

Experimental facilities: MePS at ELBE

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- Historical remarks
- Defect detection by positrons
- Overview: The EPOS-System at FZD
- Mono-energetic Positron Beam (MePS)



Discovery of the Positron



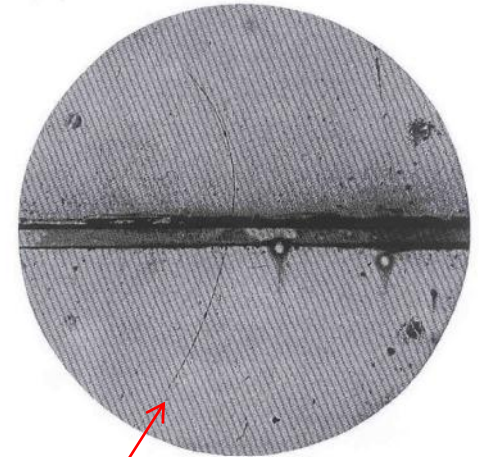
P.A.M. Dirac

- Positron was predicted in 1928 by Paul A.M. Dirac
- Discovery in 1932 in cloud chamber pictures by C.D. Anderson



C.D. Anderson

- Positronium as bound state of e^- and e^+ - lightest atom - was predicted (1934) and discovered (1951)
- Annihilation in matter was studied beginning in the 40th
- Positrons can be obtained by
 - pair production from gamma radiation ($E_\gamma > 1022 \text{ keV}$)
 - β^+ decay from isotopes (mostly ^{22}Na)



- first Identification of a positron in a cloud chamber
- 5 mm lead plate
- photo taken by C.D. Anderson



Positrons are sensitive for Crystal Lattice Defects

- 1950...1960: different experimental techniques were developed
- Positron lifetime spectroscopy and Doppler broadening spectroscopy
- end of 60s: lifetime is sensitive to lattice imperfections
 - Brandt et al. (1968): **vacancies in ionic crystals**
 - Dekhtyar et al. (1969): **plastically deformed semiconductors**
 - MacKenzie et al. (1967): **vacancies in thermal equilibrium in metals**
- Positrons are localized (trapped) by open-volume defects

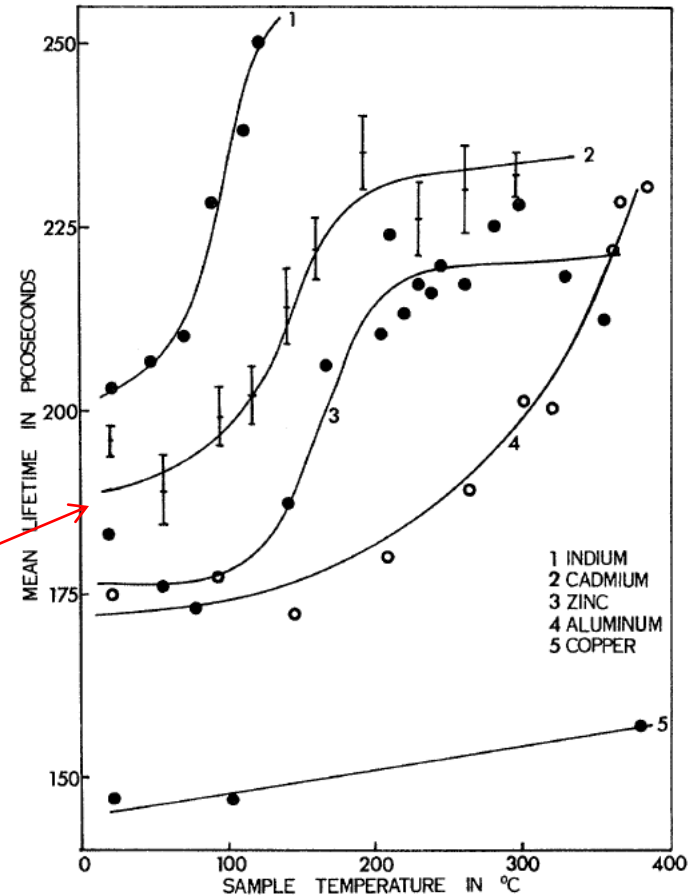


FIG. 1. Positron mean lifetimes in several metals as a function of temperature.

$$C_{1v}(T) = \exp\left(\frac{S_{1v}^F}{k}\right) \exp\left(\frac{H_{1v}^F}{kT}\right)$$

