Apparatus for In-situ Defect Analysis (AIDA)

DPG Dresden 2014

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Content

- Motivation
- AIDA system
- How to detect defects? – sheet resistance and PAS
- First results on $Fe_{60}Al_{40}$ with AIDA
- Conclusions

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Motivation

- Development of a UHV system for
    - Defect manipulation by
        - Ions
        - Material deposition
        - Temperature
- Analysis of defects by
    - PAS
    - Resistivity
- Final aim → USER FACILITY

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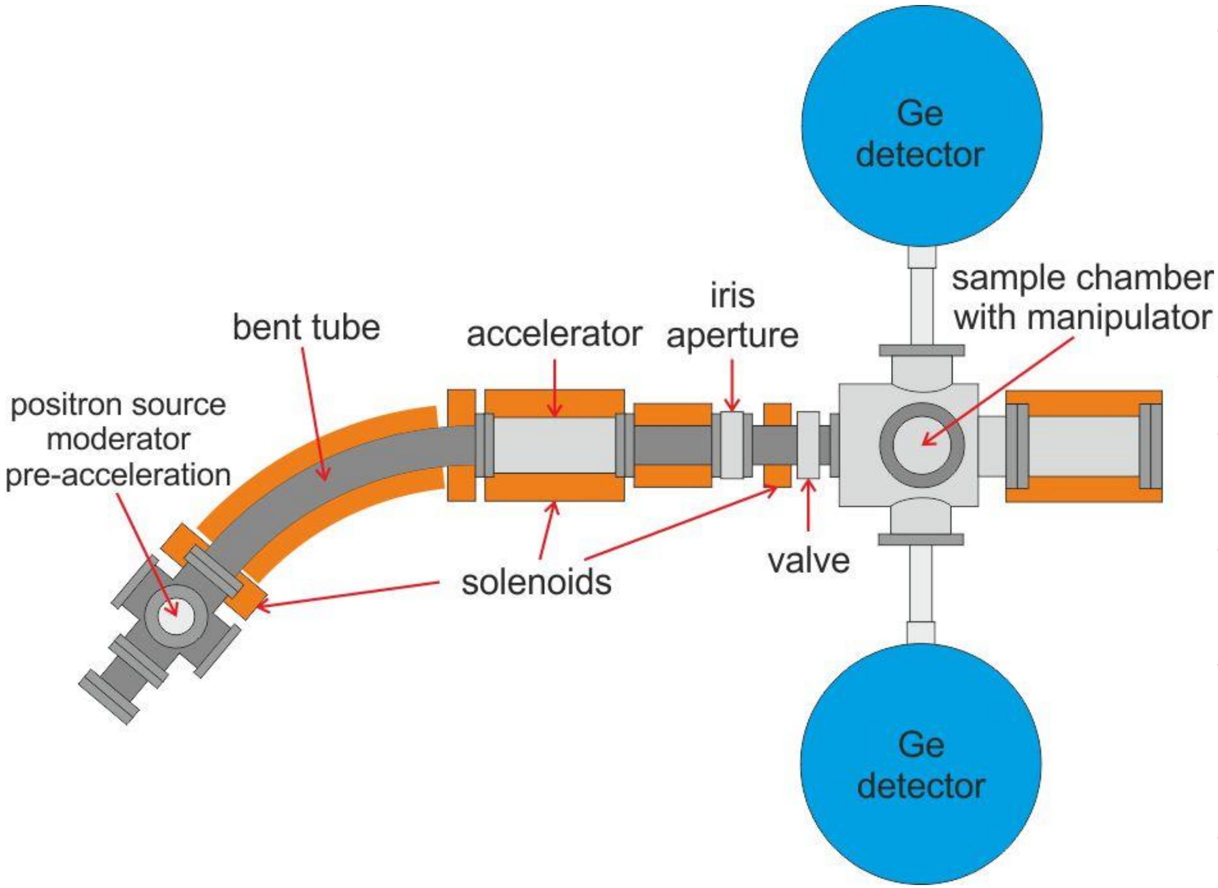
AIDA as the end-station of the SPONSOR (Slow-Positron System of Rossendorf)



Characteristics:

- Magnetically guided positron beam from an intensive ^{22}Na source
  - Moderated with a W moderator
  - Guided by solenoids
- Beam diameter: d ~4 mm
- Accelerator voltage for positrons: 30 eV to 36 keV
- Energy resolution: (1.09 + 0.01) keV at 511 keV
- Two Germanium detectors for coincident Doppler Broadening Spectroscopy
- Samples mounted on a linear manipulator
- Base pressure: ~10^{-7} mbar

Courtesy M. Butterling

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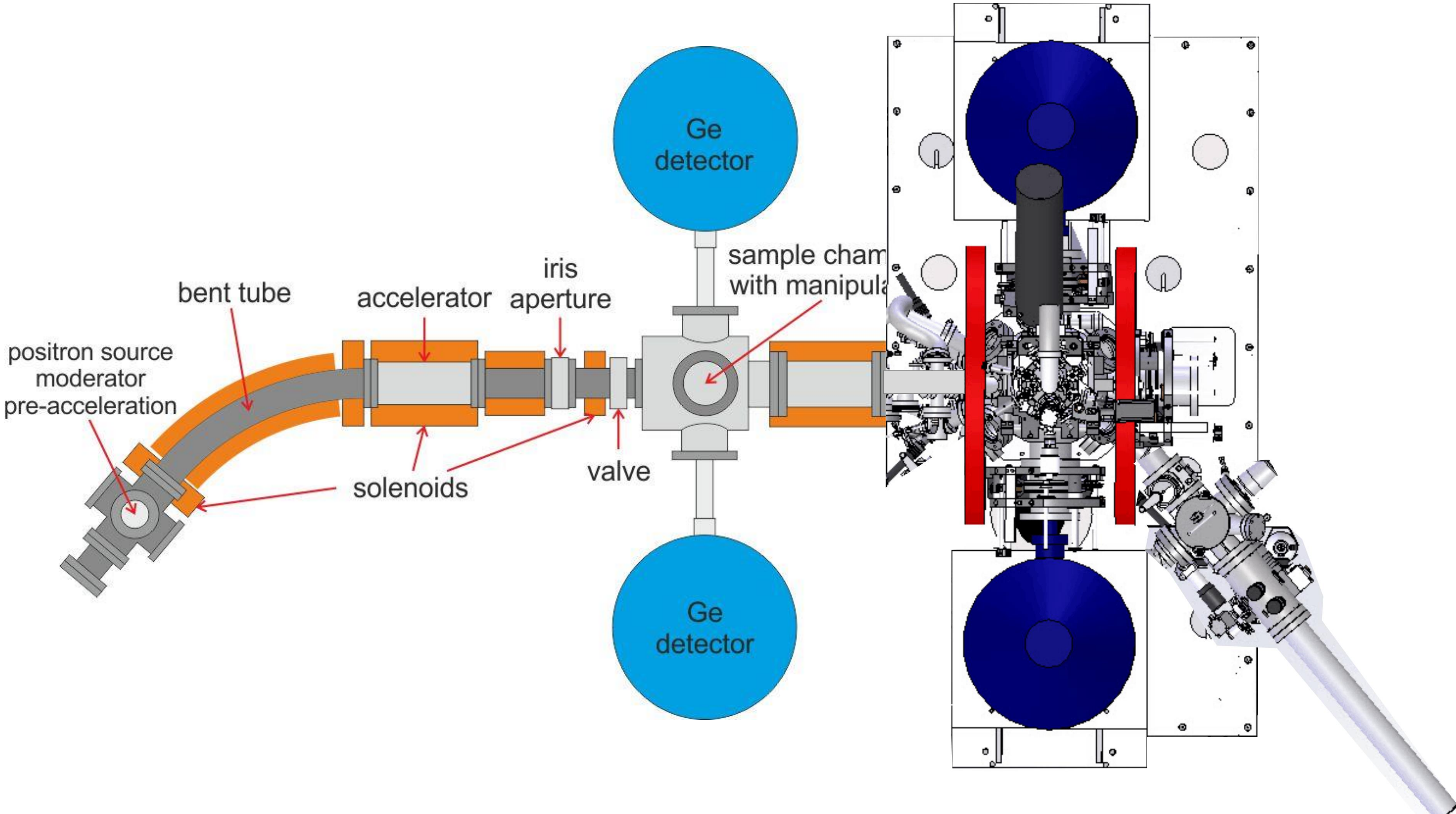
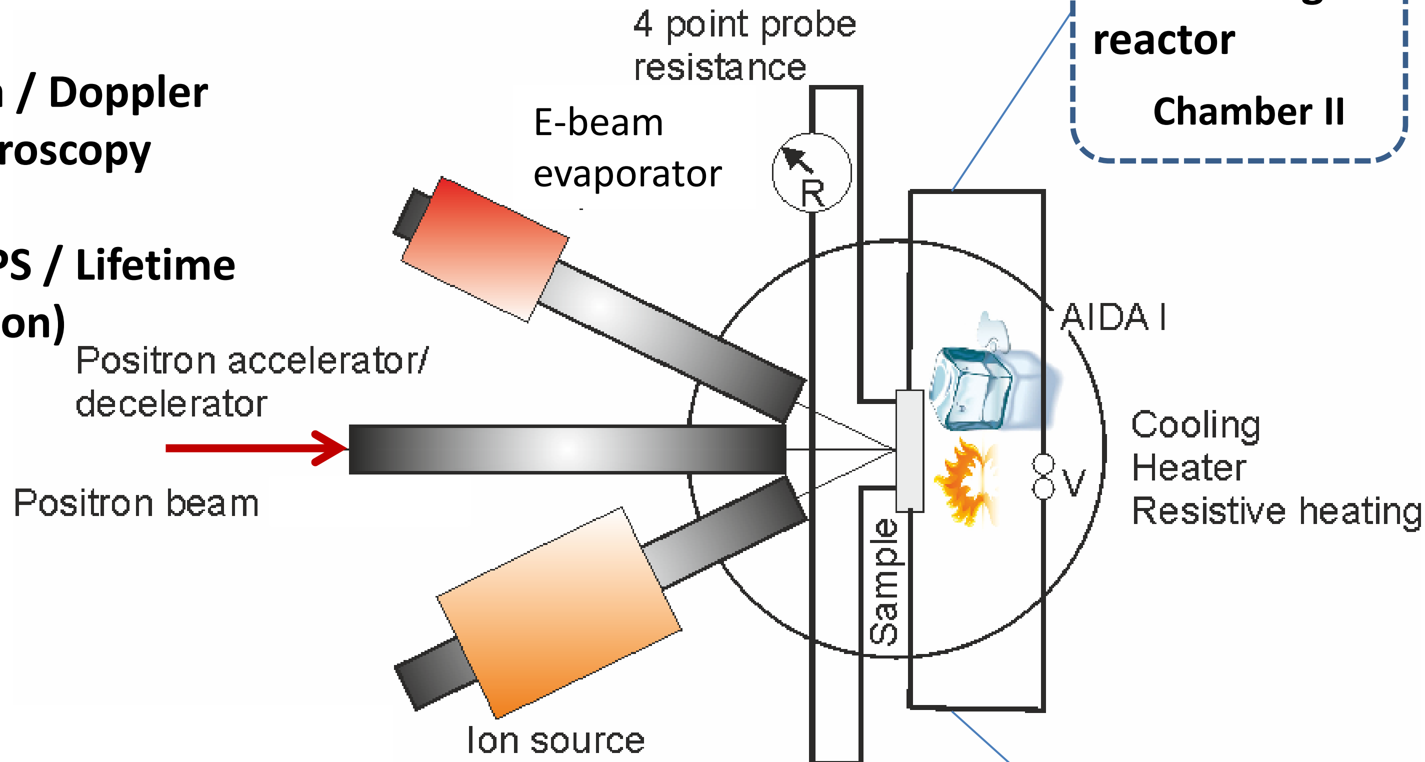


