = E_{\text{kinetisch}} = eU$$

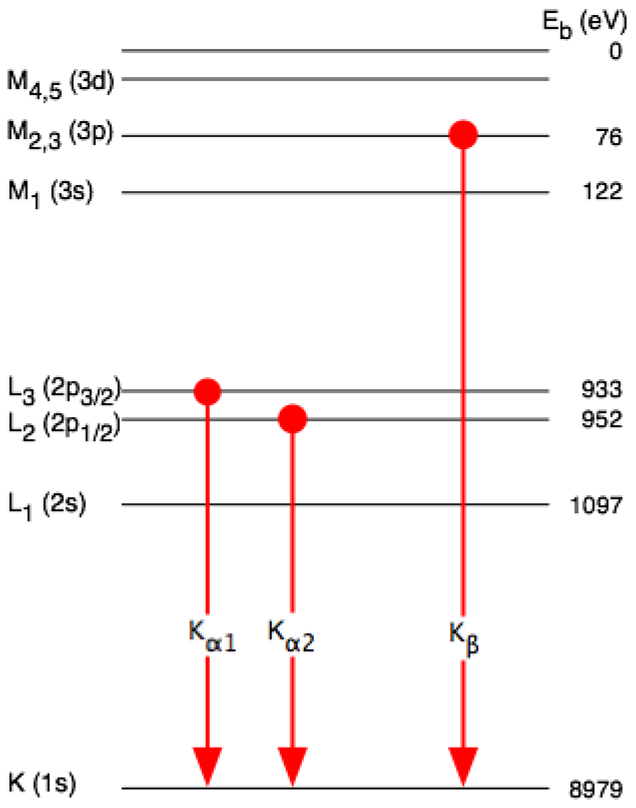
$$\lambda_{\text{min}} = \frac{h \cdot c}{e \cdot U} \qquad \lambda_{\text{min}} = \frac{1{,}24 \cdot 10^{-6}\,\text{V} \cdot \text{m}}{U}$$

$$J(\lambda) = K \cdot I \cdot Z \cdot \left(\frac{\lambda}{\lambda_{\text{min}}} - 1\right) \cdot \frac{1}{\lambda^2}$$

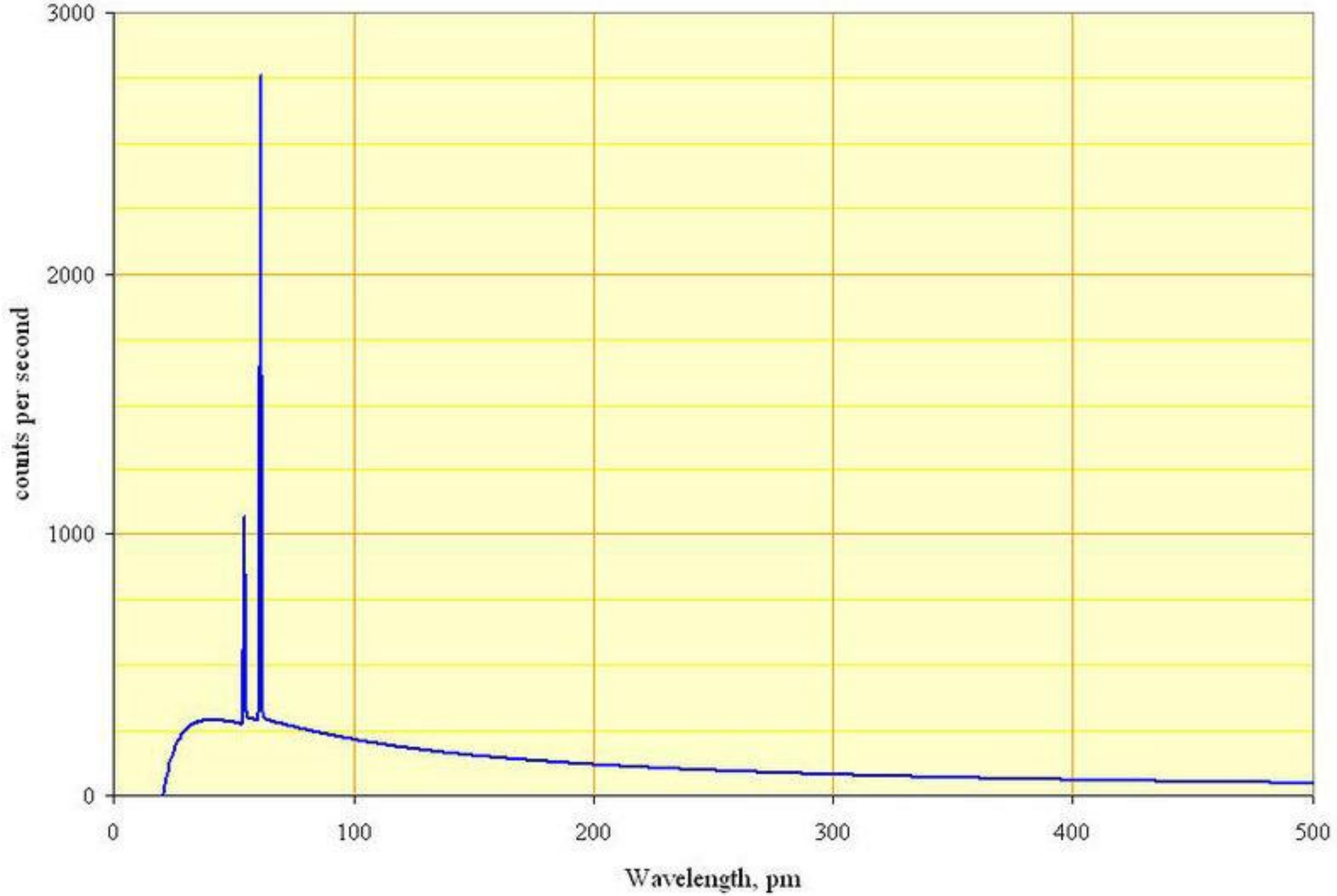
The maximum energy of the produced X-ray photon is limited by the energy of the incident electron, which is equal to the voltage on the tube, so an 80 kV tube cannot create X-rays with an energy greater than 80 keV.

Characteristic radiation





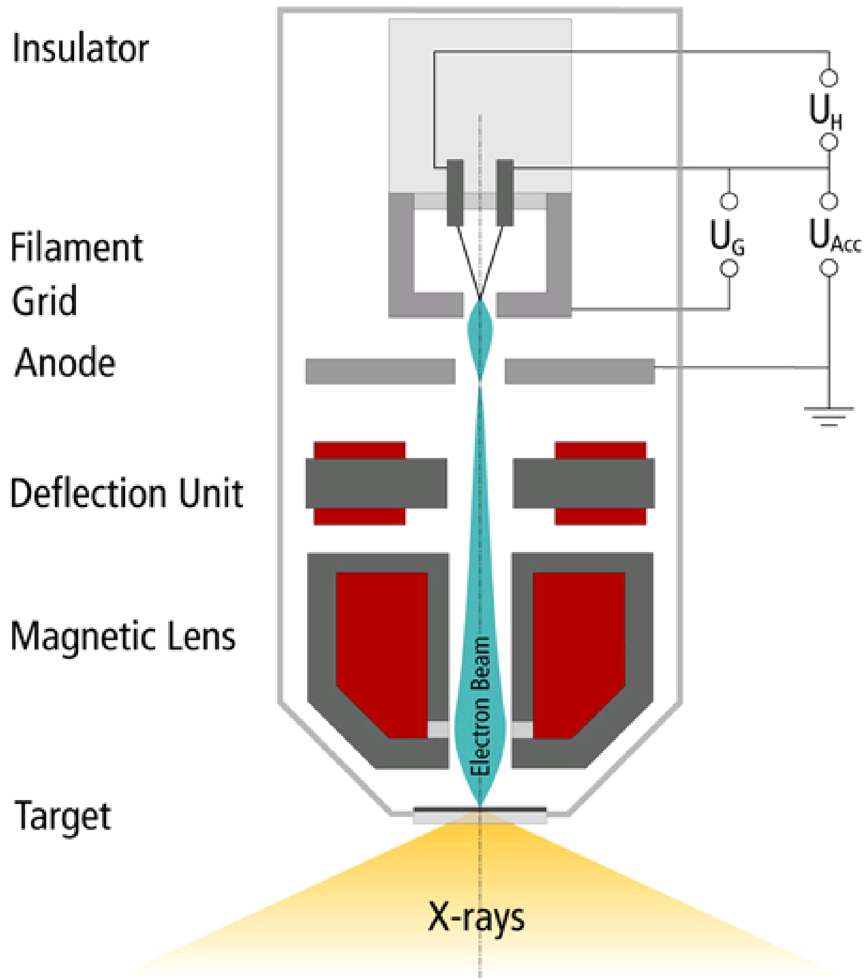
Spectrum



Spectrum of the X-rays emitted by an X-ray tube with a rhodium target, operated at 60 kV.

http://upload.wikimedia.org/wikipedia/commons/5/5c/TubeSpectrum.jpg

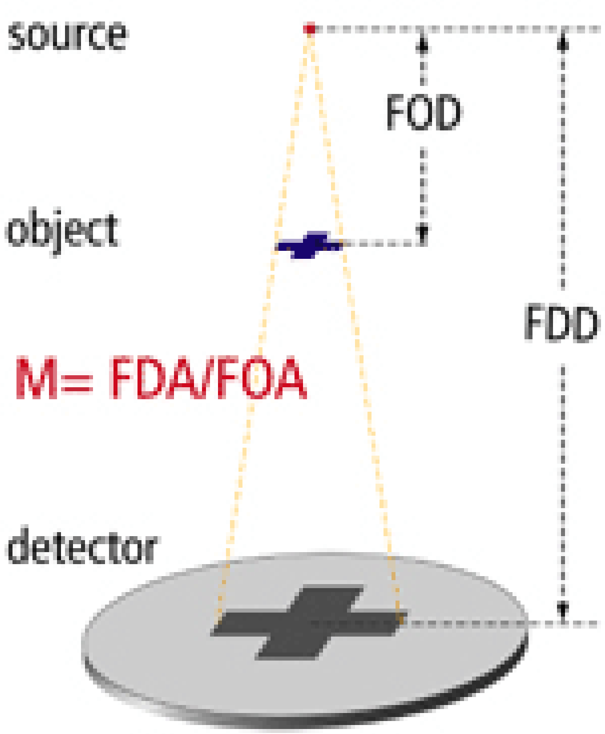
Microfocus tubes



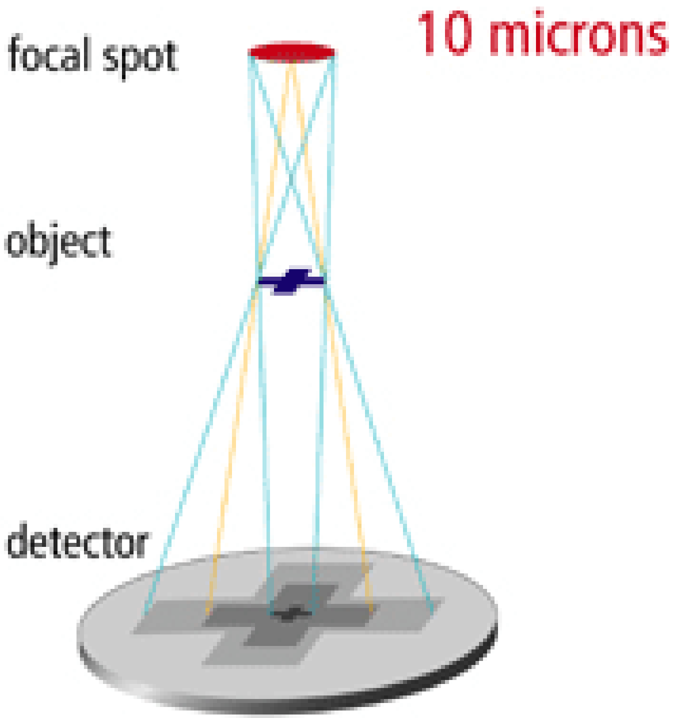
http://www.microfocus-x-ray.com/en/company/technology/principles_of_operation/principle_025.html