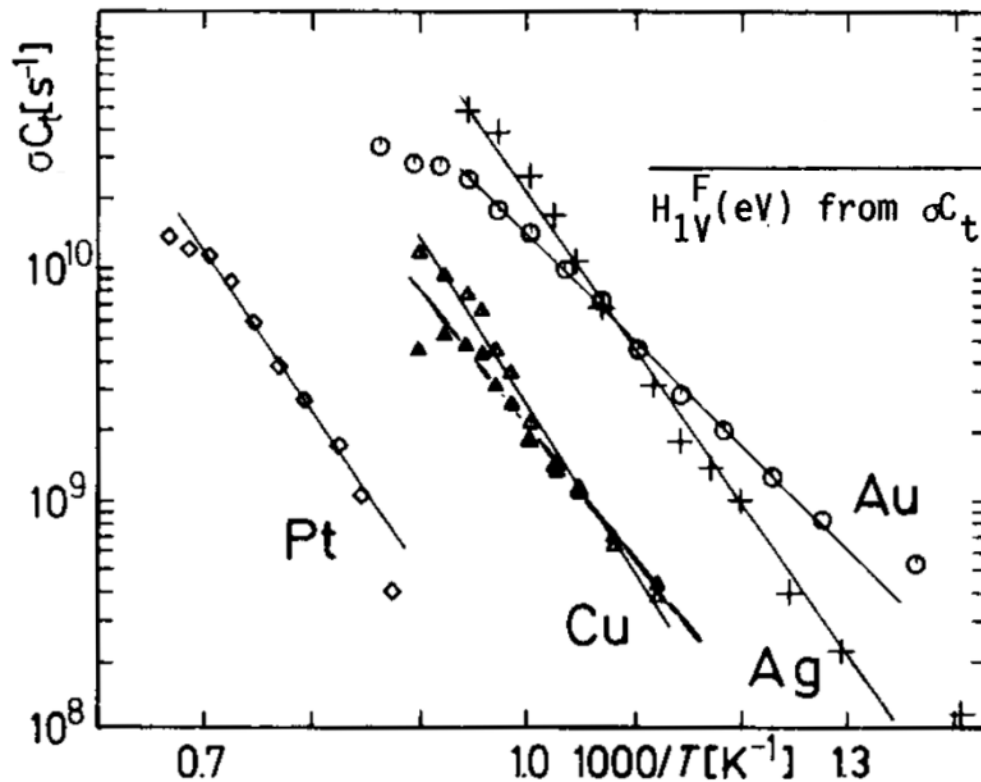
Determination of Vacancy Formation Enthalpy

THERMAL VACANCIES IN THE NOBLE METALS Cu, Ag, Au, AND IN Pt
STUDIED BY POSITRON LIFETIME SPECTROSCOPY

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H_{1V}^F (eV) from σ_t

Cu	Ag	Au	Pt
$1.13 \pm 0.04^*$	1.31 ± 0.07	$.89 \pm 0.02$	1.35 ± 0.09

- Arrhenius-Plot delivers H_{1V}
- was performed for many alloys



Study of non-equilibrium Defects

PHYSICAL REVIEW B

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15 JANUARY 1982

Vacancies and carbon impurities in α -iron: Electron irradiation

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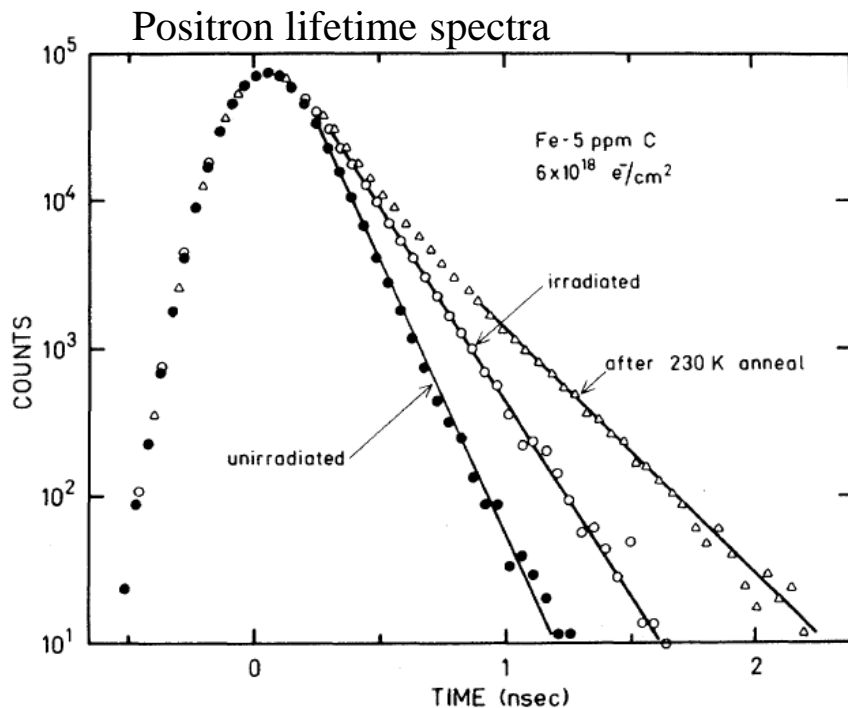