Courtesy M. Butterling

AIDA1 and AIDA2

Working principle

Chamber I @ ^{22}Na / Doppler Broadening Spectroscopy (in operation)
Chamber II @MEPS / Lifetime (during construction)



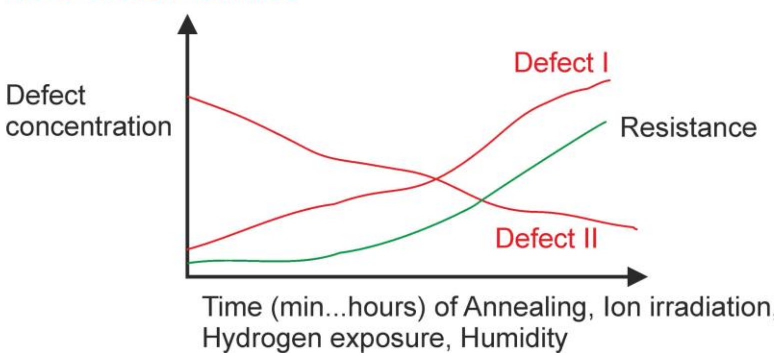
Gas loading reactor

Chamber II

XPS analysis unit

Chamber II

RESULTS EXPECTED



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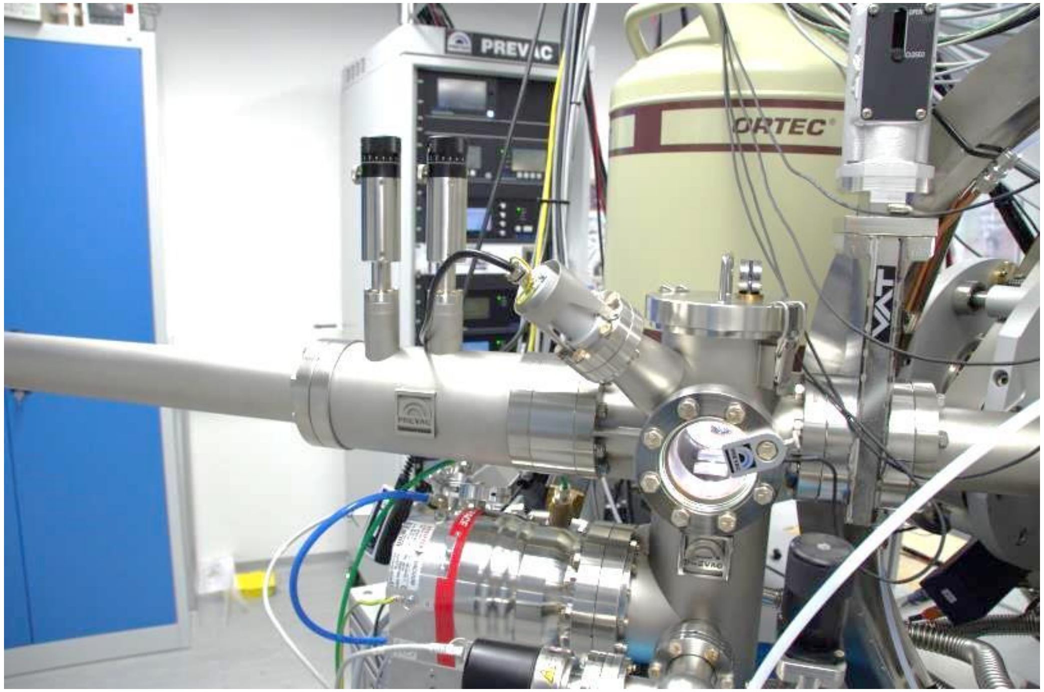
AIDA - components

Load-lock

- Base pressure: 10^{-8} mbar
- Fast sample transfer (20-30 min.)
- Halogen lamp pre-heating (up to 300°C)
- Standard sample size: up to 12 x 12 x 10 mm



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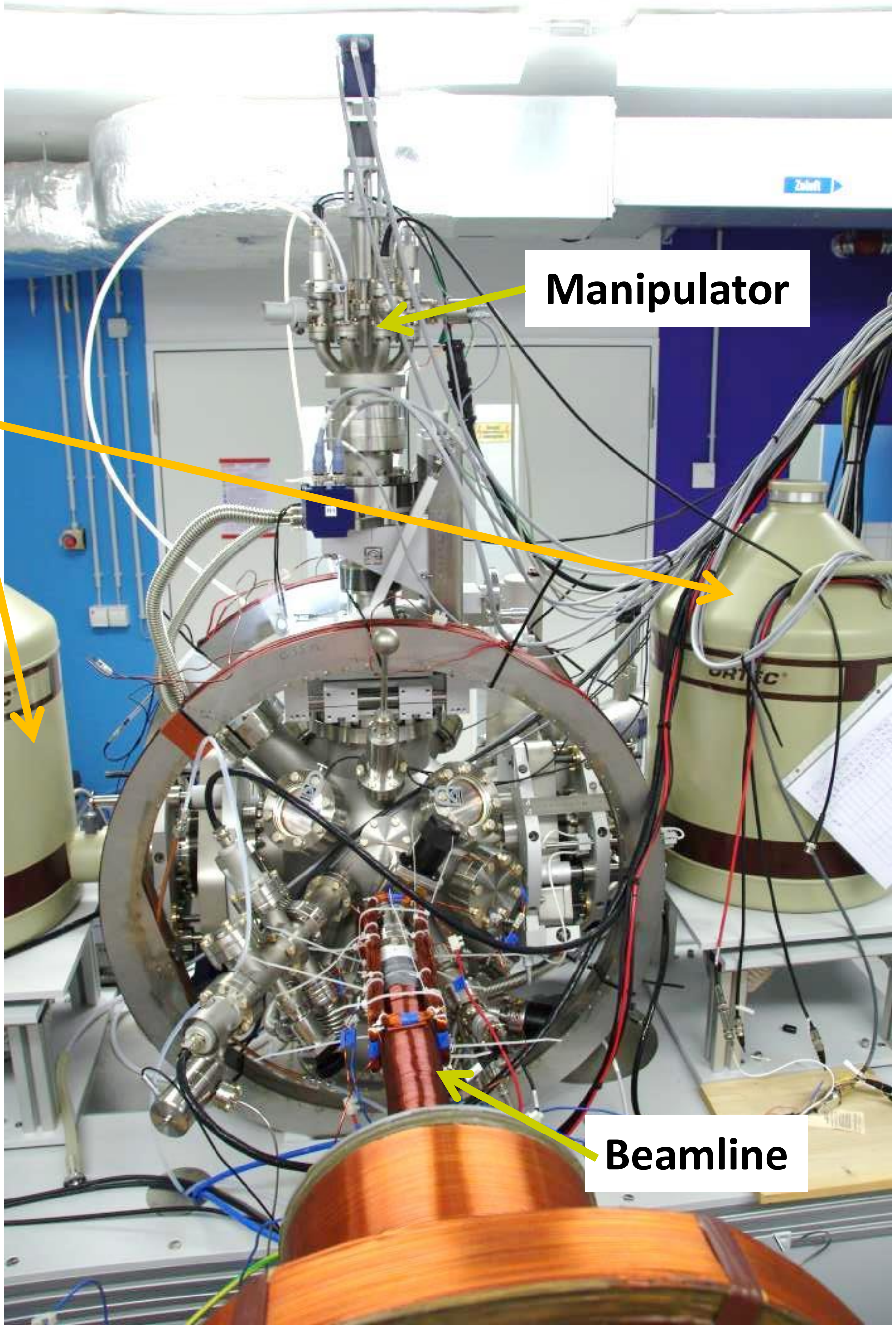
AIDA - components