Microfocus tubes

Magnification



Resolution



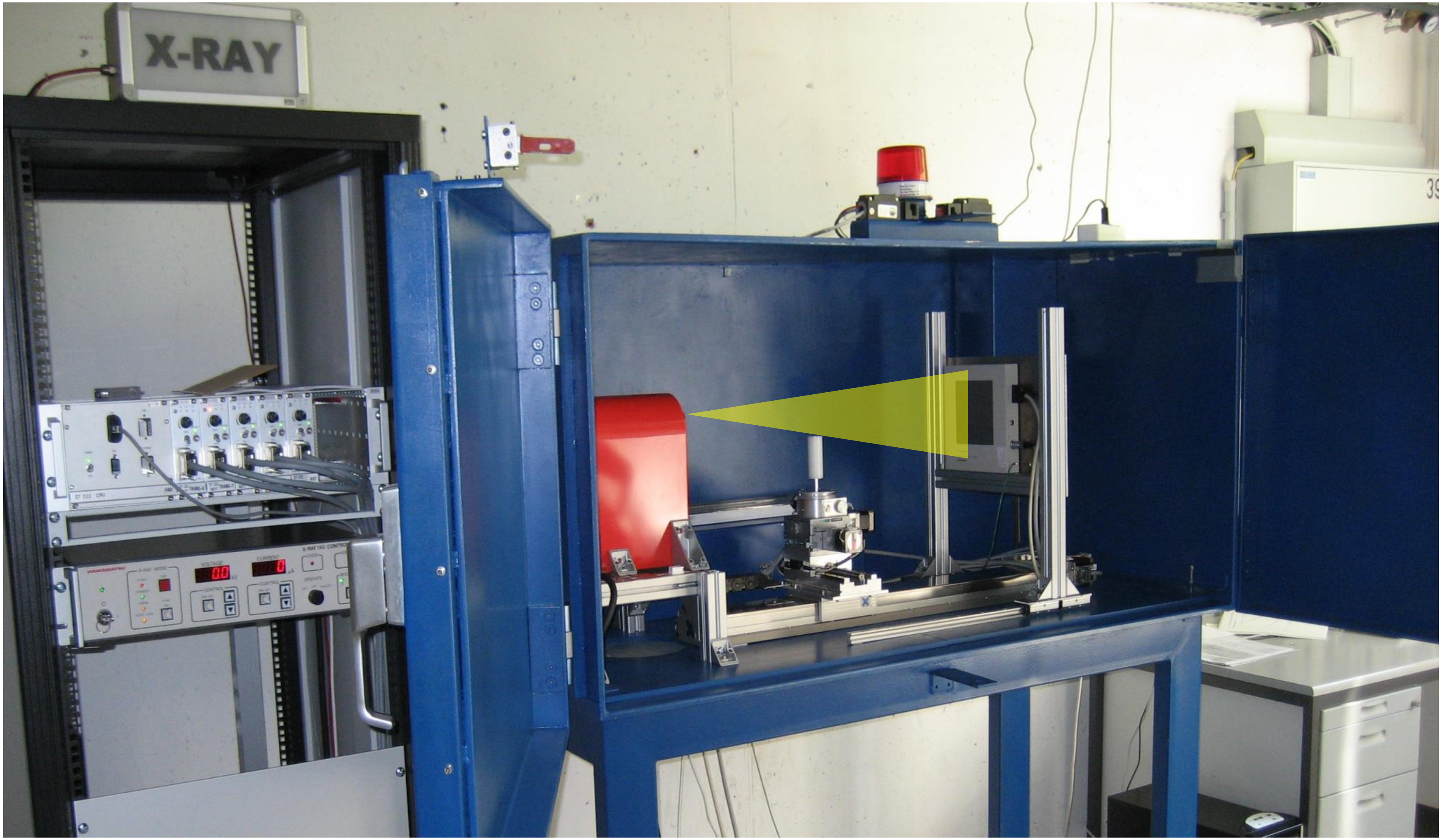
http://www.microfocus-x-ray.com/en/company/technology/principles_of_operation/principle_025.html

150 keV X-ray tube

Sealed Type	
150 kV	
L8121-03	
40 to 150	
Small Spot Mode	0 to 250 (10 W Max.)
Middle Spot Mode	0 to 500 (30 W Max.)
Large Spot Mode	0 to 500
Small Spot Mode	10
Middle Spot Mode	30
Large Spot Mode	75
Small Spot Mode	7 (5 μm at 4 W)
Middle Spot Mode	20
Large Spot Mode	50
43	
25 degrees (ReflectionType)	
17	
20	
Continuous	
CE (IEC61326-1) (IEC61010-1)	

X-ray sources

μ-CT scanner



X-ray applications

- Amber fossils : optically obscured or distorted

http://www.inct.be/en/home

Sample image: X-ray showing frontal view of both hands.

Resolution

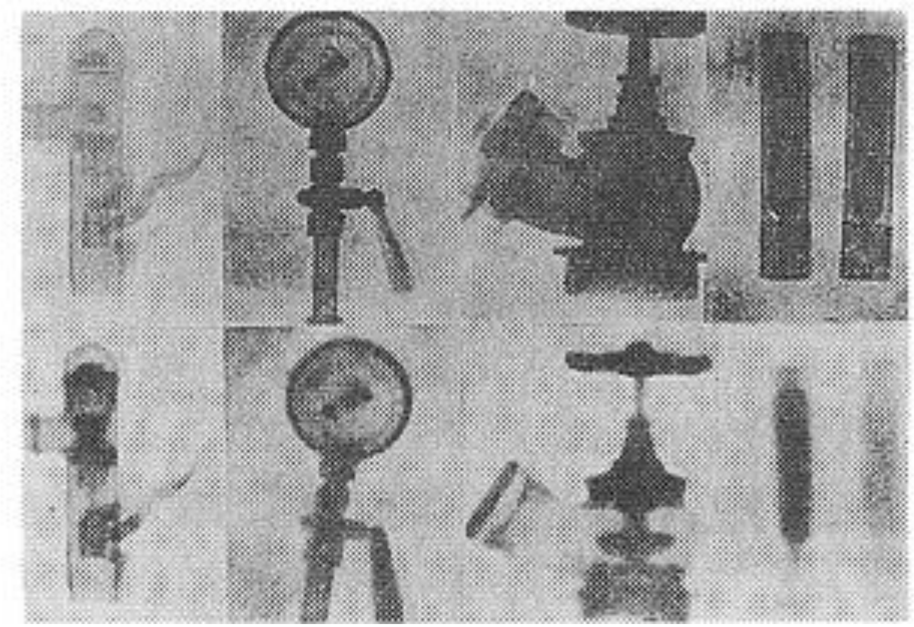
- Beam optimisation
- Detector development

Contrast

- Interaction with matter
  - X-rays
  - Neutrons (sources, interaction, detectors)

Roots of neutron radiography

Taken from C.O. Fischer's article in WCNR-4



As typical: valves, manometers, injectors

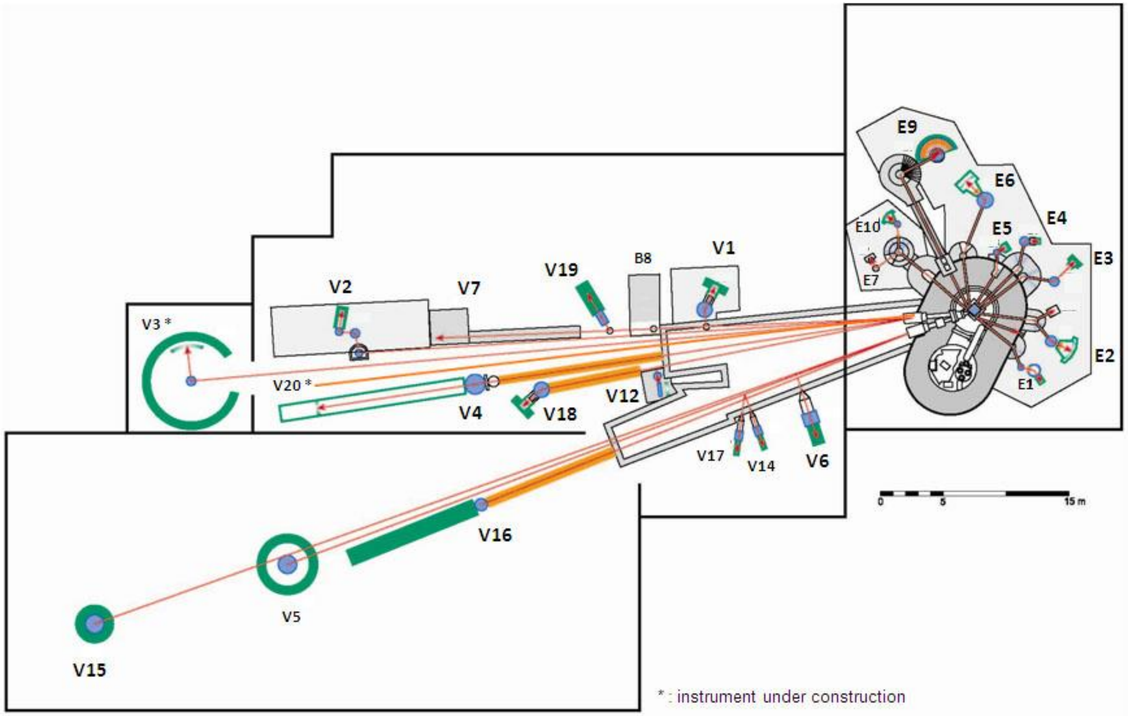
Berlin, 1935 – 1938
H. Kallmann & Kuhn with Ra-Be and neutron generator

Berlin until Dec. 1944
O. Peter with an accelerator neutron source

But the real programs with neutrons started after World War II at research reactors

Neutron sources

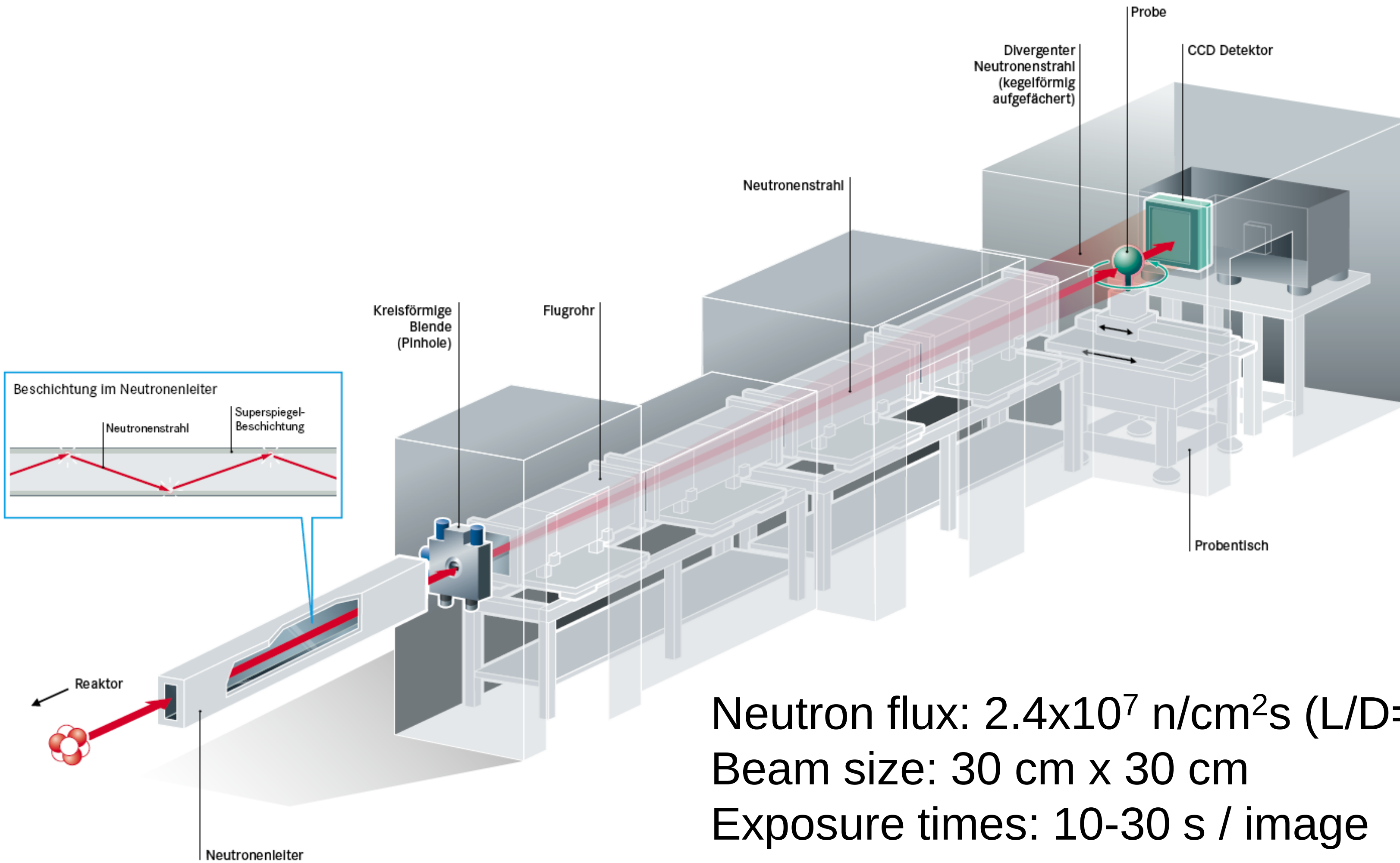
BER-2



Type: open, light-water-moderated swimming pool reactor
Capacity:
more than 10^{14} neutrons per square centimeter per second in the core
10 megawatts thermal power

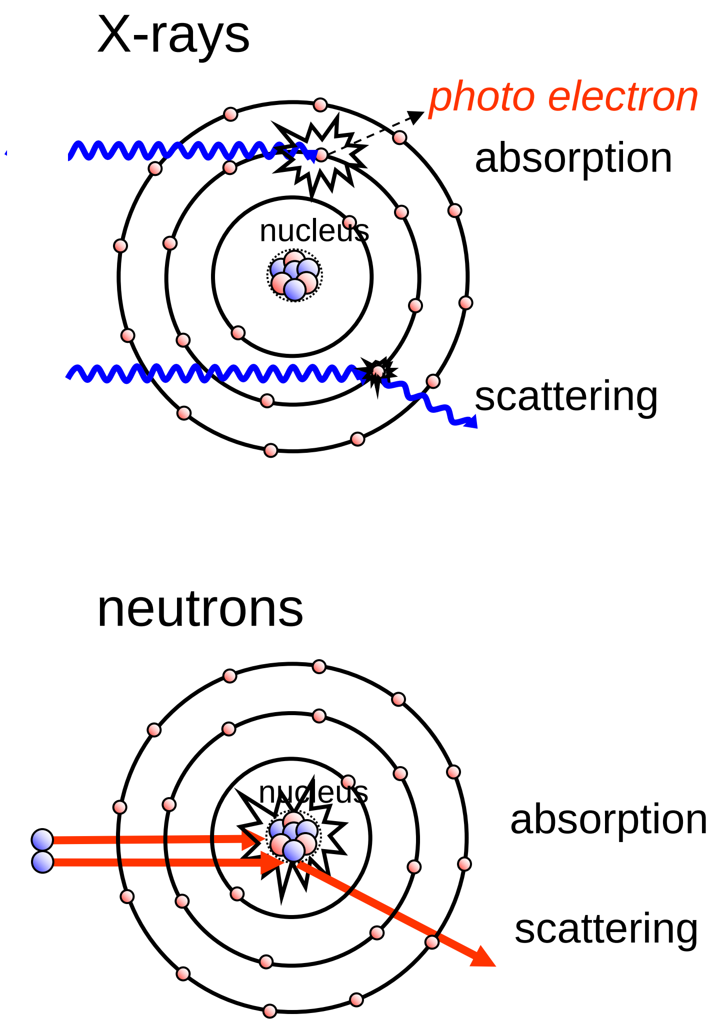
The research reactor BER II is a source of neutron beams for structural and materials research.