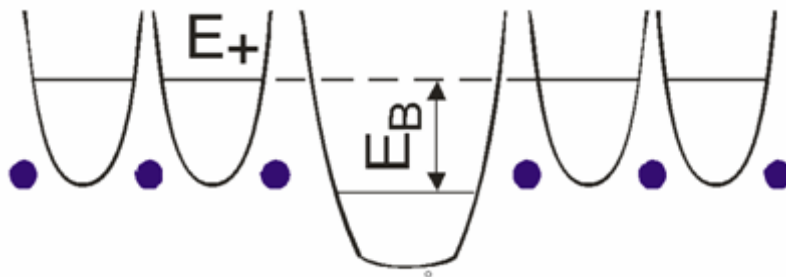
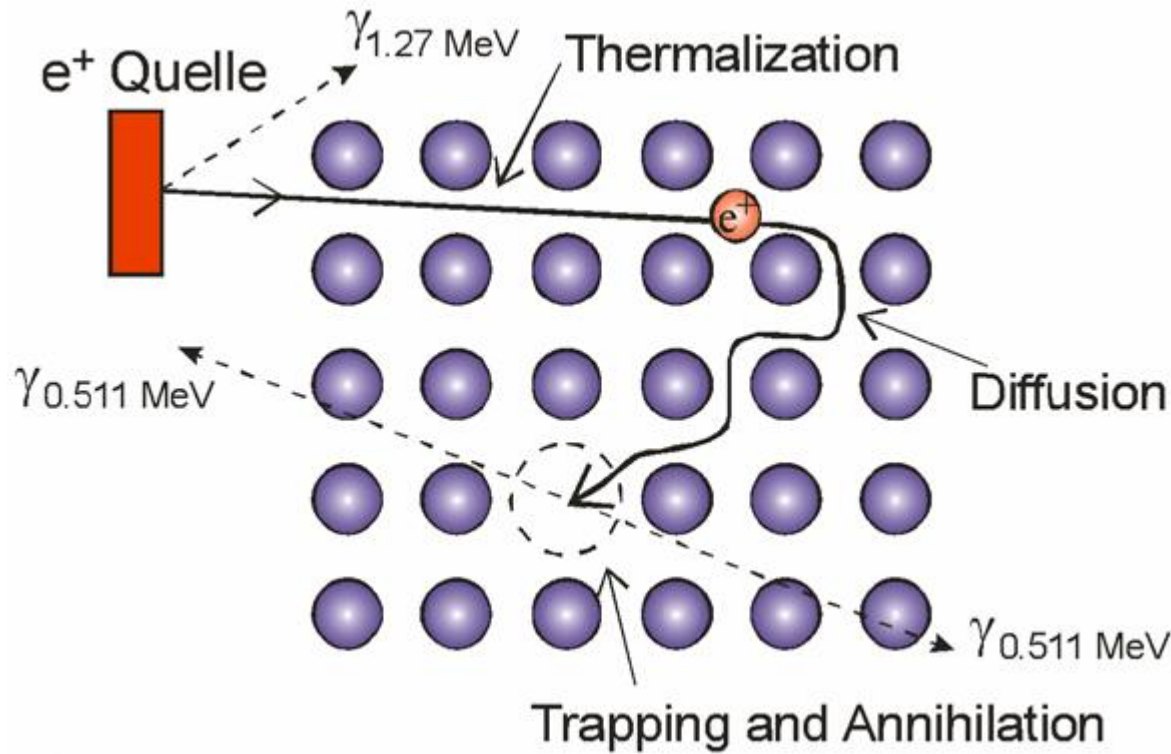
FIG. 1. Positron-lifetime spectra after source-background subtraction in electron-irradiated ($6 \times 10^{18} e^-/\text{cm}^2$) high-purity iron at various stages of isochronal annealing. The dramatic occurrence of a long-lifetime component after 230 K annealing is clearly visible.