Main chamber

- Base pressure: 10^{-9} mbar
- Detectors: 2 Germanium detectors for coincidence - Doppler broadening spectroscopy on motorized tables
  - Distance to the sample: ~ 30–120 mm



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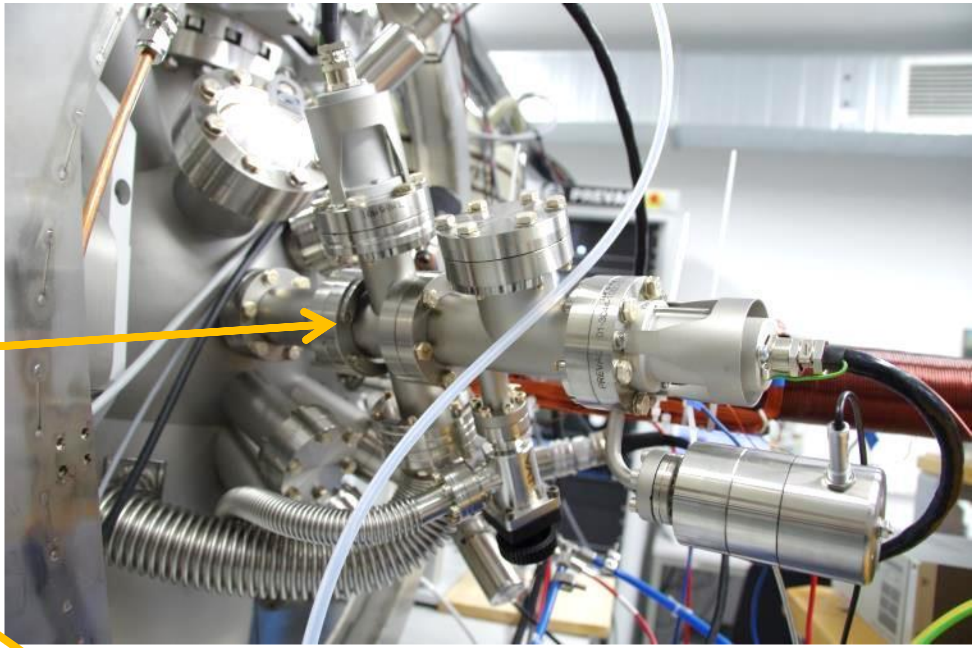
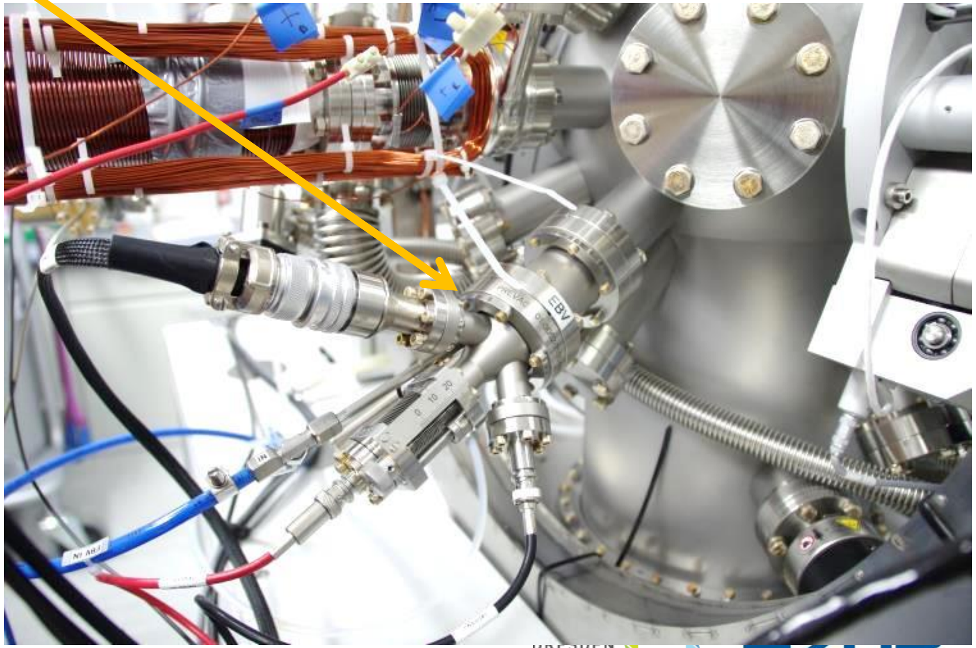
AIDA - components

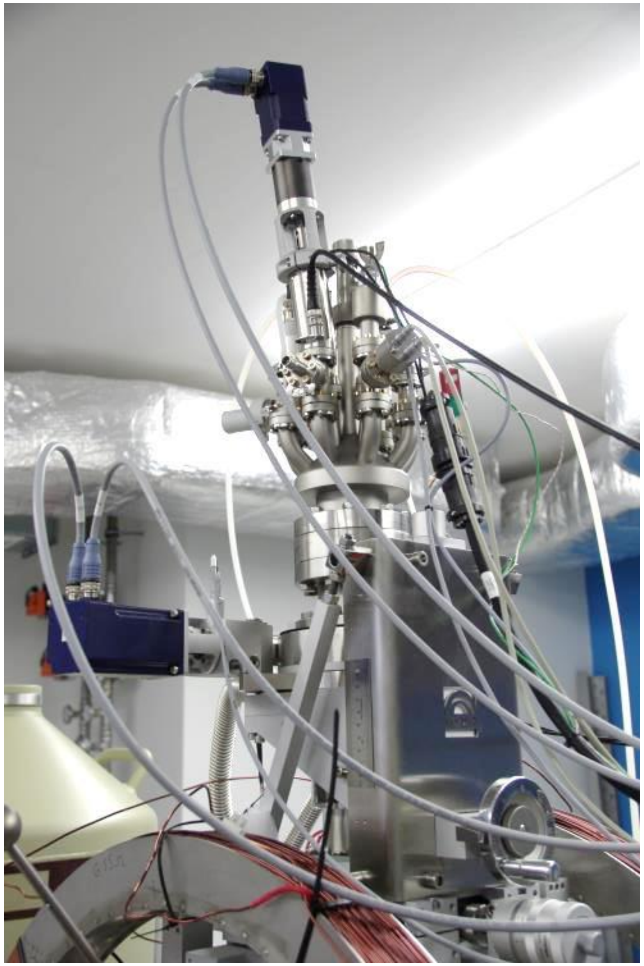
Main chamber

- Ions: Prevac scanning ion source
  - Noble gases, N, O, H
  - Ion current density: <200 μAcm^{-2}
  - Energy: 1 eV...5 keV
  - Possibility of varying ion incident angle
- Deposition: Prevac rod-evaporator
  - Metals: Fe, Co, Ni, Au, Ag, Cr, Pt, Mo
  - Rate: 0.1-0.5 Å/s, Quartz microbalance controlled
  - Flanges for 3 additional evaporation sources
- Shutter for beams blocking