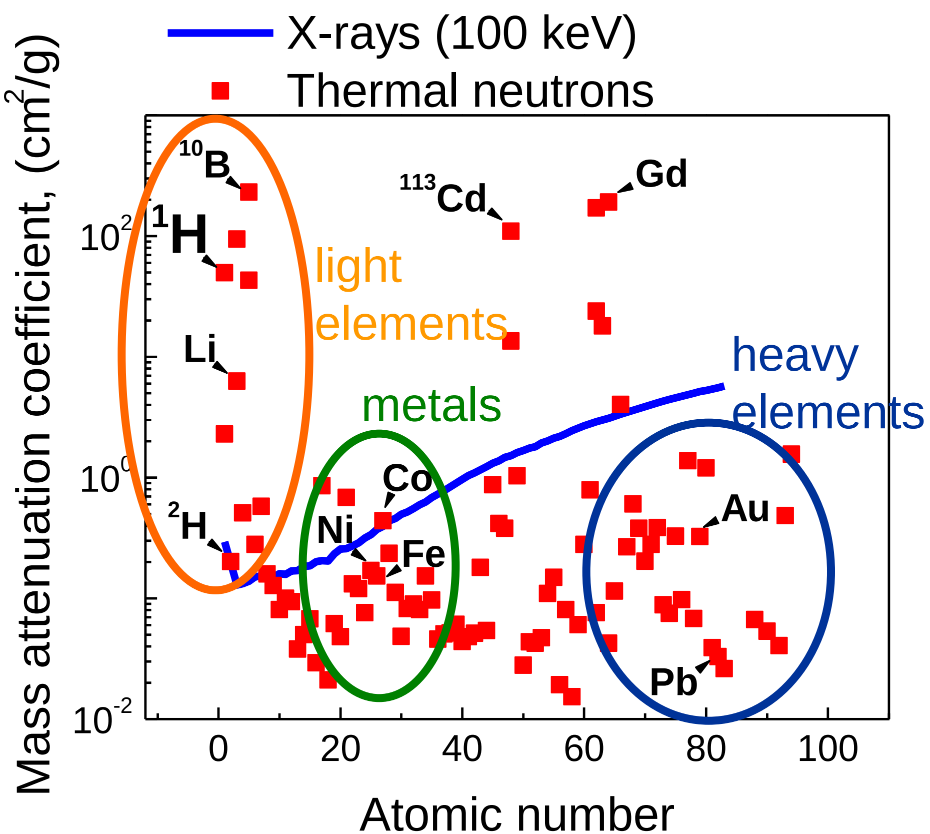
CONRAD-2 Instrument



Neutron flux: 2.4×10^7 n/cm^{2}s (L/D=350)
Beam size: 30 cm x 30 cm
Exposure times: 10-30 s / image

Neutron interaction with matter





Neutron interaction

Attenuation coefficients with X-ray [cm?^{1}]

1a	2a	3b	4b	5b	6b	7b	8			1b	2b	3a	4a	5a	6a	7a	0
H 0.02																	He 0.02
Li 0.06	Be 0.22											B 0.28	C 0.27	N 0.11	O 0.16	F 0.14	Ne 0.17
Na 0.13	Mg 0.24											Al 0.38	Si 0.33	P 0.25	S 0.30	Cl 0.23	Ar 0.20
K 0.14	Ca 0.26	Sc 0.48	Ti 0.73	V 1.04	Cr 1.29	Mn 1.32	Fe 1.57	Co 1.78	Ni 1.96	Cu 1.97	Zn 1.64	Ga 1.42	Ge 1.33	As 1.50	Se 1.23	Br 0.90	Kr 0.73
Rb 0.47	Sr 0.86	Y 1.61	Zr 2.47	Nb 3.43	Mo 4.29	Tc 5.06	Ru 5.71	Rh 6.08	Pd 6.13	Ag 5.67	Cd 4.84	In 4.31	Sn 3.98	Sb 4.28	Te 4.06	I 3.45	Xe 2.53
Cs 1.42	Ba 2.73	La 5.04	Hf 19.70	Ta 25.47	W 30.49	Re 34.47	Os 37.92	Ir 39.01	Pt 38.61	Au 35.94	Hg 25.88	Tl 23.23	Pb 22.81	Bi 20.28	Po 20.22	At	Rn 9.77
Fr	Ra 11.80	Ac 24.47	Rf	Ha													
Lanthanides	Ce 5.79	Pr 6.23	Nd 6.46	Pm 7.33	Sm 7.68	Eu 5.66	Gd 8.69	Tb 9.46	Dy 10.17	Ho 10.91	Er 11.70	Tm 12.49	Yb 9.32	Lu 14.07			
*Actinides	Th 28.95	Pa 39.65	U 49.08	Np	Pu	Am	Cm	Bk	Vf	Es	Fm	Md	No	Lr x-ray			

Legend

Attenuation coefficient [cm?^{1}] = sp.gr. * μ/δ

sp.gr.: Handbook of Chemistry and Physics, 56th Edition 1975-1976.

μ/δ: J. H. Hubbell+ and S. M. Seltzer Ionizing Radiation Division, Physics Laboratory National Institute of Standards and Technology Gaithersburg, MD 20899, http://physics.nist.gov/PhysRefData/XrayMassCoef/tab3.html.

X-ray

Neutron interaction

Attenuation coefficients with neutrons [cm?¹]

1a	2a	3b	4b	5b	6b	7b	8			1b	2b	3a	4a	5a	6a	7a	0
H 3.44																	He 0.02
Li 3.30	Be 0.79											B 101.60	C 0.56	N 0.43	O 0.17	F 0.20	Ne 0.10
Na 0.09	Mg 0.15											Al 0.10	Si 0.11	P 0.12	S 0.06	Cl 1.33	Ar 0.03
K 0.06	Ca 0.08	Sc 2.00	Ti 0.60	V 0.72	Cr 0.54	Mn 1.21	Fe 1.19	Co 3.92	Ni 2.05	Cu 1.07	Zn 0.35	Ga 0.49	Ge 0.47	As 0.67	Se 0.73	Br 0.24	Kr 0.61
Rb 0.08	Sr 0.14	Y 0.27	Zr 0.29	Nb 0.40	Mo 0.52	Tc 1.76	Ru 0.58	Rh 10.88	Pd 0.78	Ag 4.04	Cd 115.11	In 7.58	Sn 0.21	Sb 0.30	Te 0.25	I 0.23	Xe 0.43
Cs 0.29	Ba 0.07	La 0.52	Hf 4.99	Ta 1.49	W 1.47	Re 6.85	Os 2.24	Ir 30.46	Pt 1.46	Au 6.23	Hg 16.21	Tl 0.47	Pb 0.38	Bi 0.27	Po	At	Rn
Fr	Ra 0.34	Ac	Rf	Ha													
*Lanthanides	Ce 0.14	Pr 0.41	Nd 1.87	Pm 5.72	Sm 171.47	Eu 94.58	Gd 1479.04	Tb 0.93	Dy 32.42	Ho 2.25	Er 5.48	Tm 3.53	Yb 1.40	Lu 2.75			
**Actinides	Th 0.59	Pa 8.46	U 0.82	Np 9.80	Pu 50.20	Am 2.86	Cm	Bk	Cf	Es	Fm	Md	No	Lr neut.			

Legend

$$\text{Attenuation coefficient [cm?}^{1}\text{]} = \frac{\sigma\text{-total} * \text{sp.gr.} * 0.6023}{\text{at.wt.}}$$

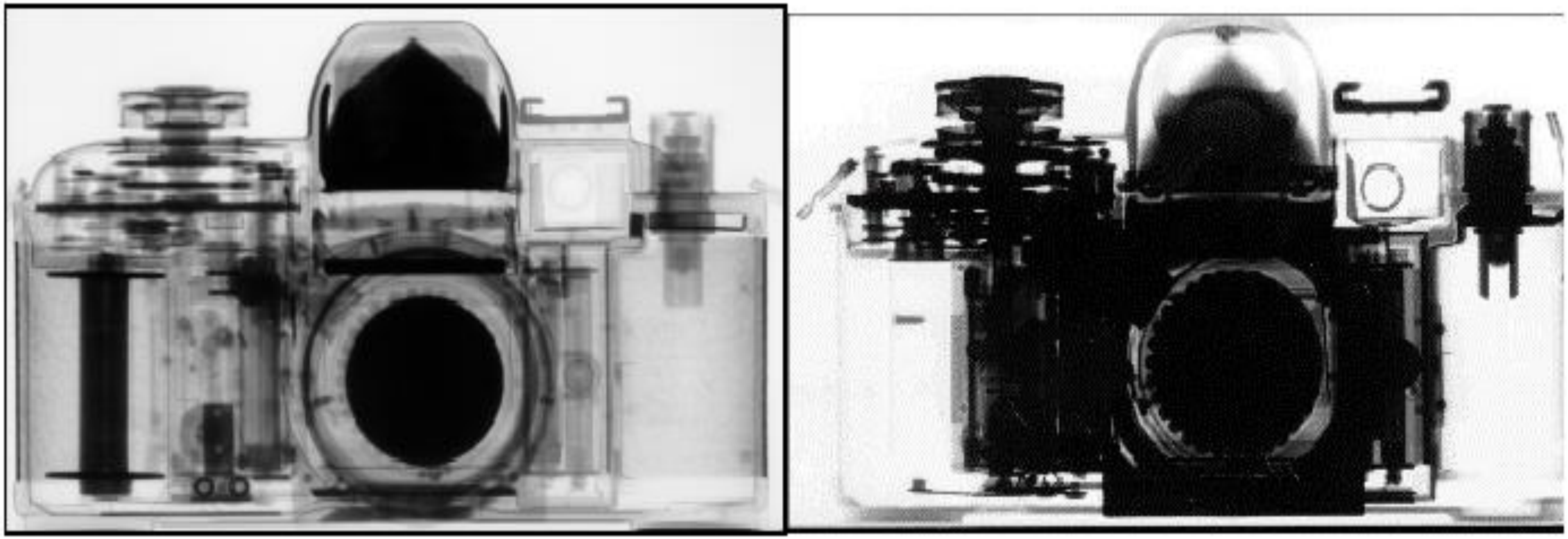
thermal neutrons

σ-total: JEF Report 14, TABLE OF SIMPLE INTEGRAL NEUTRON CROSS SECTION DATA FROM JEF-2.2, ENDF/B-VI, JENDL-3.2, BROND-2 AND CENDL-2, AEN NEA, 1994.
and Special Feature: Neutron scattering lengths and cross sections, Varley F. Sears, AECL Research, Chalk River Laboratories Chalk River, Ontario, Canada KOJ 1JO, Neutron News, Vol. 3, 1992, http://www.ncnr.nist.gov/resources/n-lengths/list.html.

sp.gr.: Handbook of Chemistry and Physics, 56th Edition 1975-1976.

at.wt.: Handbook of Chemistry and Physics, 56th Edition 1975-1976.