- positron lifetime is very sensitive for vacancy-type defects
- here: lifetime increases after irradiation
- and further increase after first annealing: vacancy clustering



The positron lifetime spectroscopy

^{22}Na



- positron wave-function can be localized in the attractive potential of a defect
- annihilation parameters change in the localized state
- e.g. positron lifetime increases in a vacancy
- lifetime is measured as time difference between appearance of 1.27 (start) and 0.51 MeV (stop) quanta
- defect identification and quantification possible

Defect Sensitivity



atomic open-volume defects

- vacancies ($\rho_v > 10^{-7}$)
- vacancy clusters ($n=1...50$)
- dislocations ($> 10^8 \text{ cm}^{-2}$)
- grain boundaries (only ultra-fine grained materials)
- surface

non-open volume defects

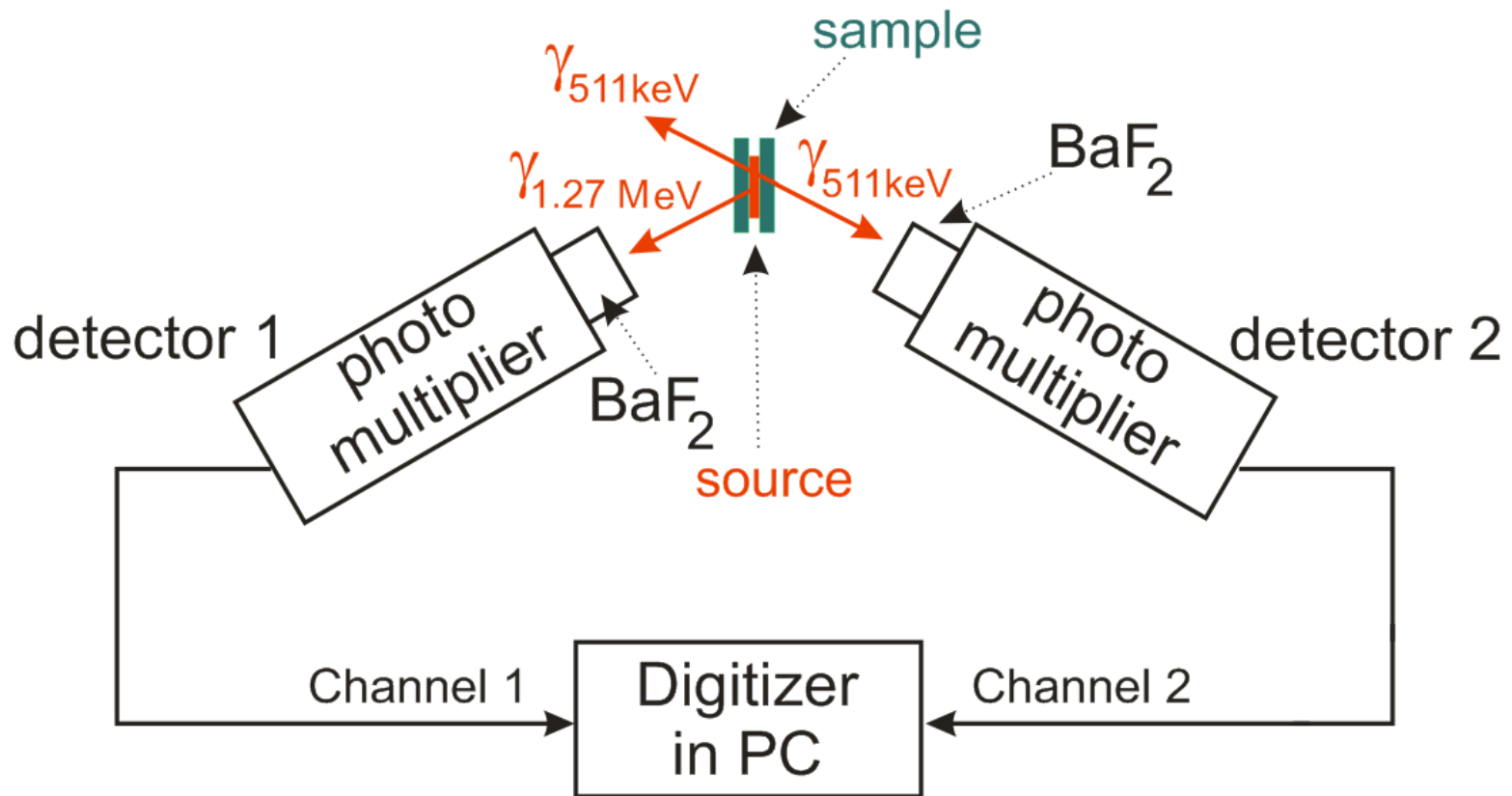
- coherent precipitates (e.g. GPZ in Al-Zn)
- negatively charged acceptors in semiconductors ("shallow traps")

large open volume 1...50 nm (Positronium formation)