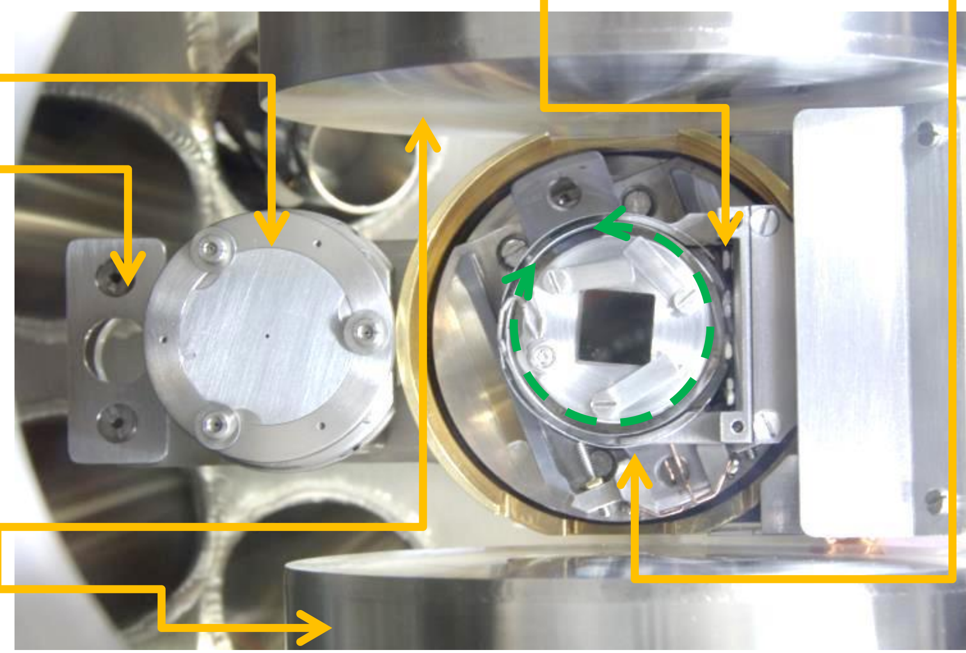


AIDA - components



Manipulator/Sample holder

- Manual X (±12.5mm), Y (±12.5mm), Z (100 mm) movement
- Motorized continues in-plane rotation
- Motorized out-of-plane rotation (±175°)
- Temperature range at sample holder: 50...1200 K
- Aperture for the positron beam positioning
- Faraday cup
- Resistometer: 4-point-probe



Ge detectors pockets

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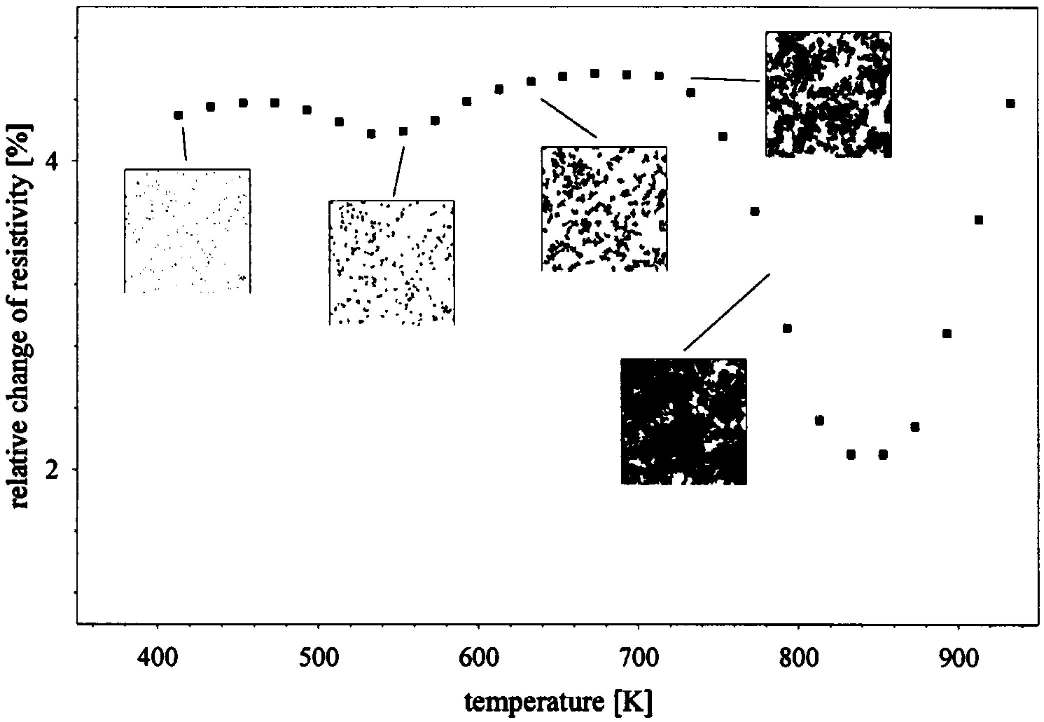
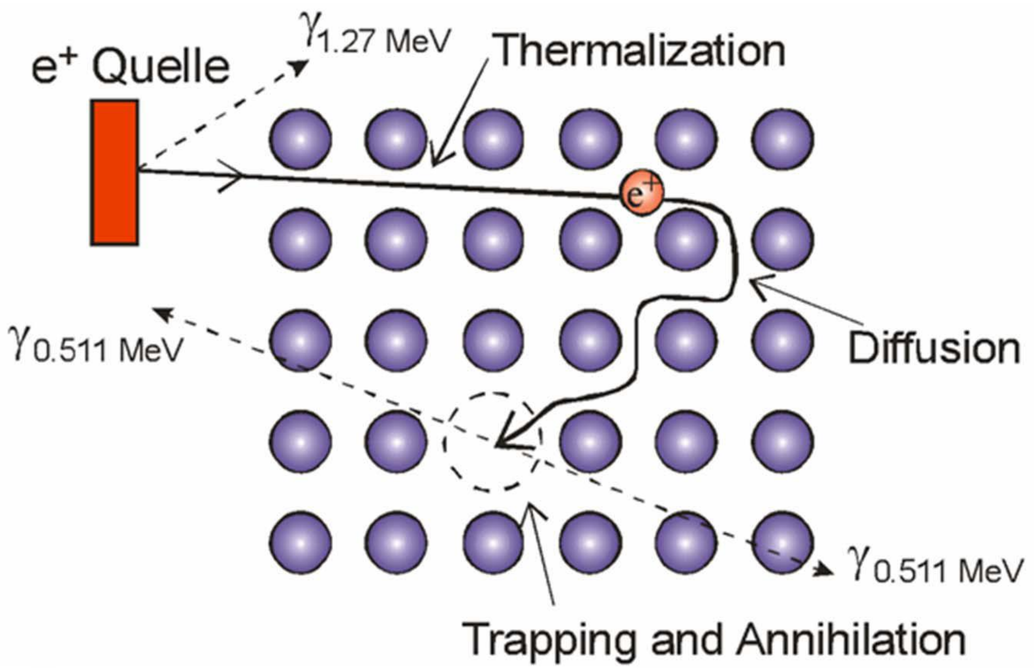
How to detect defects? – Resistivity



- Residual resistivity measurements during isochronal annealing (t=20 min, T=20 K) of Fe–44.8 at.% Al after quenching from 773 K
- Schematical representation of vacancies distribution as a function of temperature

W. Pfeiler, B. Sprušil, Materials Science and Engineering A324 (2002) 34–42

How to detect defects? – Positron Annihilation



- **Positron wave-function is localized in the attractive defect potential**
- **Annihilation parameters change in the localized state**
  - **Positron lifetime increases in a vacancy**
  - **Doppler broadening decreases in the open volume defect**
  - **Coincidence spectrum depends on local chemistry**

AIDA = combination of resistivity and PAS

Courtesy R. Krause-Rehberg

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AIDA – first results on FeAl

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Order-disorder transition in bulk $Fe_{60}Al_{40}$

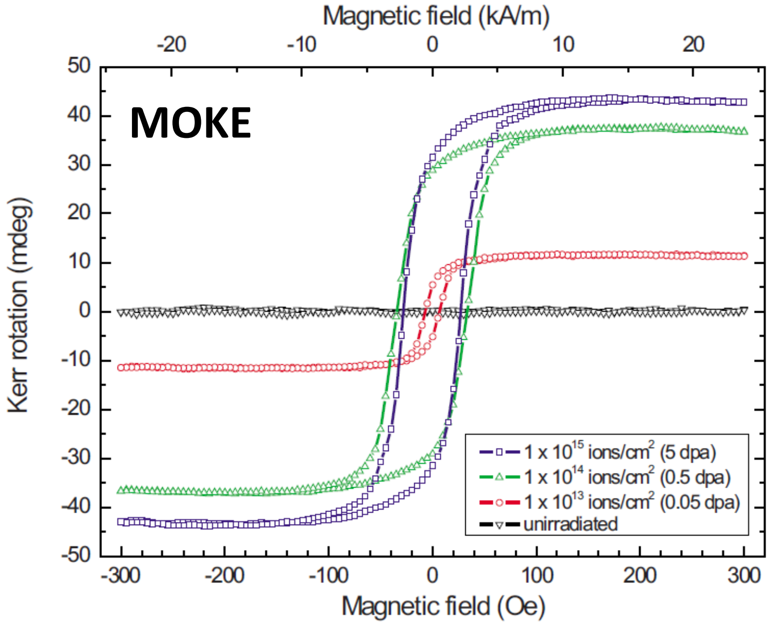
J. Fassbender, M. O. Liedke, T. Strache, W. Moeller, E. Menendez, J. Sort, K. V. Rao, S. C. Deevi, J. Nogues, *Phys. Rev. B* **77**, 174430 (2008)