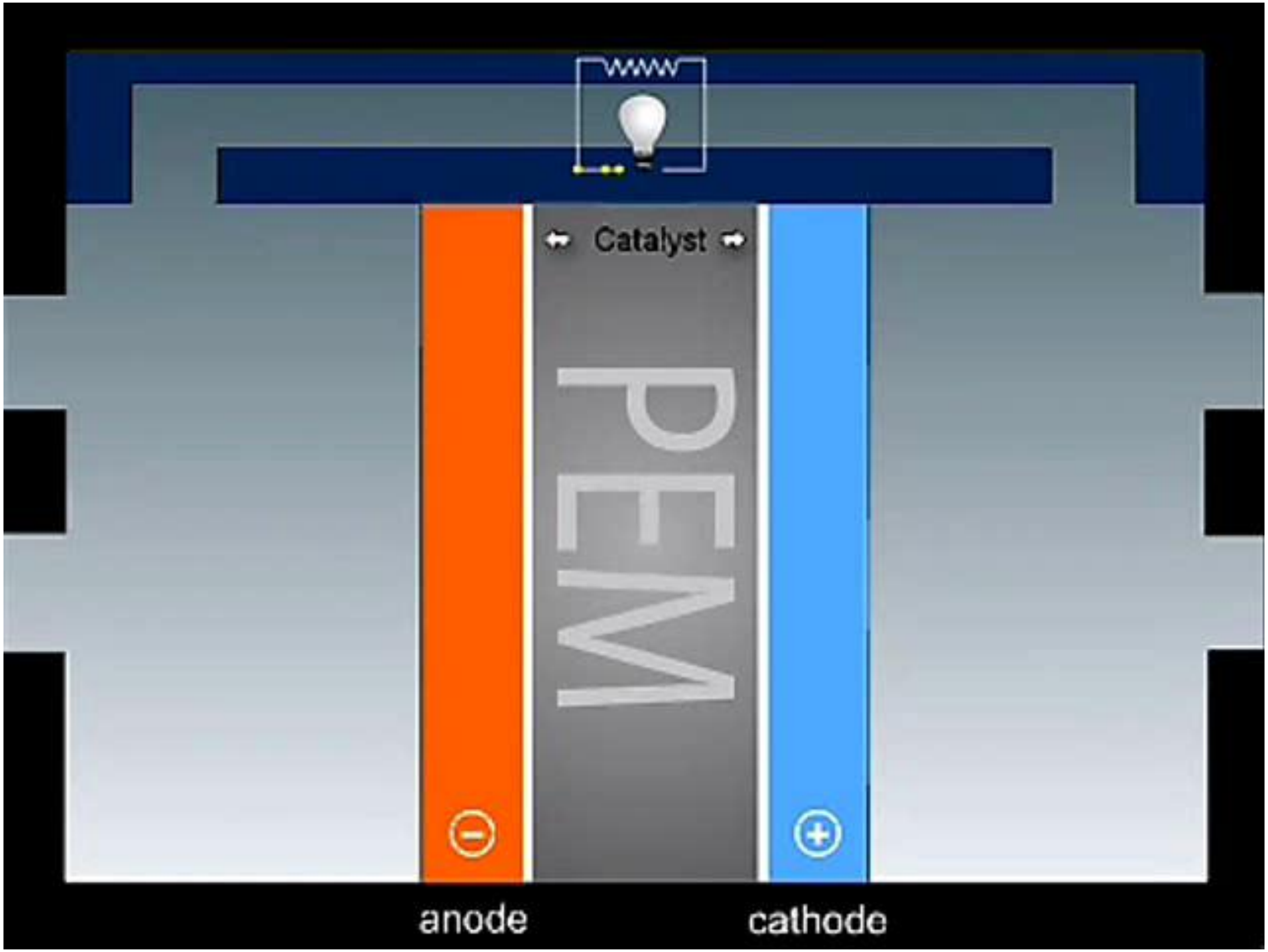
Neutron radiography - examples



The example for a camera helps to explain differences in neutron (left) and X-ray (right) radiography. Whereas the hydrogen containing parts can be visualised with neutron even at thin layers, thicker metallic components are hard to penetrate with X-rays.