- open volume between molecular chains in polymers ($> 100 \text{ \AA}^3$)
- mesoporous dielectrics ($1 \text{ nm} < d_{\text{pore}} < 50 \text{ nm}$)

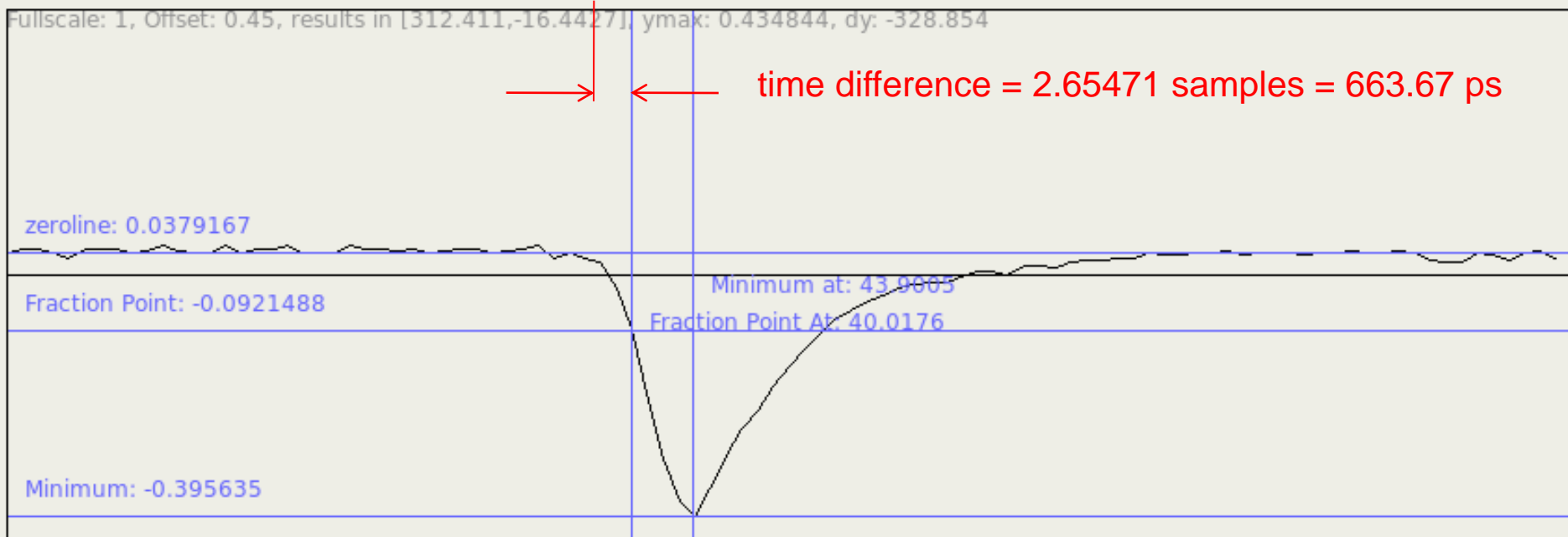
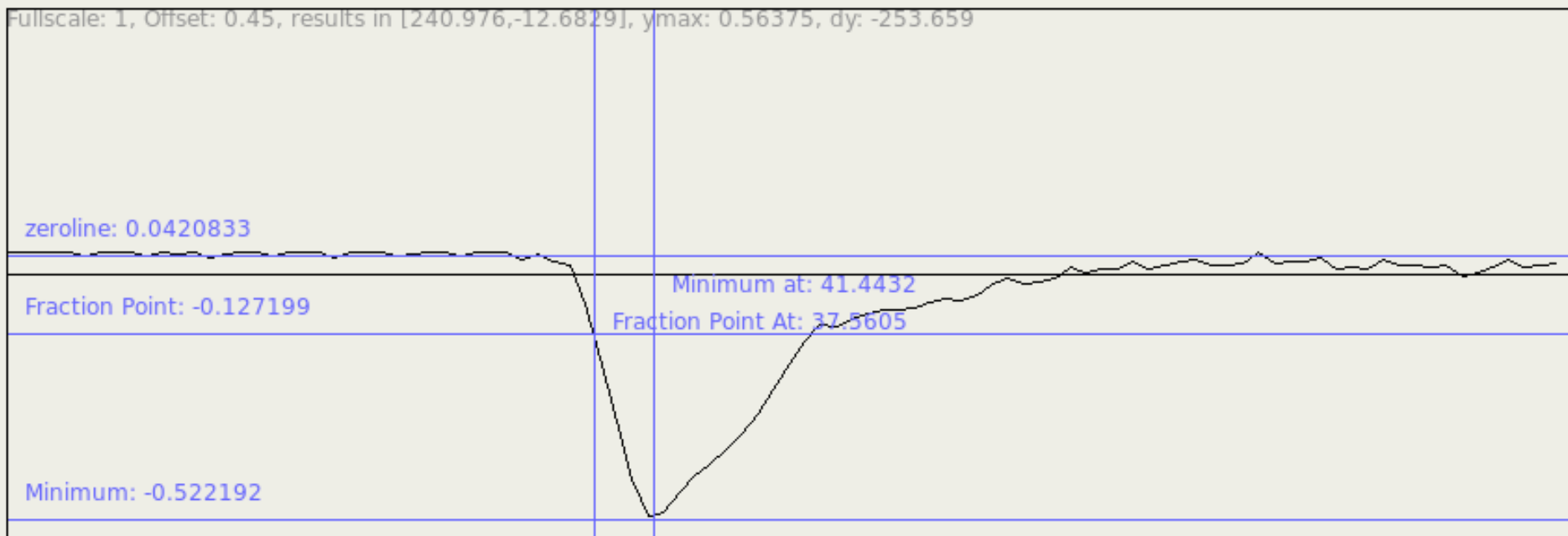