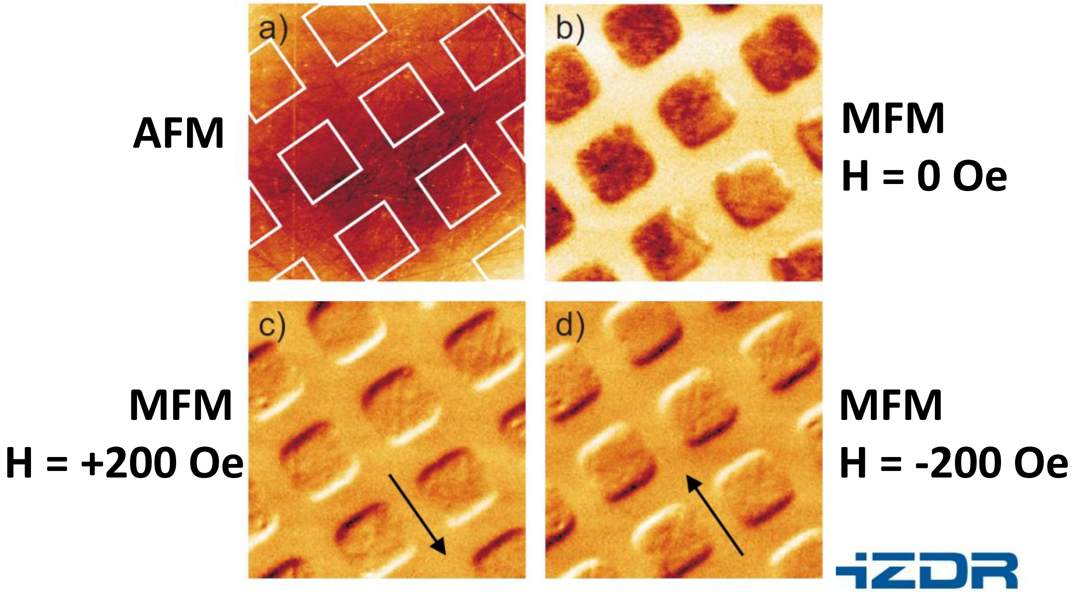
Chemically **ORDERED** $Fe_{60}Al_{40}$ (B2 phase) → **paramagnetic**

Chemically **DISORDERED** $Fe_{60}Al_{40}$ (A1 phase) → **ferromagnetic**

Ion irradiation induced ferromagnetism = disorder = more Fe nearest neighbors = ferromagnetism

Do open volume defects play a role?





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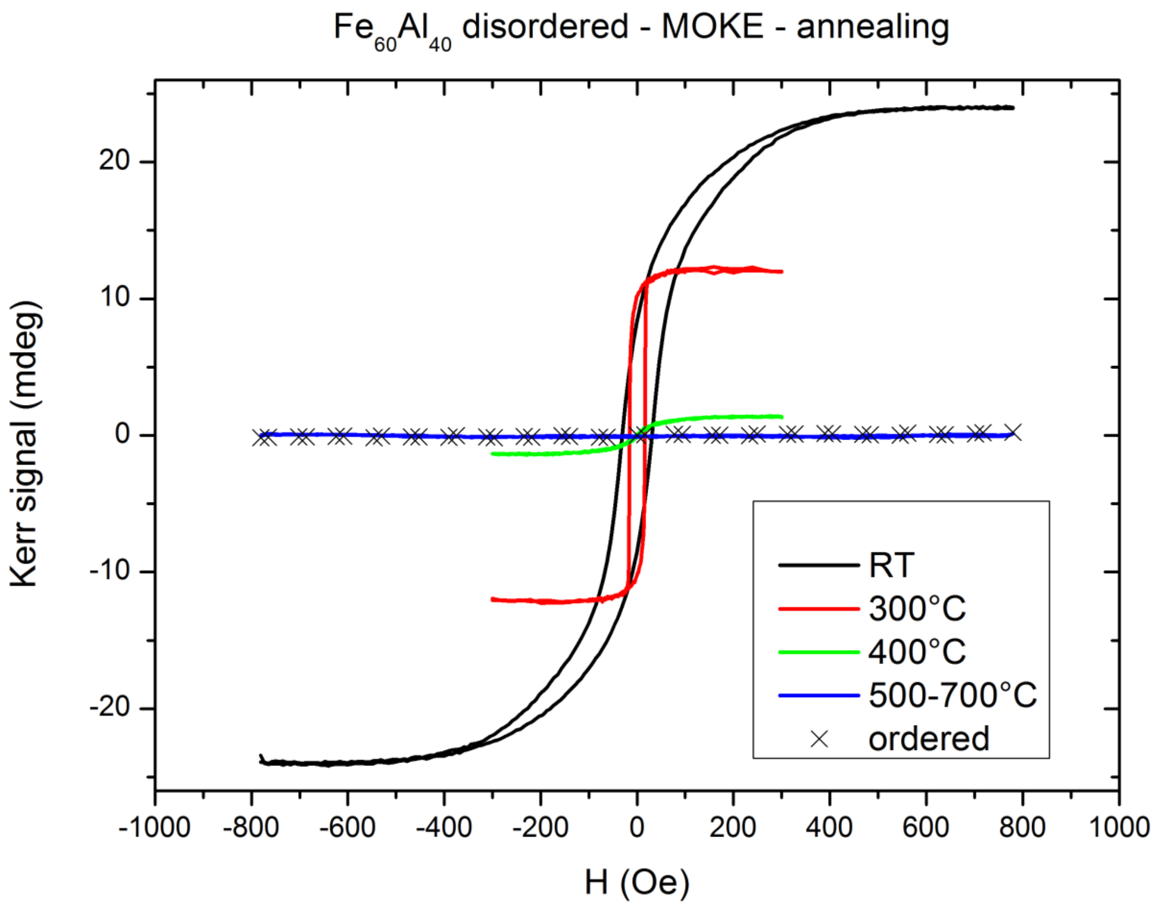
MOKE results – annealing of the disordered $Fe_{60}Al_{40}$



- 2 samples:
  - $SiO_2/Fe_{60}Al_{40}$ (250nm) sputter deposited at RT = disordered = ferromagnetic
  - $SiO_2/Fe_{60}Al_{40}$ (250nm) Ar furnace annealed at 500°C = ordered = paramagnetic
- Decrease and final disappearance of the magnetic signal as a function of the annealing temperature
- No magnetic signal in the 500-700°C range

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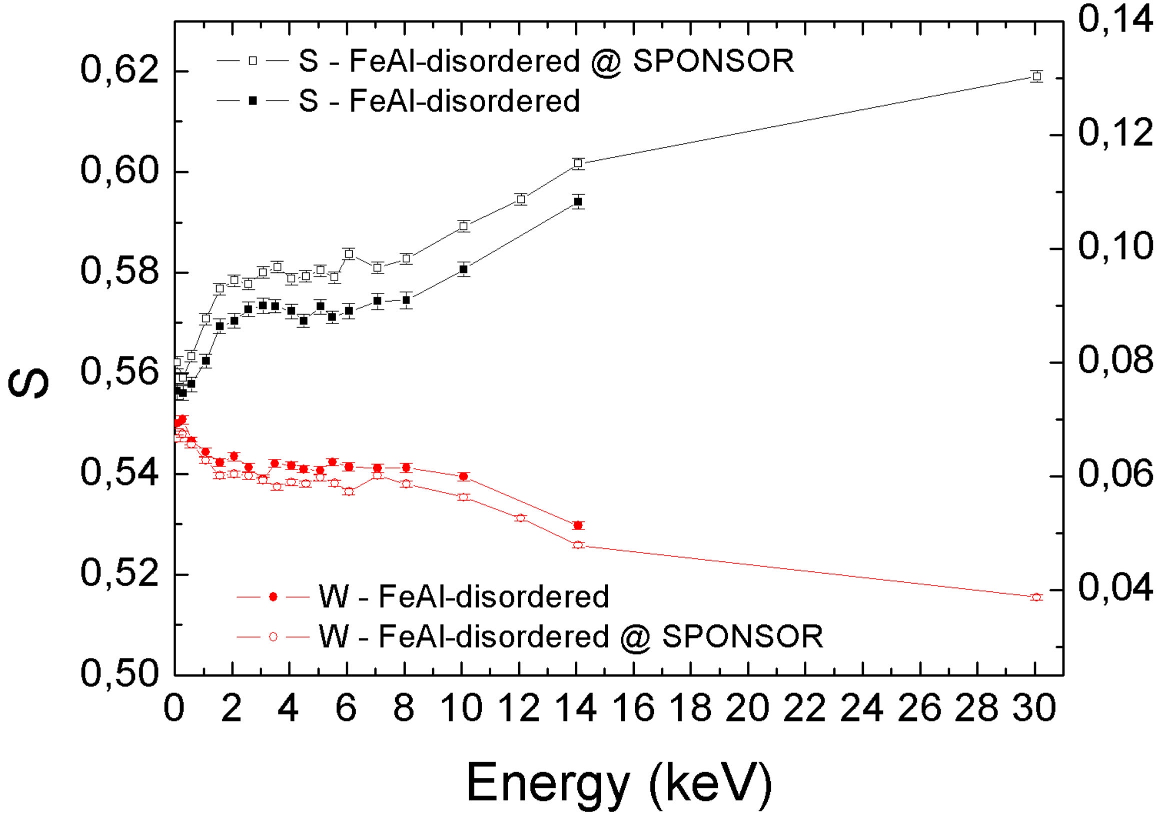
S and W as a function of positron Energy – SPONSOR vs. AIDA @ RT –