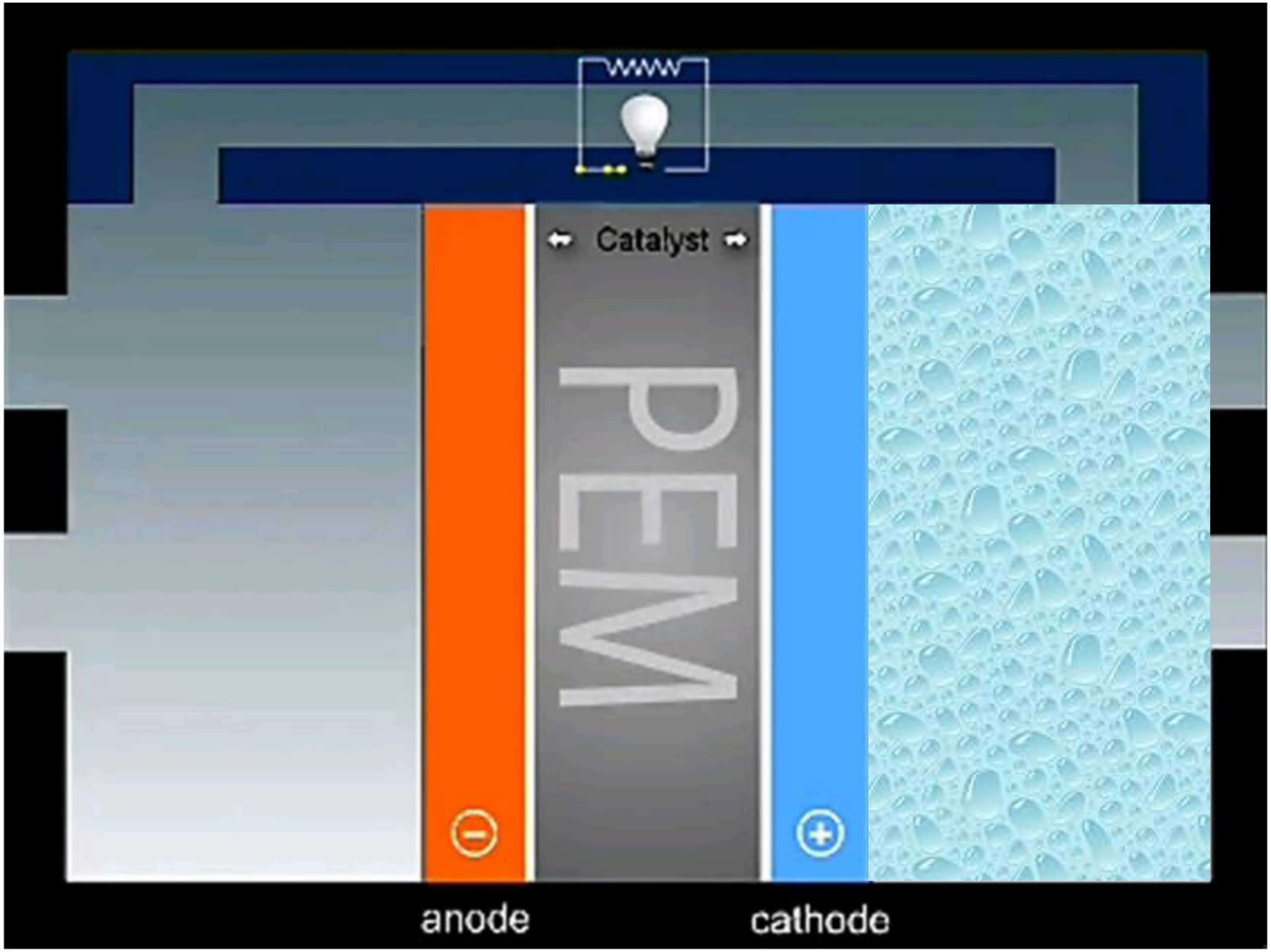
Attenuation Contrast

Fuel cells



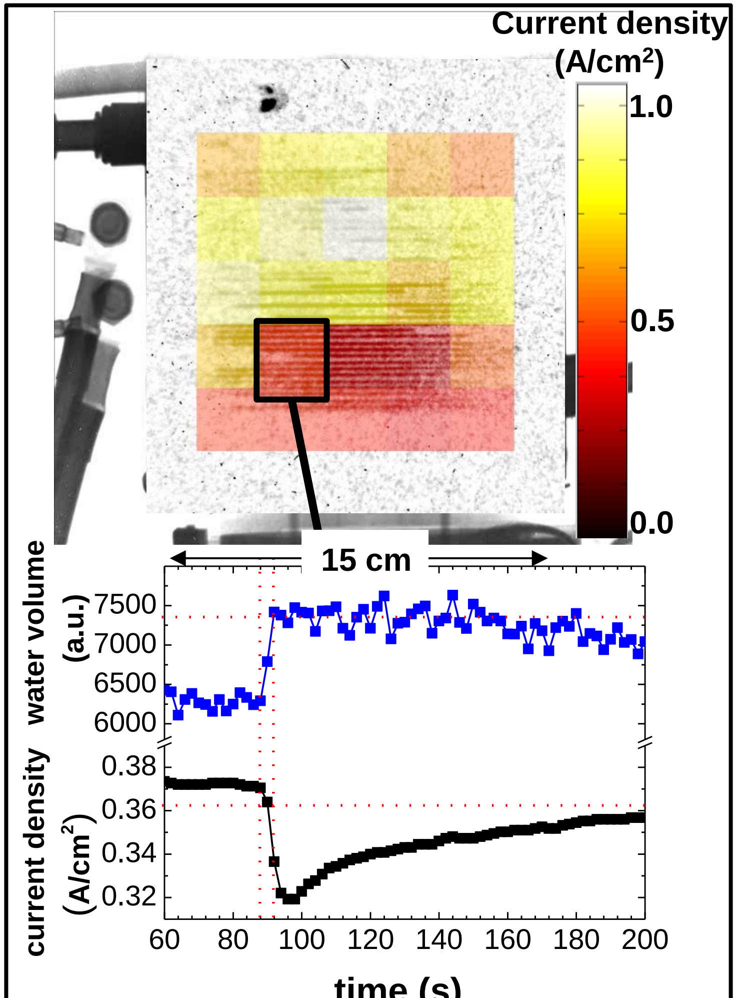
Attenuation Contrast

Fuel cells

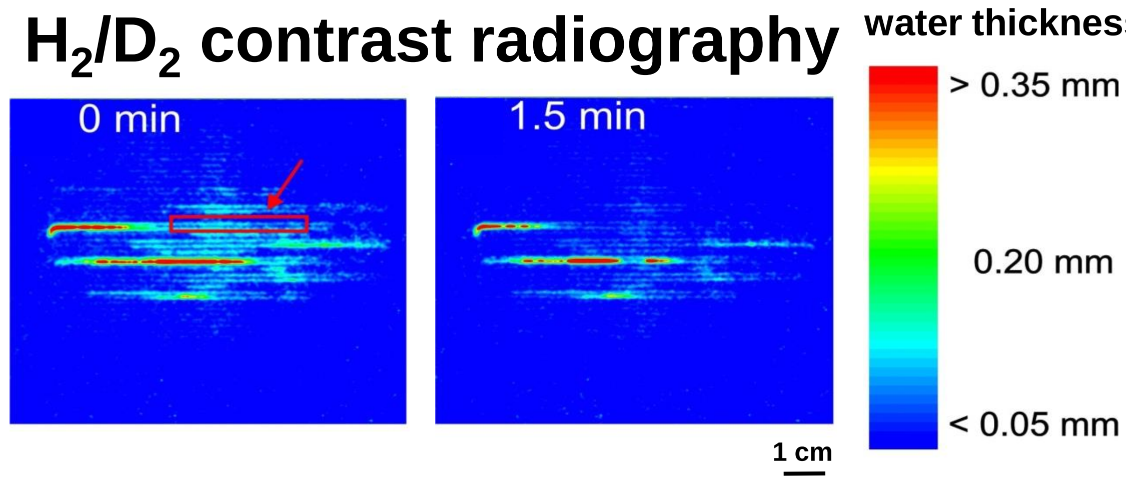


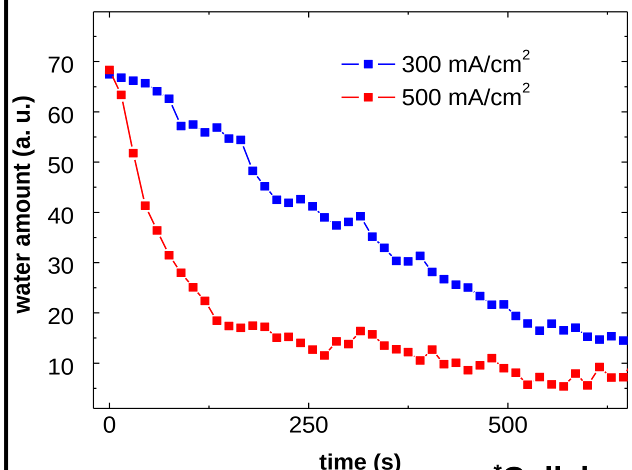
Attenuation Contrast

Fuel cells



H_2/D_2 contrast radiography



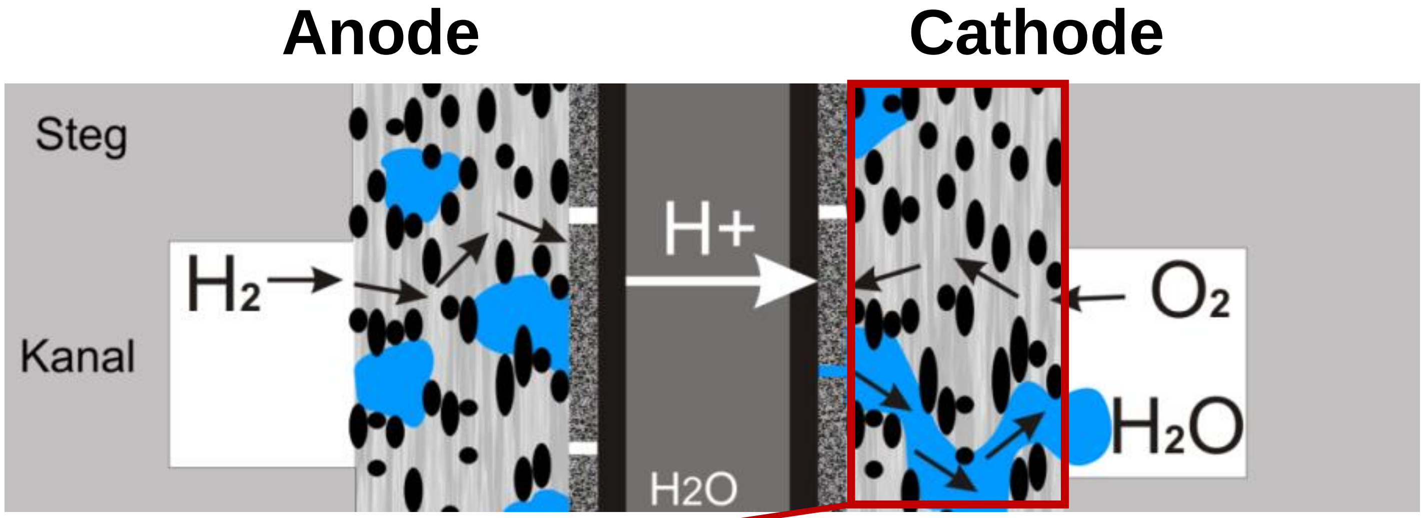


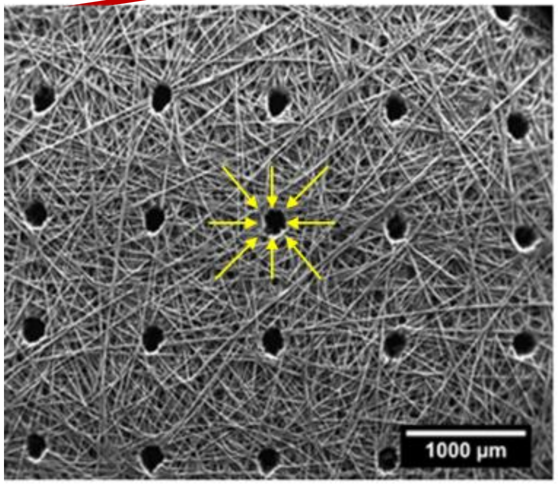
I. Manke et al., APL 90, 174105 (2007)
I. Manke et al, APL 90, 184101 (2007)
Ch. Hartnig et al., APL 92, 134106 (2008)
I. Manke et al., APL 92, 244101 (2008)