Digital lifetime measurement

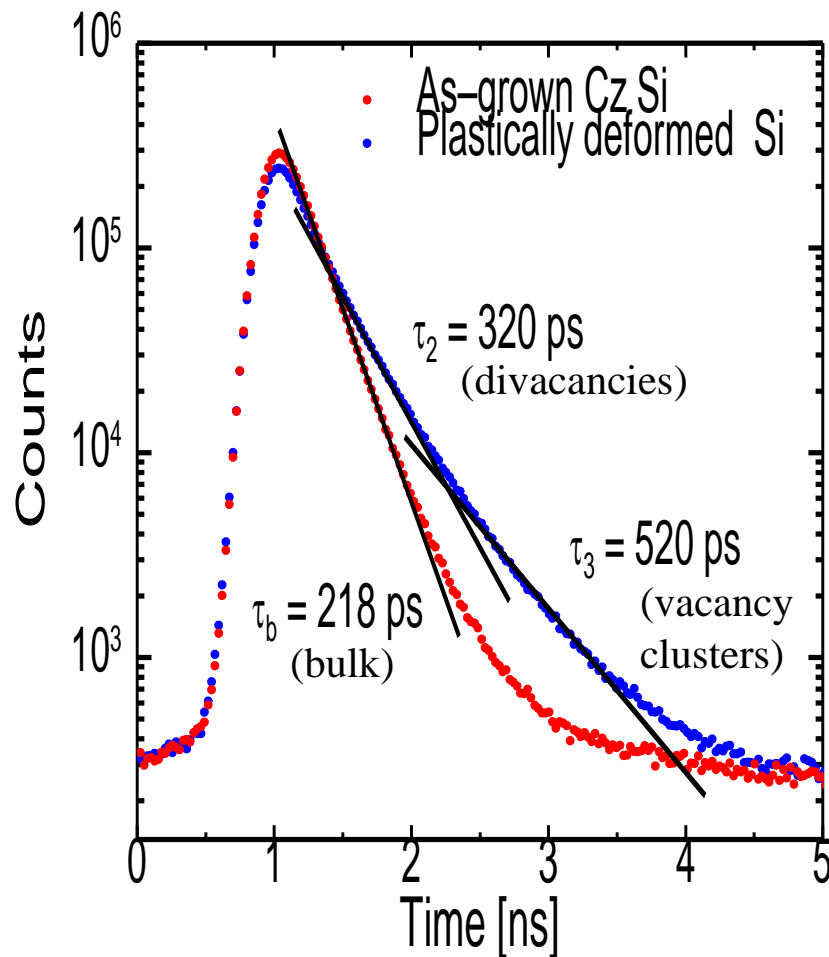


- very simple setup
- timing very accurate
- pulse-shape discrimination (suppress "bad pulses")
- each detector for start & stop (double statistics)

screenshot of two digitized anode pulses



Positron lifetime spectroscopy



- positron lifetime spectra consist of exponential decay components
- positron trapping in open-volume defects leads to long-lived components
- longer lifetime due to lower electron density
- analysis by non-linear fitting: lifetimes τ_i and intensities I_i

positron lifetime spectrum:

$$N(t) = \sum_{i=1}^{k+1} \frac{I_i}{\tau_i} \exp\left(-\frac{t}{\tau_i}\right)$$