- SPONSOR and AIDA data show the same trend
- Offset in S and W between the data from both systems due to different detector efficiency
- The same measurement time (about 1h per energy step) but lower count rate at AIDA (~80 vs. ~45 cps)
- Si substrate signal starts from about 8-10 keV

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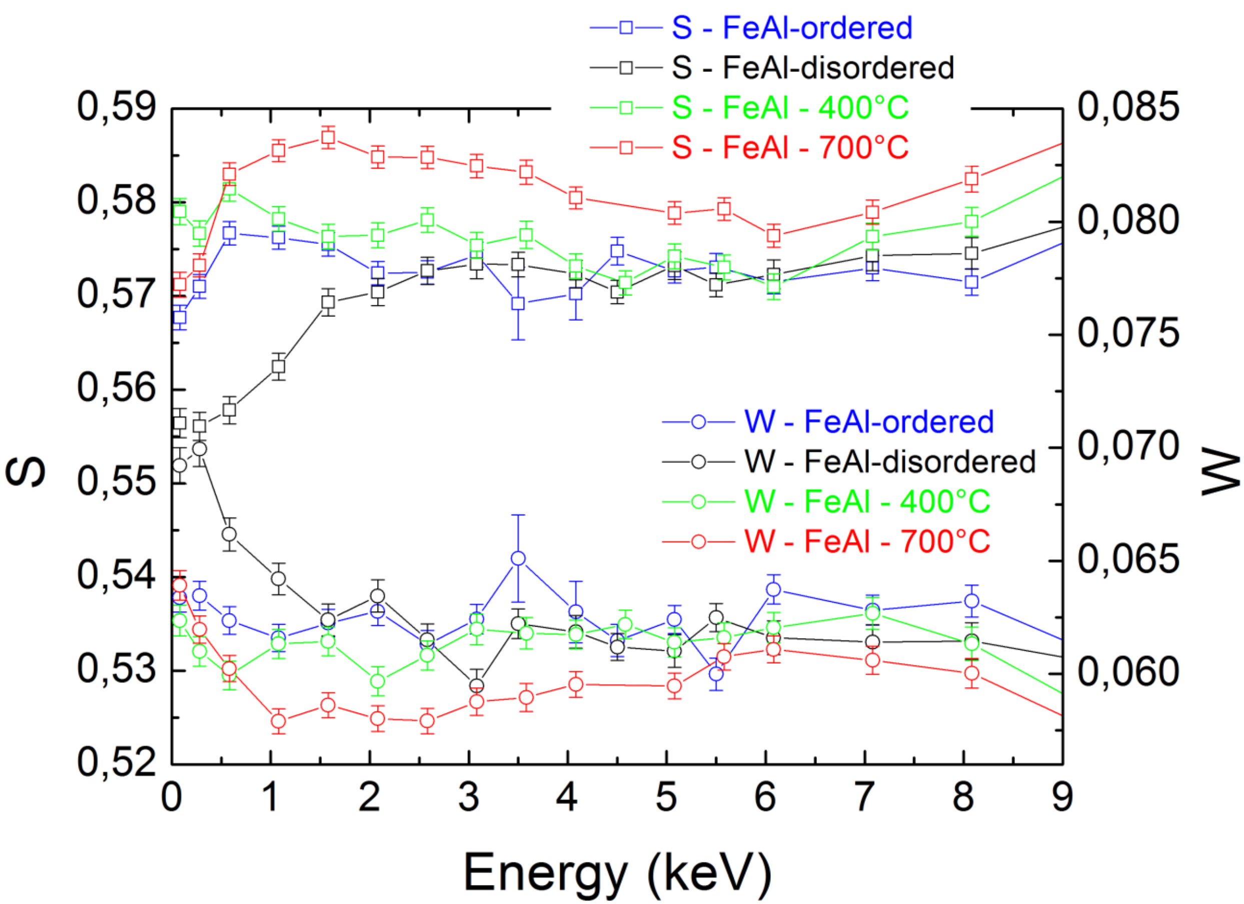
S and W as a function of positron Energy
– FeAl ordered vs. disordered vs. annealing –



- Slight scattering for some energies → too low statistic
- Visible differences in S and W close to the film surface only
- S similar for all cases across 250 nm of the film, besides 700°C
- W smaller in the case of the 700°C annealed film → different defect decoration
- Clear plateau from about 3 to 8 keV → additional measurements with both Ge detectors at 5 keV

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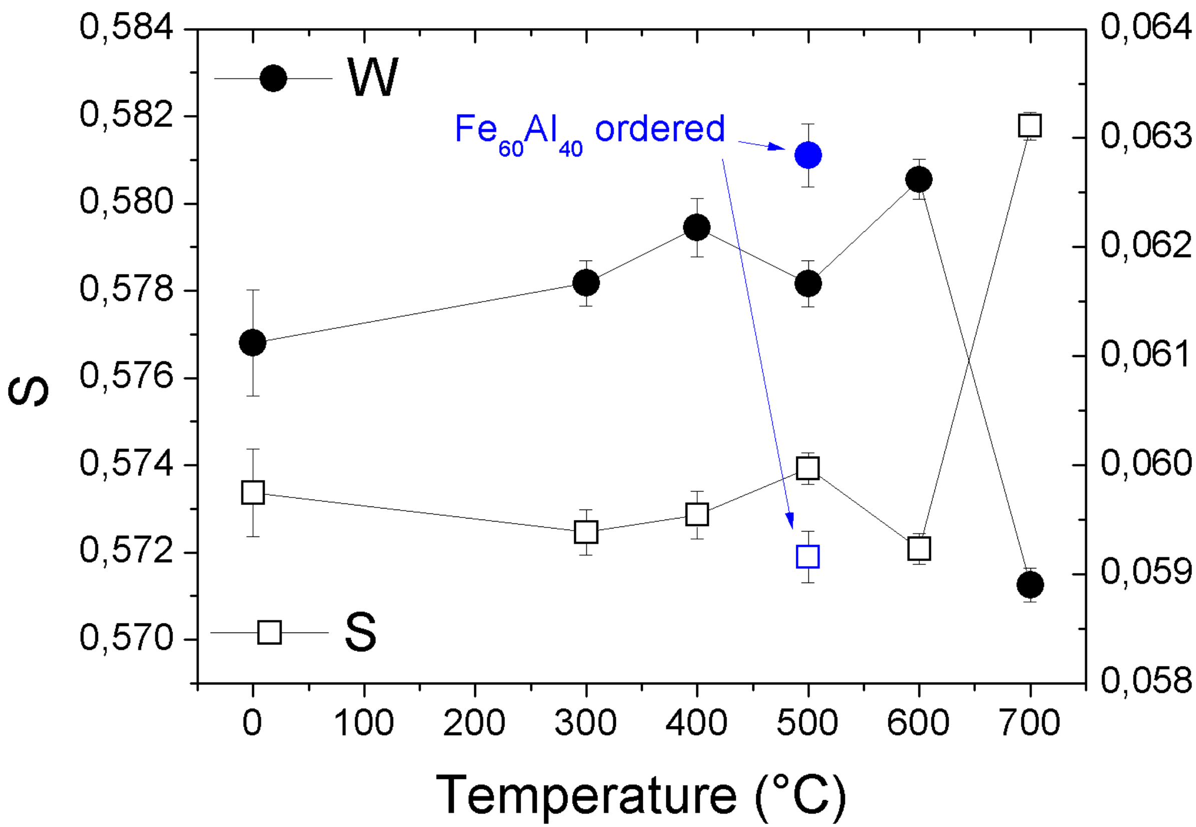
S and W as a function of annealing temperature @ 5 keV



- Roughly constant values of S and W parameters up to 500°C
  - Slight decreasing trend for S and increasing for W
  - Clearly visible change for 700°C → clustering?
- Ordered sample shows smaller open volume defect
- Ongoing coincidence measurements in order to address defects decoration

NO clear connection between open volume defect and magnetic properties

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Realized

- PAS on Si and FeAl
- Annealing and PAS on FeAl

TO DO

- Increase of the count rate
  - New ^{22}Na source
  - Improve of positron moderation
- Ion irradiation and PAS
- Sheet resistance measurements and PAS
- AIDA as USER FACILITY!