*Collaboration work with ZSW, Ulm

Attenuation Contrast

Fuel Cells

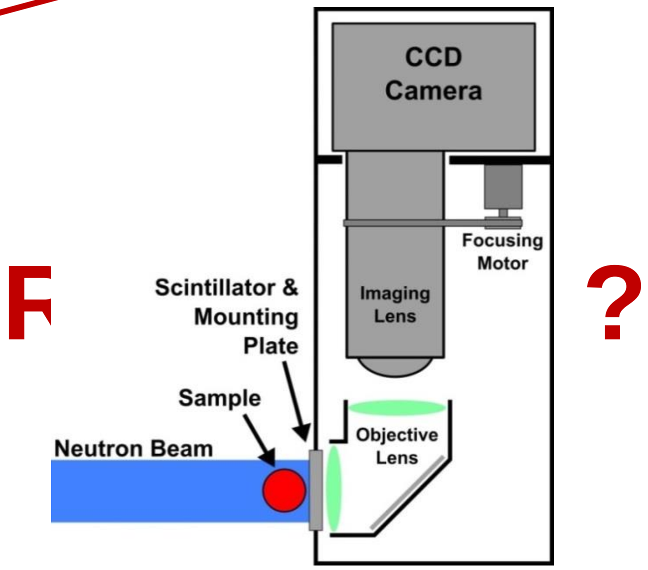




Perforation = Drainage effect

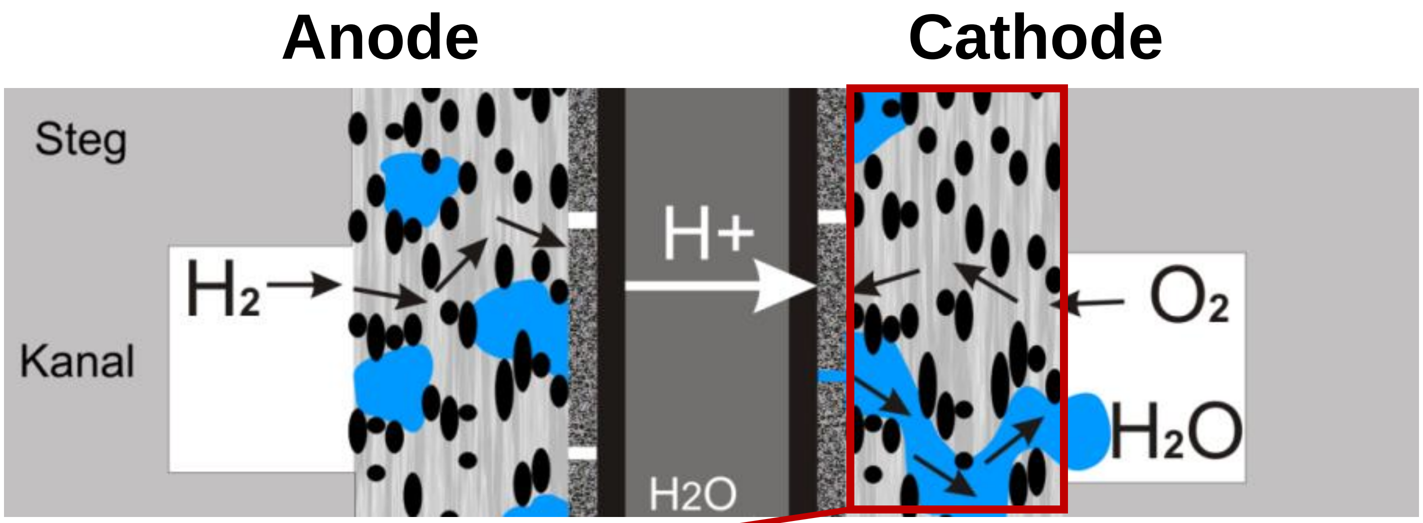
Innovative design

Hydrophobic material



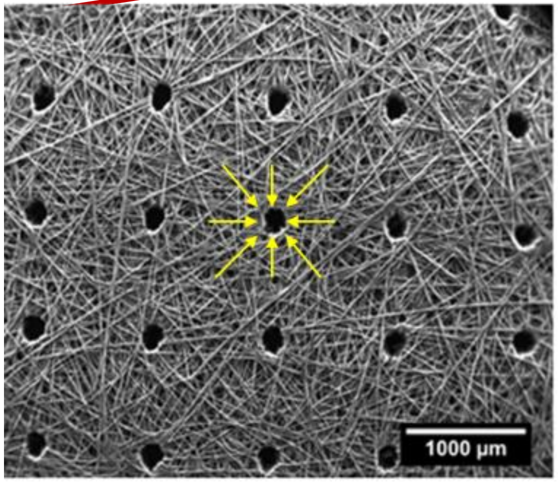
Attenuation Contrast

Fuel Cells

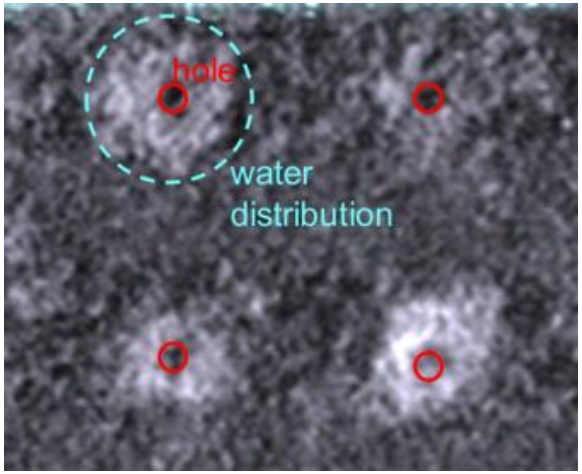


Innovative design

Hydrophobic material



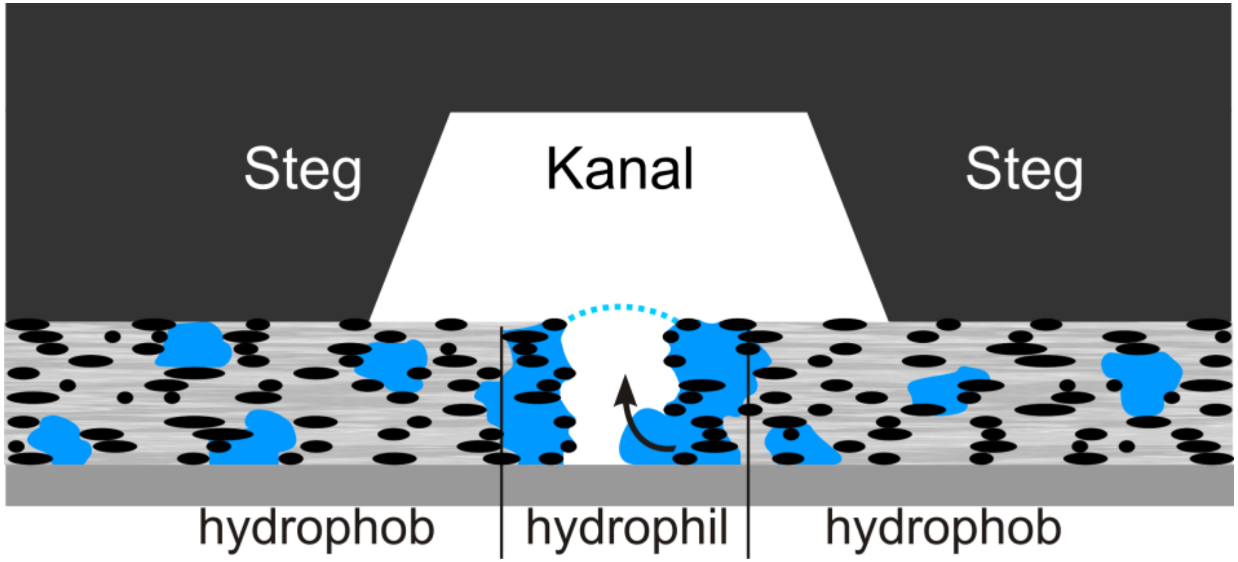
Perforation = Drainage effect

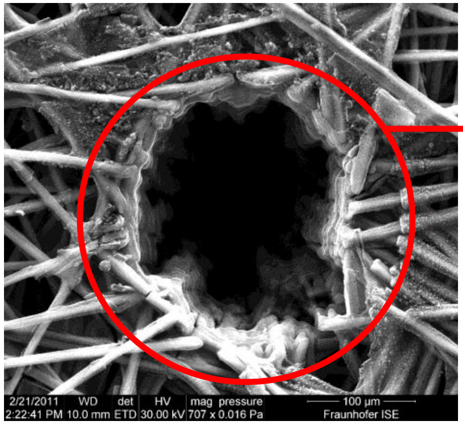


Neutron image

BER II

Fuel Cells



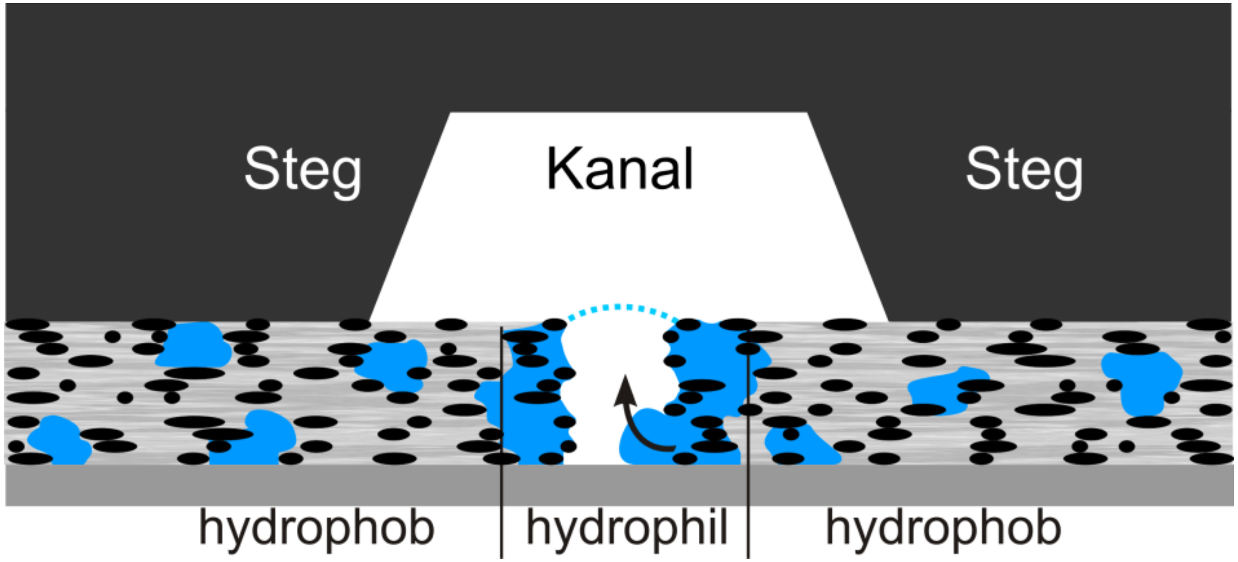


Heat affected zone

➡ **Hydrophilic areas cause water agglomerations**

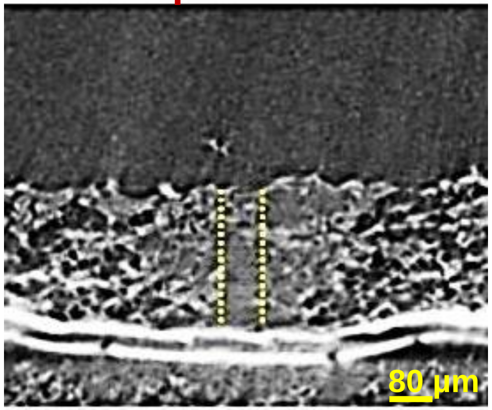
Attenuation Contrast

Fuel Cells

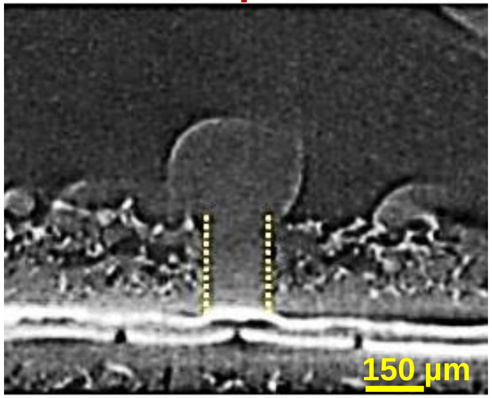


BESSY II

laser perforation



mechanic perforation

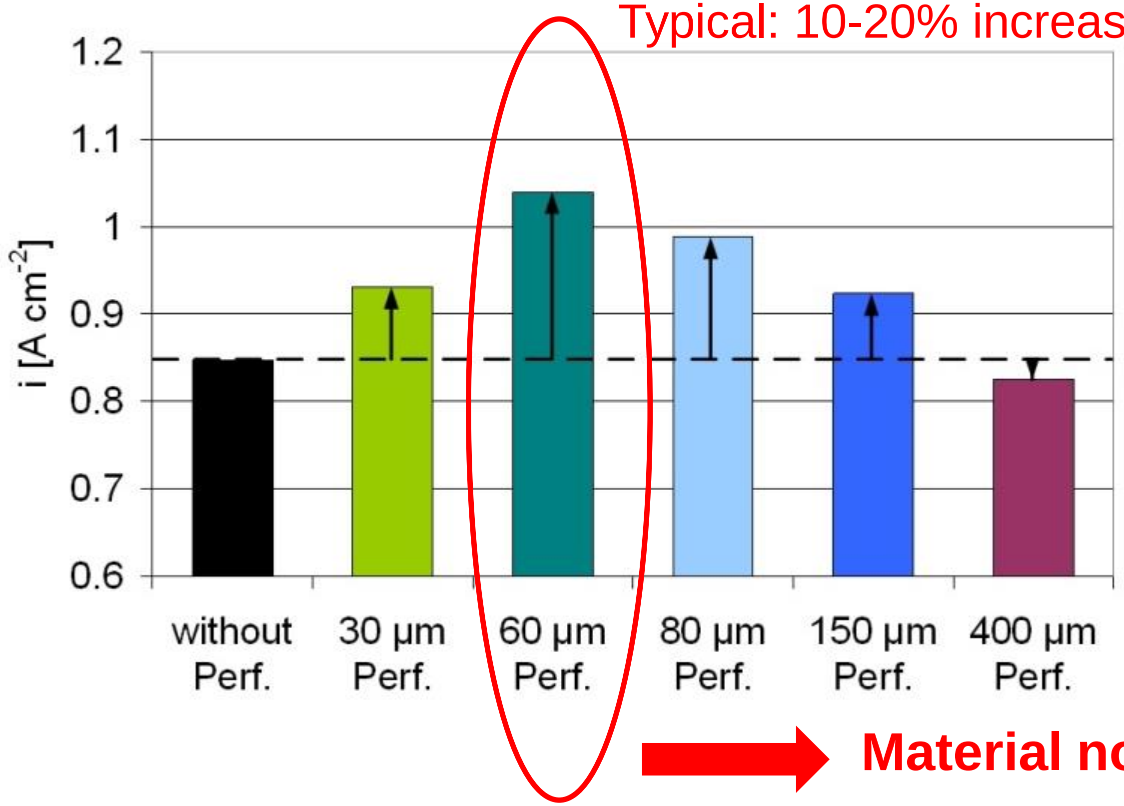


dynamic synchrotron radiography

Attenuation Contrast

Fuel Cells

Best case: 40% performance increase
Typical: 10-20% increase



Material now in production

J. Haußmann *et al*
Journal of Power Sources 239 (2013) 611

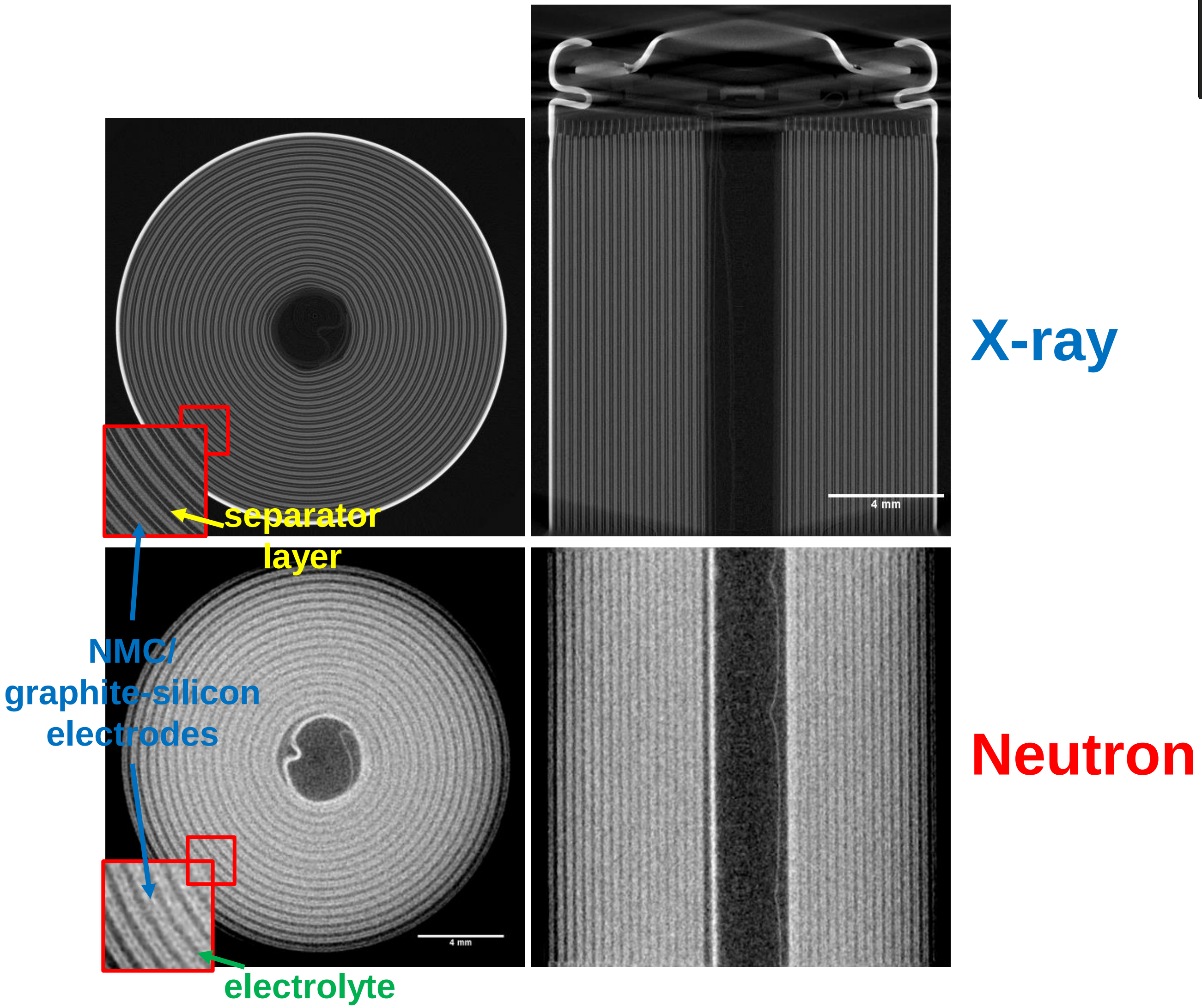
LG MJ1 18650 – NMC / graphite-silicon

Science & Technology Facilities Council ISIS

UCL

electrochemical innovation lab

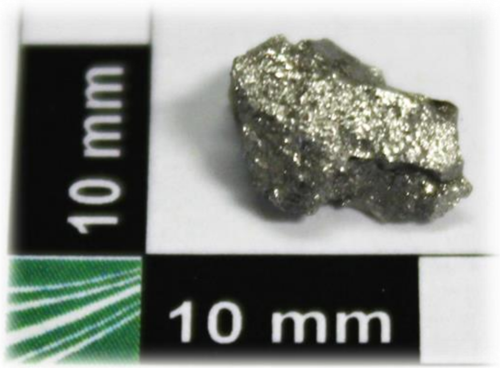
R. Ziesche
W. Kockelmann
P. Shearing



<u>MJ1:</u> Li-ion cell with NMC cathode and new graphite-silicon anode for a high capacity of 3500 mAh.

Fraunhofer IFAM
Lars Röntzsch
Felix Hubner

1. Introduction - Metal-Hydride-Composites (MHC)



	Powder bed	MHC
Second phase	-	5 wt.% Graphite
Thermal conductivity	~1W/mK	14 W/mK (radial)
System load time	>1h	< 10 min
Porosity	~ 70 vol.%	**30 vol.%**

Challenge:

Volume expansion of the crystal lattice

↓

Volume expansion and stability of MHCs?

Stress generation on container walls?

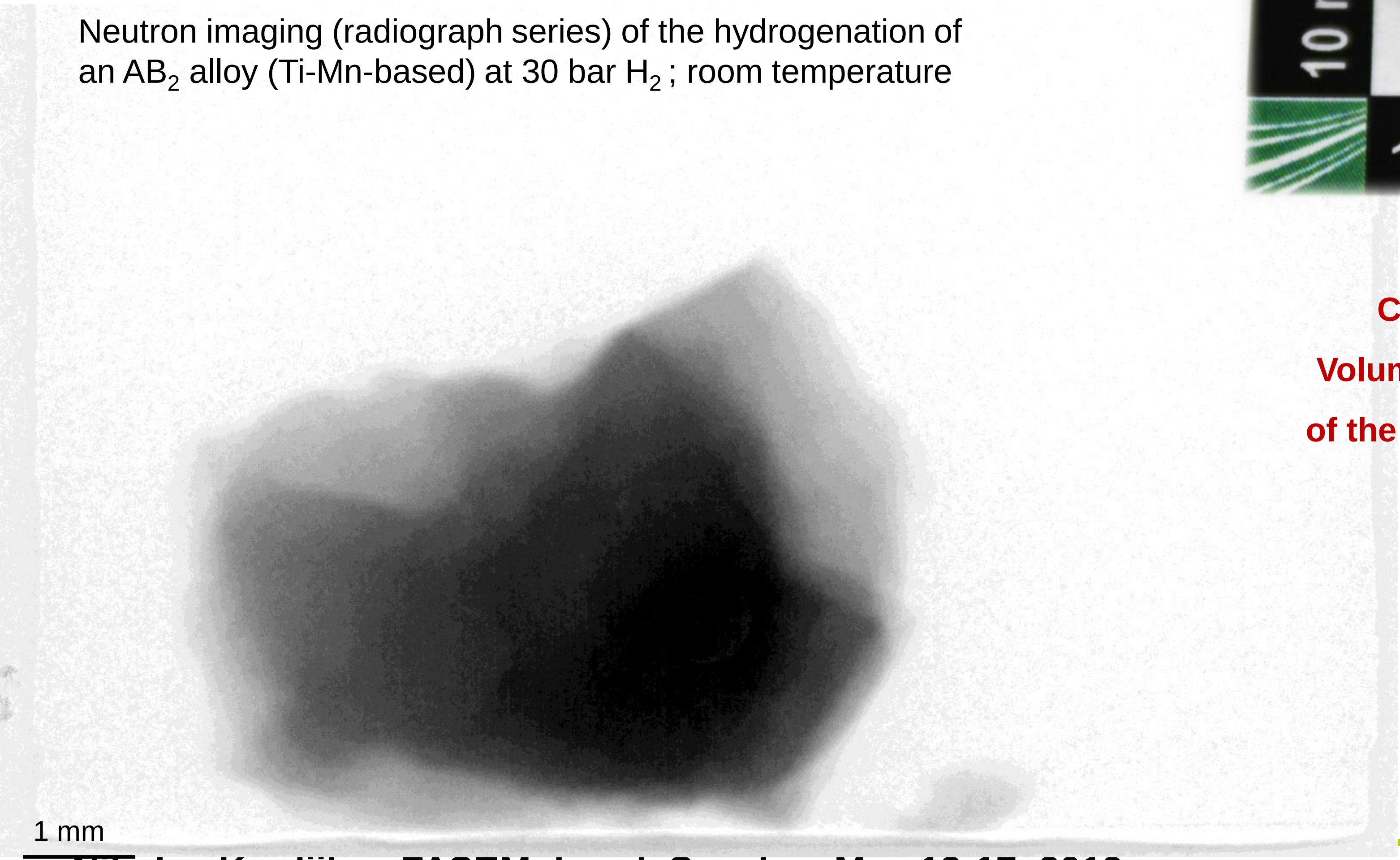
F. Heubner, et al. Journal of Power Sources 397, 262-270