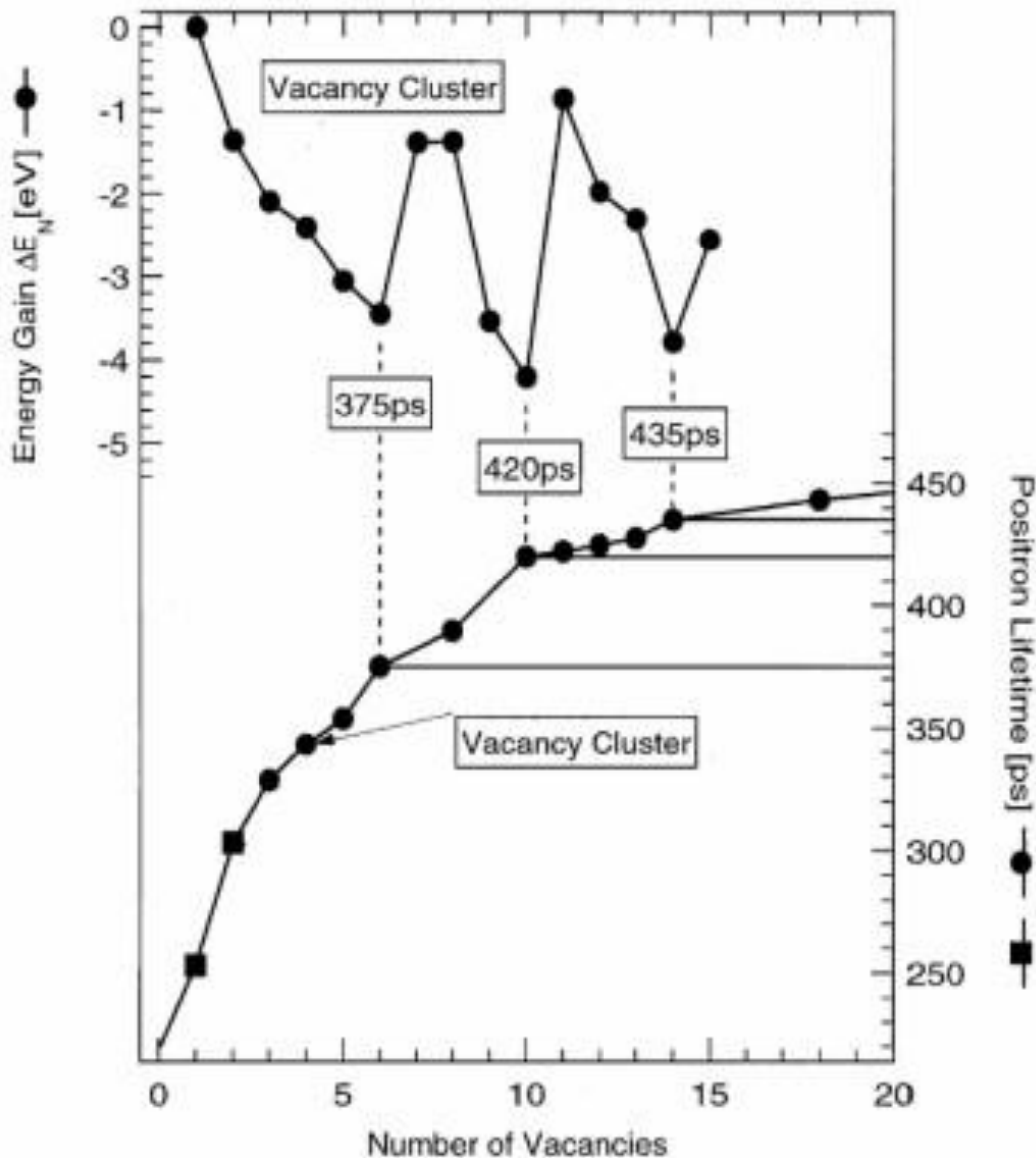
trapping coefficient

$$k_d = \mu C_d = \frac{I_2}{I_1} \left(\frac{1}{\tau_b} - \frac{1}{\tau_d} \right)$$