Ongoing/possible studies

- Defects in metal alloys with chemical order, e.g., FePt, FeRh
- Memristive effects in TiOx
- Magnetic and defect-properties of Co implanted TiO_2 (diluted magnetic oxides)
- Defects/ordering in Metal-Spintronics, e.g., Fe/Nb multilayers by hydrogen loading

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- Alireza Heidarian

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Supporting Materials

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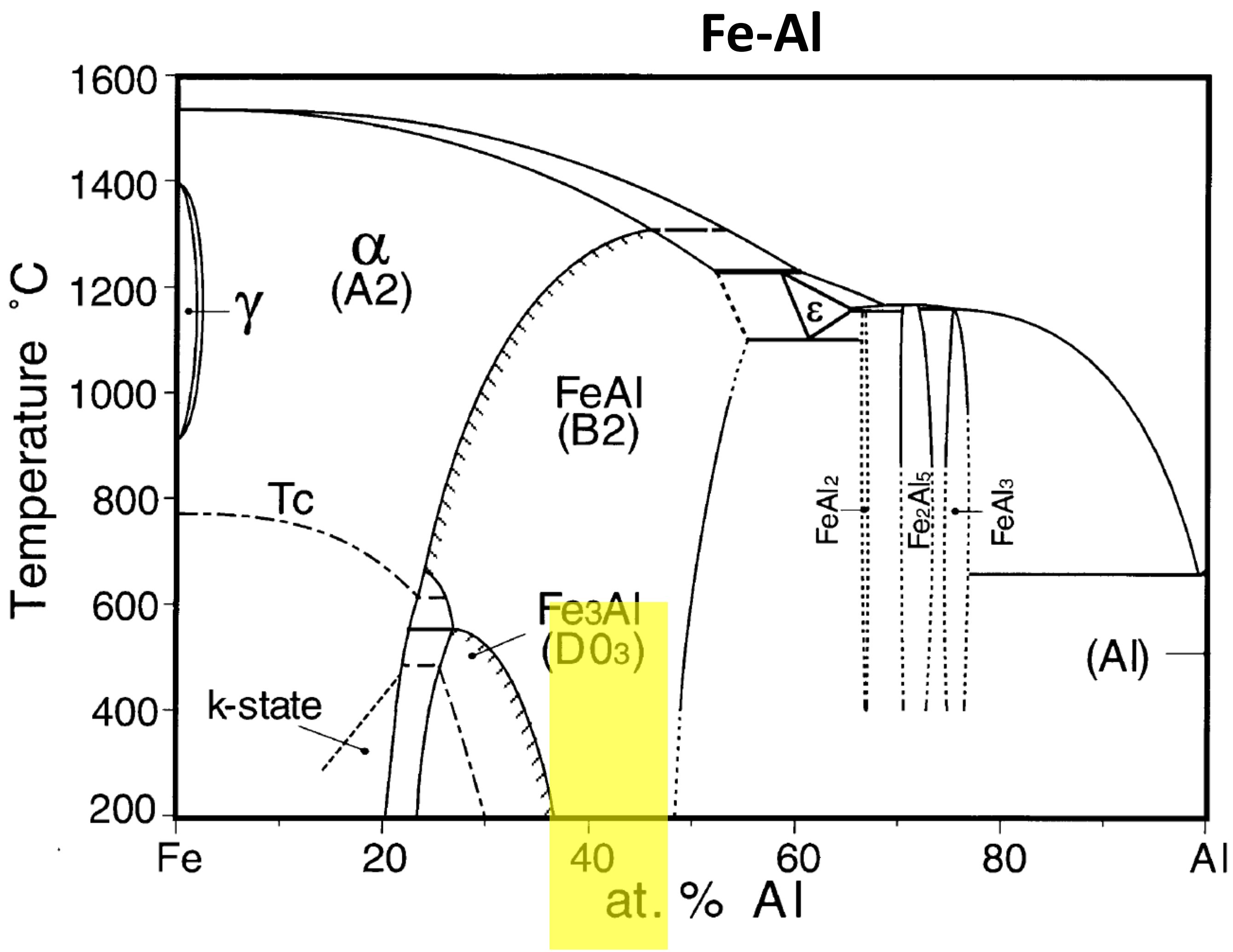
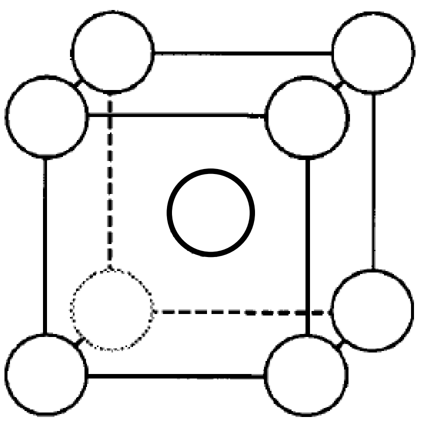
Fe-Al Phase Diagram – paramagnetic B2 phase





B2 Phase

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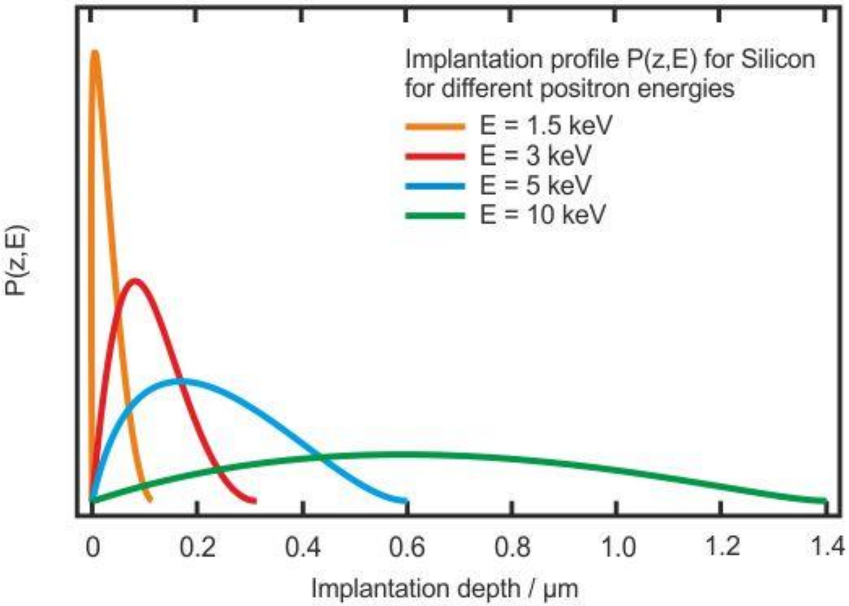
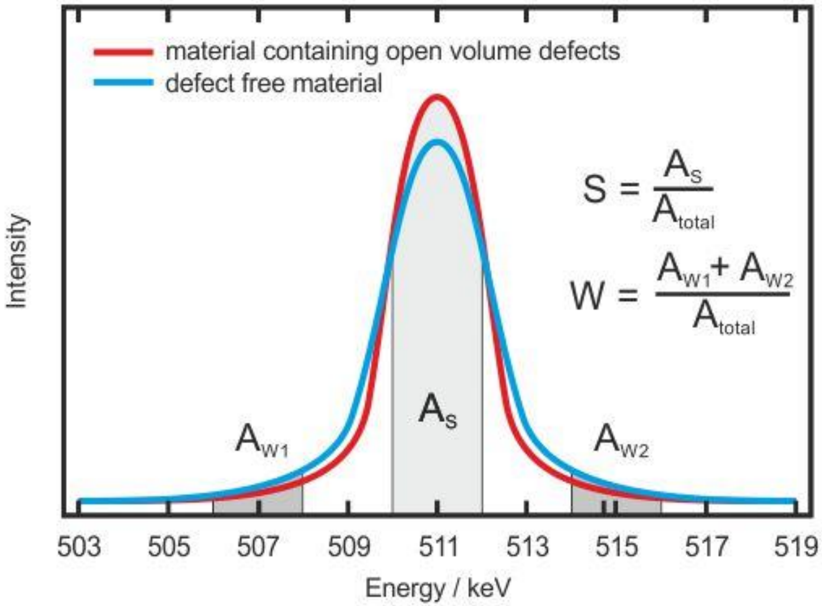