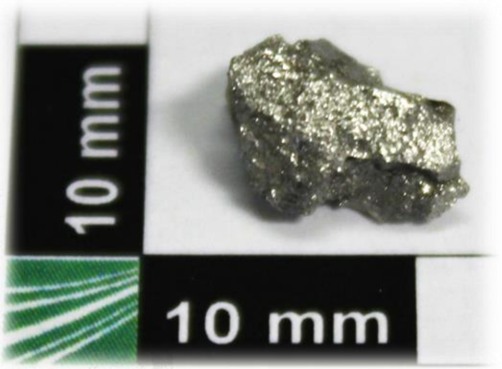
HYDROGEN STORAGE

Fraunhofer IFAM
Lars Röntzsch
Felix Hubner

1. Introduction

Neutron imaging (radiograph series) of the hydrogenation of an AB_2 alloy (Ti-Mn-based) at 30 bar H_2 ; room temperature





Challenge:

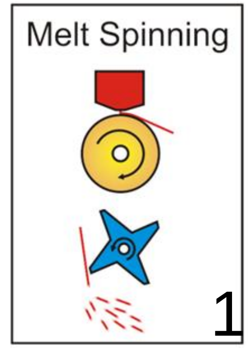
Volume expansion

of the crystal lattice

Lars Röntzsch
Felix Hubner

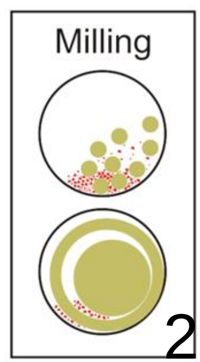
2. Materials Processing

Granules



melt spinning (flakes)

milling (powder)



Hydralloy → Ti-Mn-V-Fe-Zr Alloy	
H-capacity	~1.8 wt.%
reaction enthalpy	~ 27 kJ/mol
working temperature	-10 ... 100°C

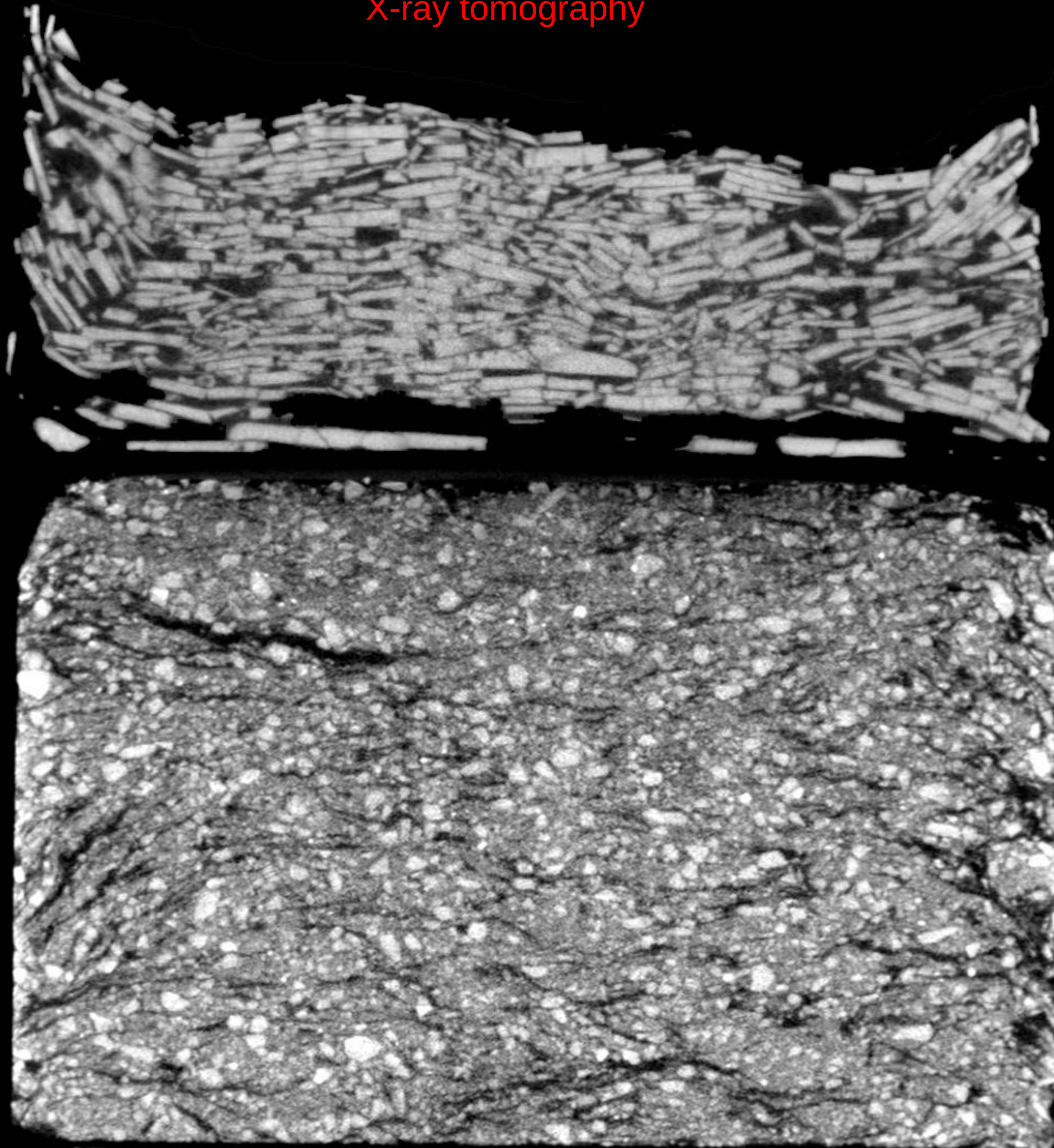
[1]Pohlmann et al. (2011), J of Alloys and Compounds 509, p. 625-628
[2]Pohlmann et al. (2013), J of Power Sources 231, p. 97-105

X-ray tomography

Flake MHC
(radially aligned flakes)

Porosity:
27-30 vol.%

Graphite:
10 vol.%



Powder MHC

1 mm

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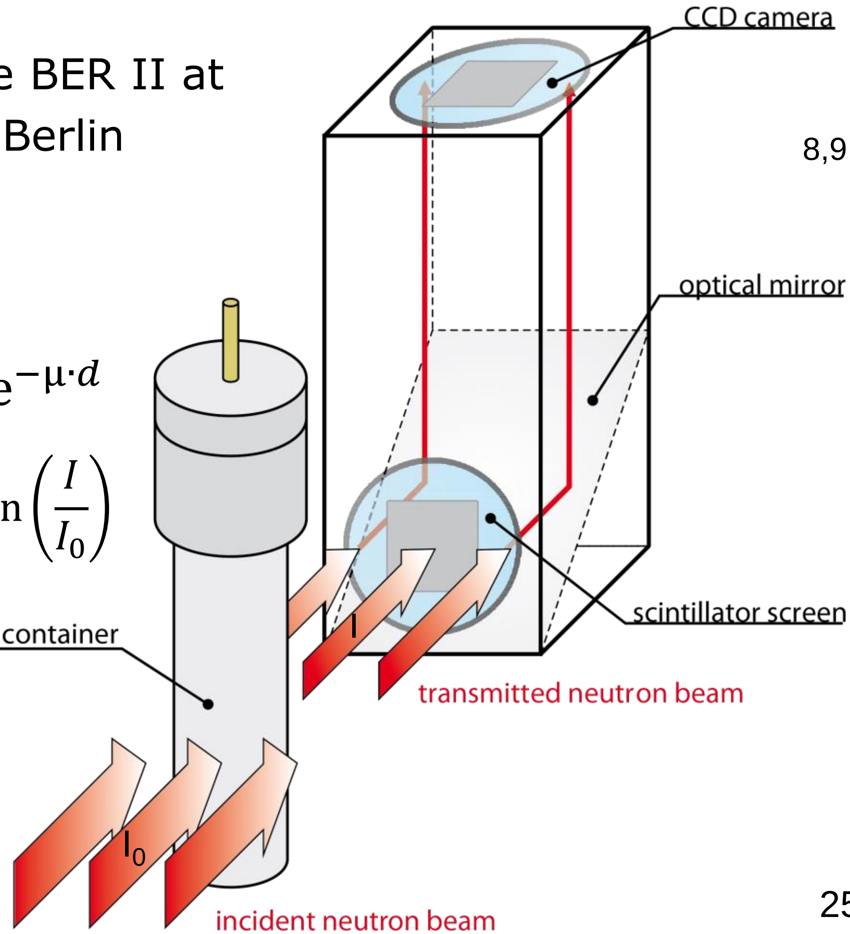
3. Neutron Imaging

- Cold Neutron Source BER II at Helmholtz-Zentrum Berlin
- Beamline (V7): Radio-/Tomography

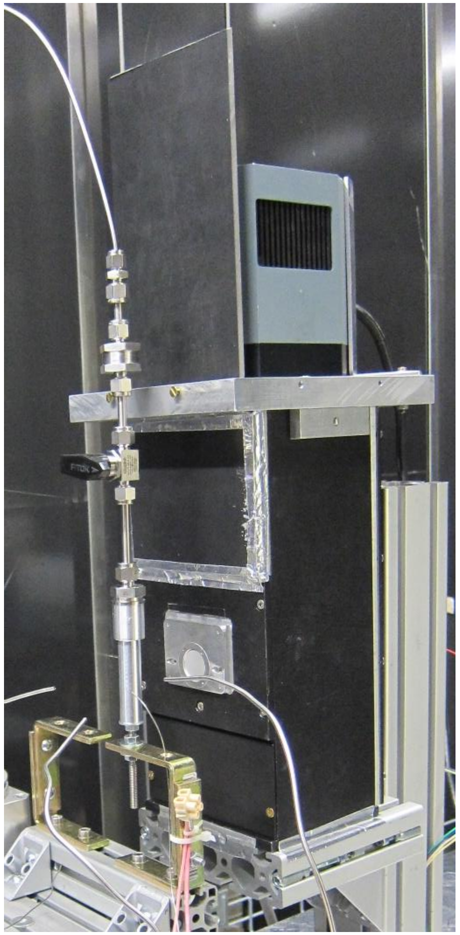
Lambert Beer: $I = I_0 \cdot e^{-\mu \cdot d}$

Extinction: $E = -\mu \cdot d = \ln\left(\frac{I}{I_0}\right)$

diameter=12mm
height=6-7mm



8,9



2560 x 2160 pixels (16 bit)
Resolution per pixel: 5.4 µm

[8]Pohlmann et al. (2015), J of Power Sources 277, p. 360-369
[9]Herbrig et al. (2015), J of Power Sources 293, p. 109-118