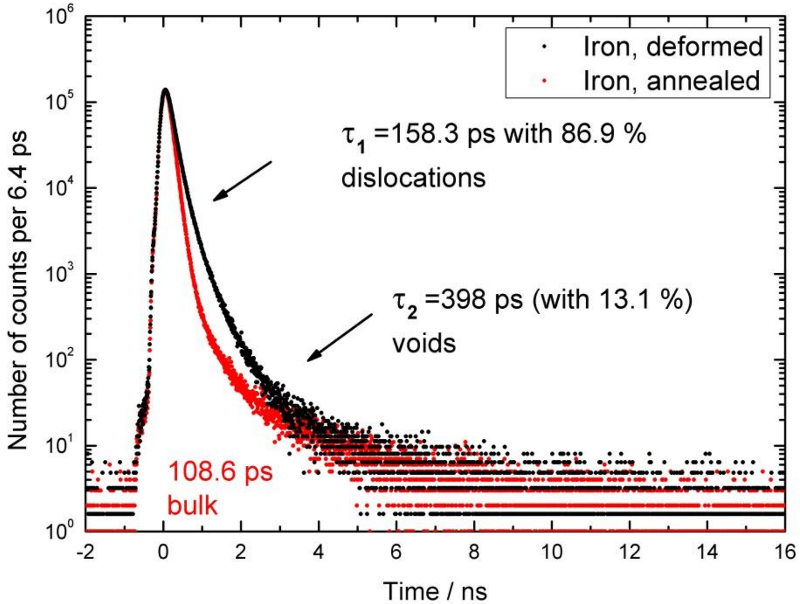


Courtesy M. Butterling

Materials analysis using positrons

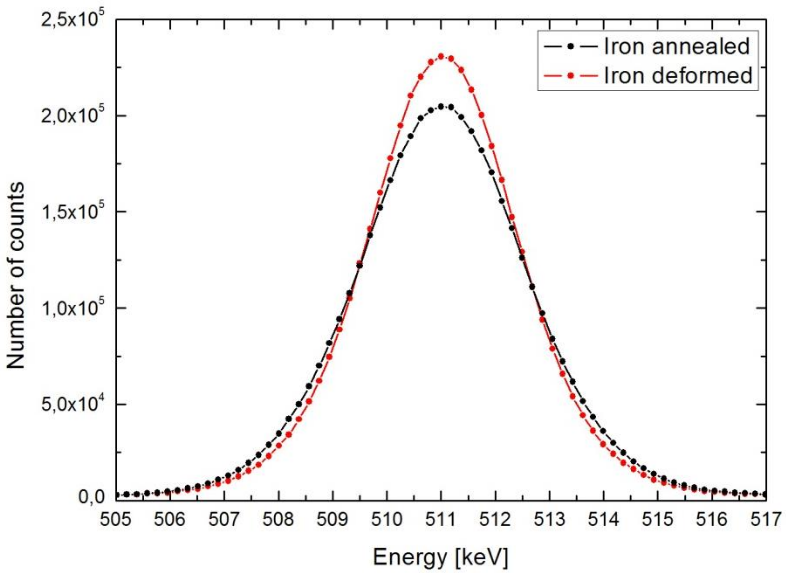
ELBE.

Positron lifetime
Time between positron generation and 511 keV gg-detection



lower electron density
→ **lower annihilation probability**
→ **longer positron lifetime**

Doppler broadening
Energy shift of the 511 keV annihilation line



different materials
→ **different kinetic energy of core electrons**
→ **different broadening of the 511 keV line**

Courtesy M. Butterling

DRESDEN concept HZDR

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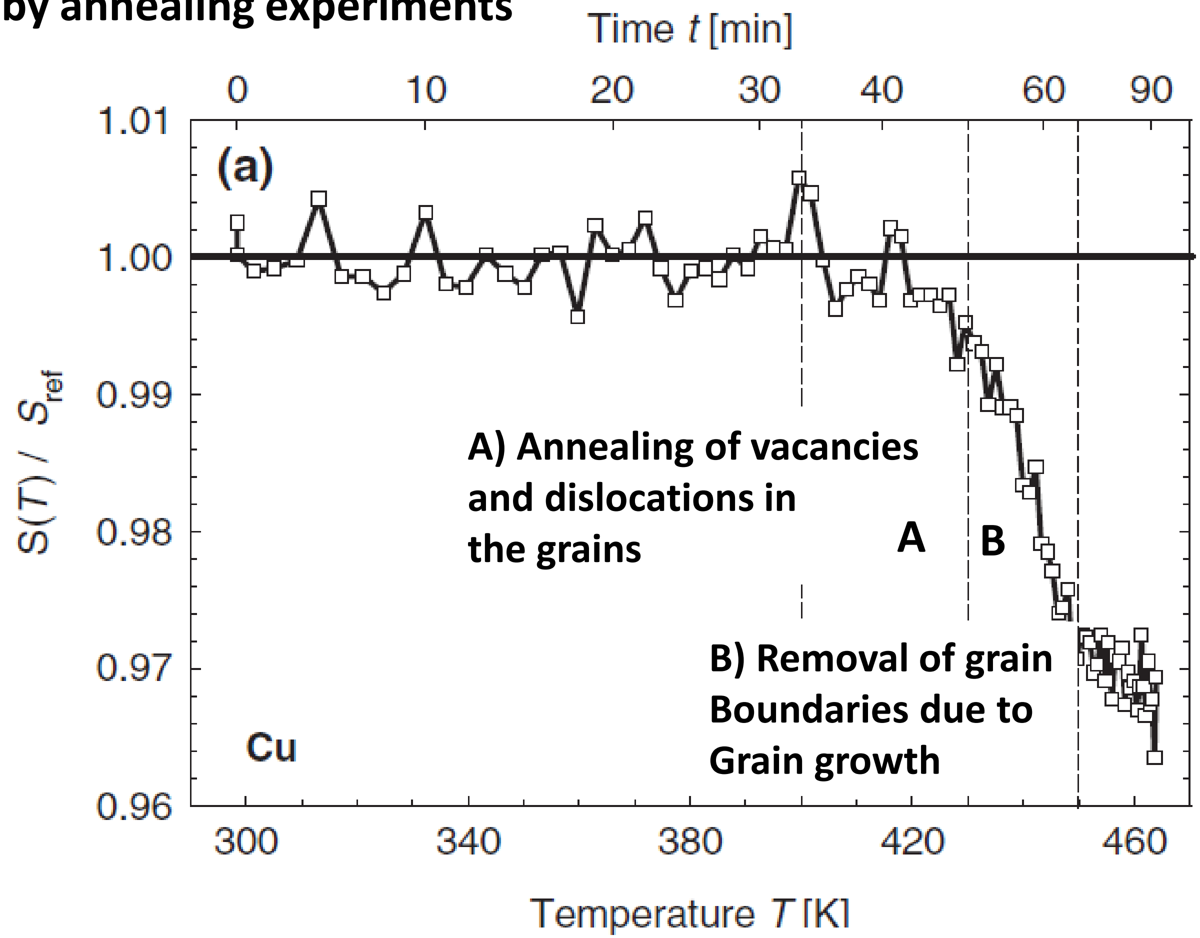
Materials analysis using positrons

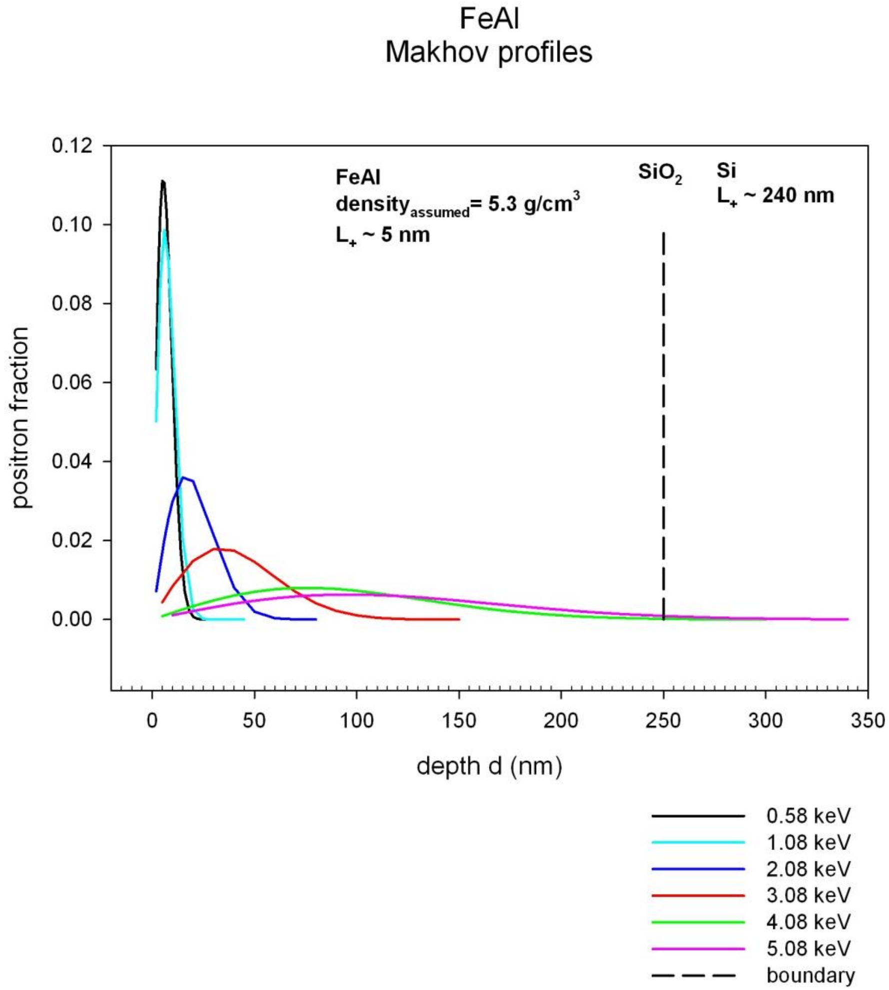
In-situ Example from Munich

*Cu disks deformed by high-pressure torsion at 2.2 Gpa
→ 270 nm granular structure
*Followed by annealing experiments



B. Oberdorfer et al., PRL 105, 146101, 2010

FeAl Makhov profiles



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