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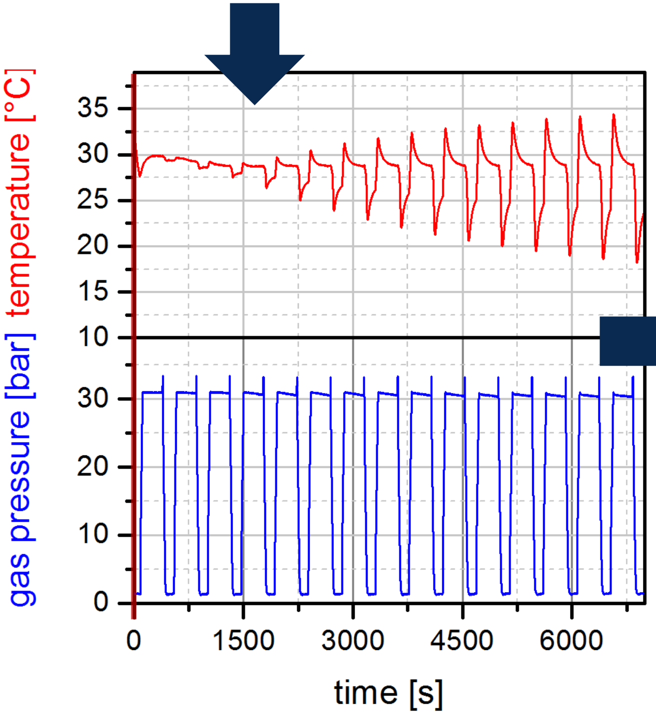
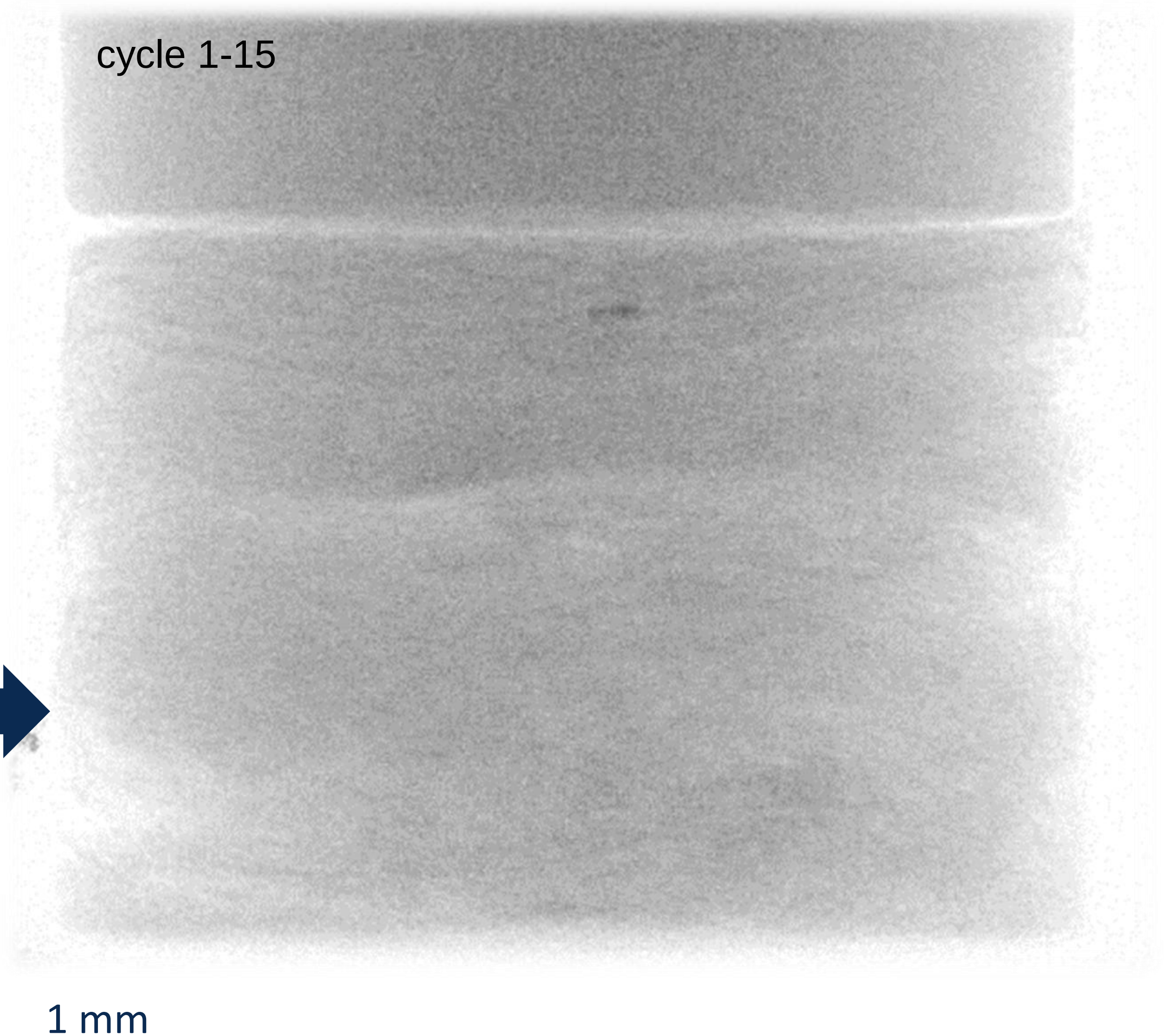
Felix Hubner

3.1 Flake MHC

1. Activation
   2x heating up to 150˚C

2. Cycling
   30/1.3 bar, RT





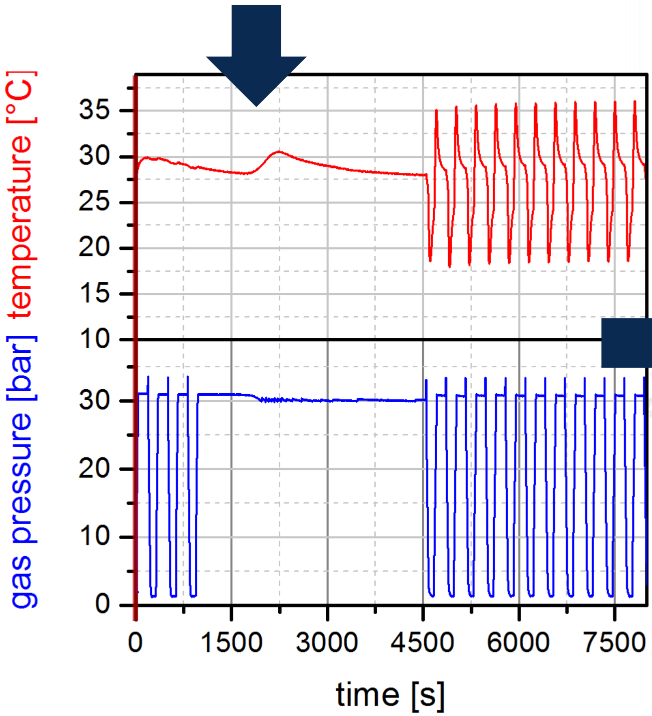
F. Heubner, et al. Journal of Power Sources 397, 262-270

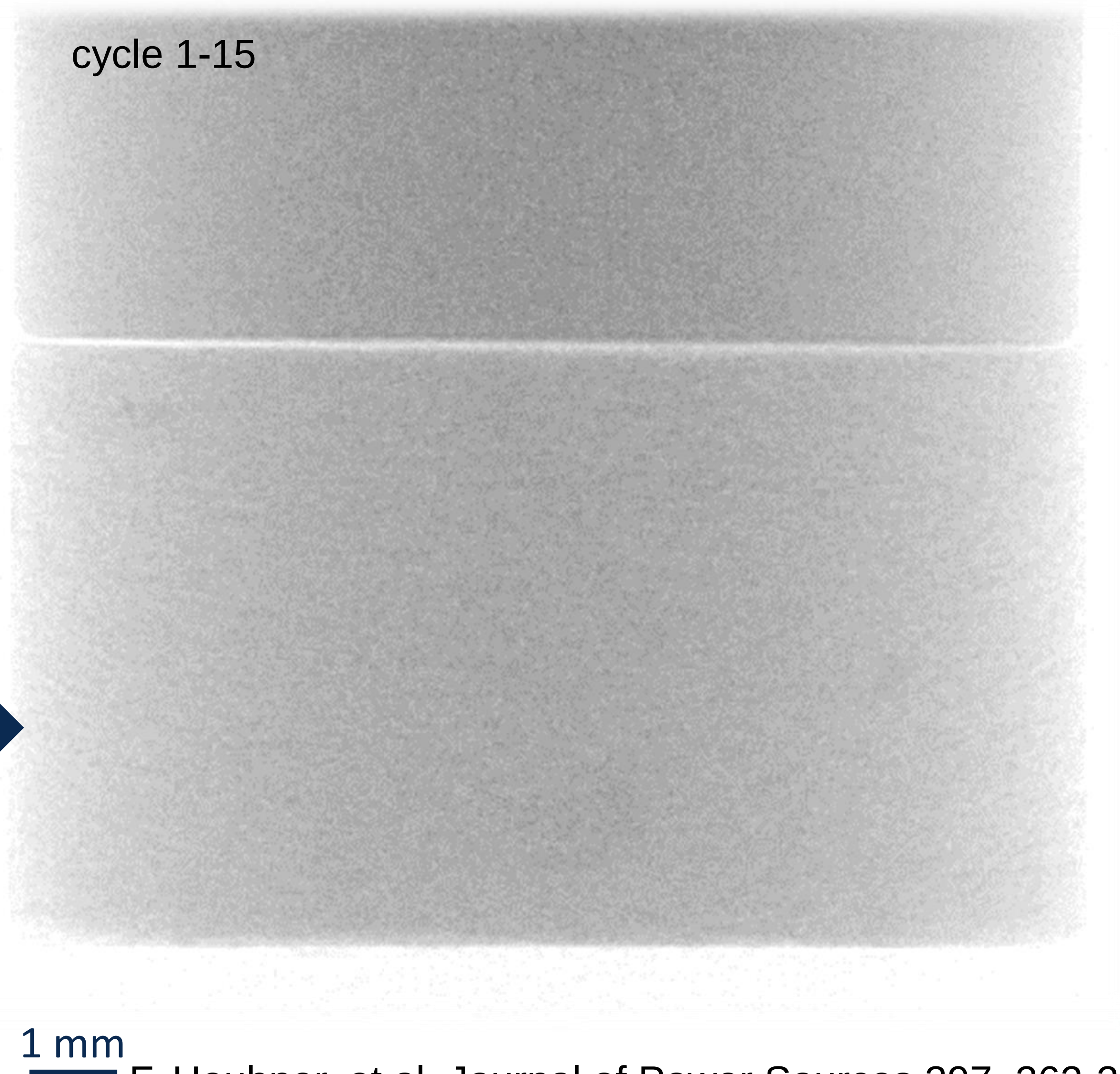
Fraunhofer IFAM
Lars Röntzsch
Felix Hubner

3.2 Powder MHC

1. Activation
   1x heating up to 150˚C

2. Cycling
   30/1.3 bar, RT

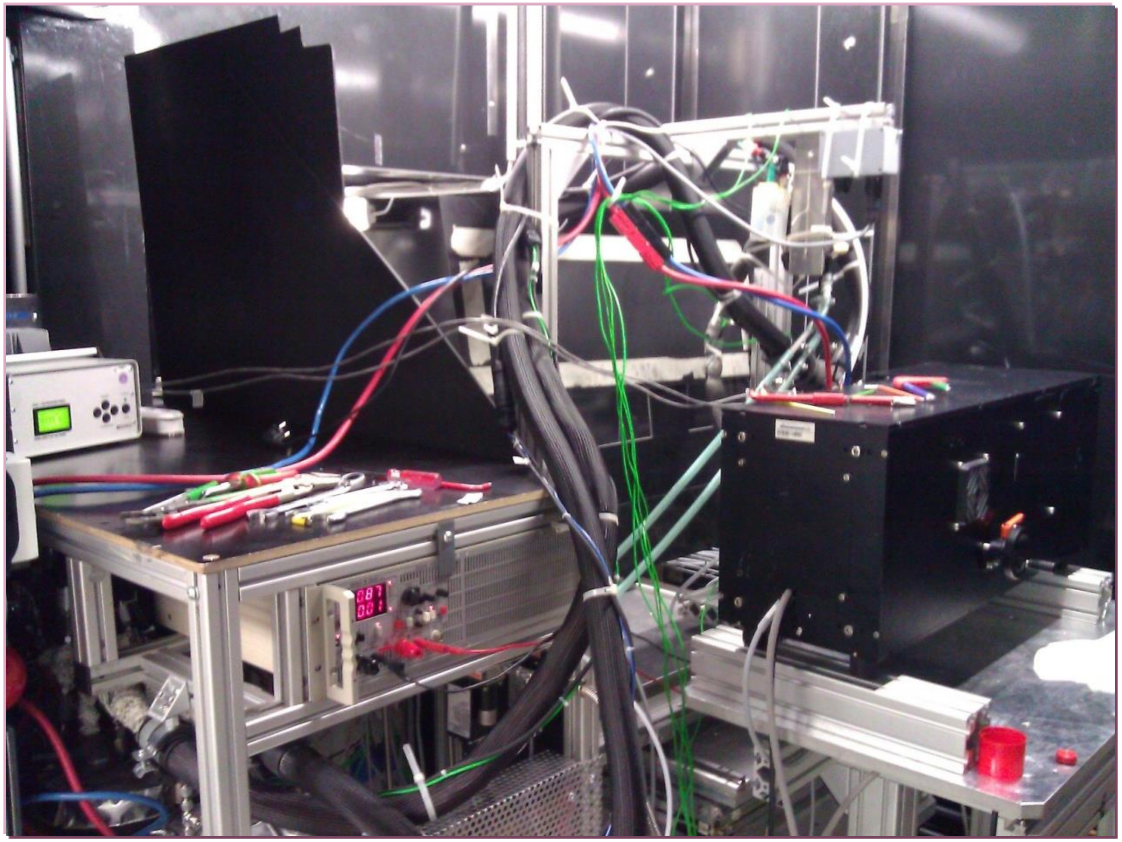




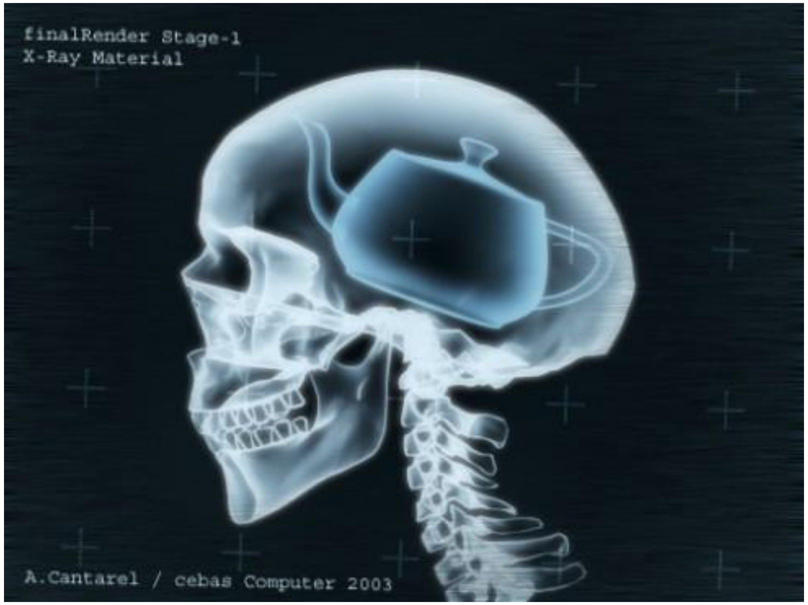
F. Heubner, et al. Journal of Power Sources 397, 262-270

User operation

User operation



Thank you !


finalRender Stage-1
X-Ray Material
A.Cantarel / cebas Computer 2003