trapping rate defect concentration



Theoretical calculation of vacancy clusters in Si



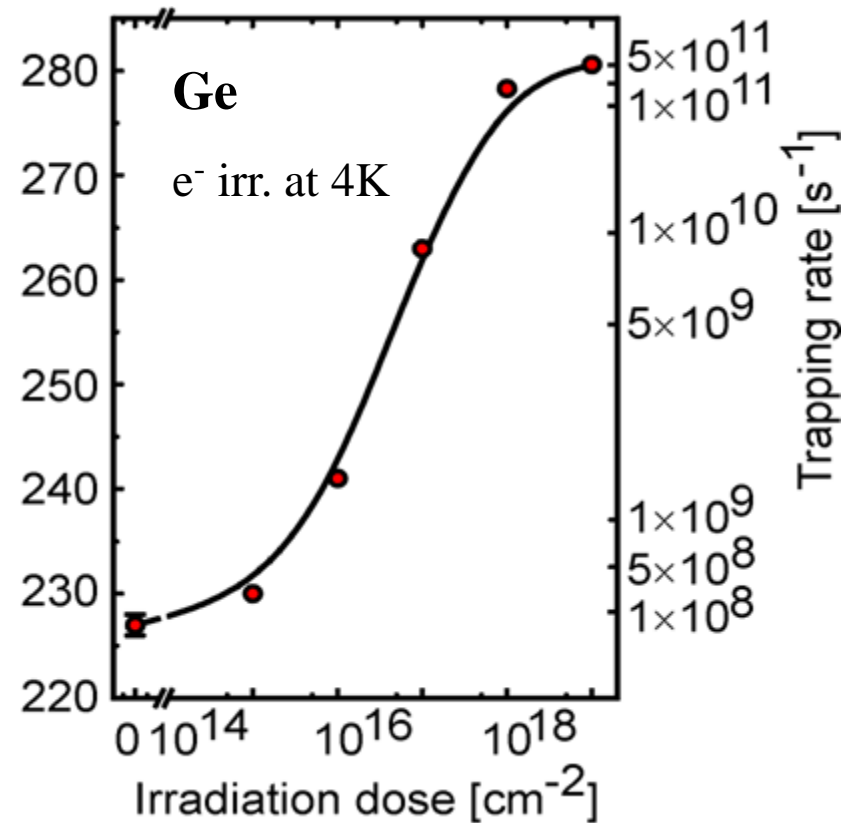
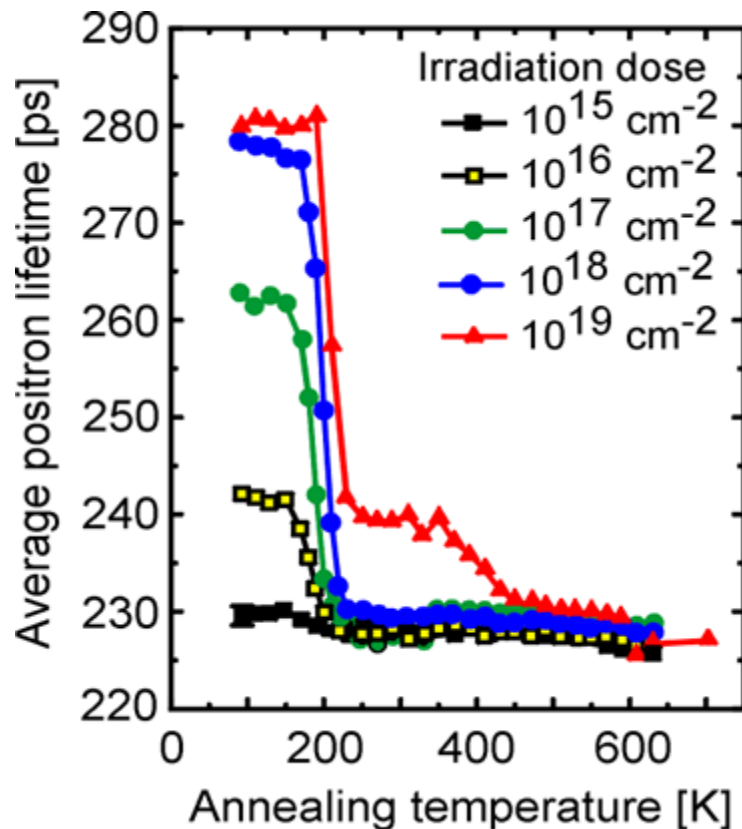
- there are cluster configurations with a large energy gain
- „Magic Numbers“ with 6, 10 und 14 vacancies
- positron lifetime increases distinctly with cluster size
- for $n > 10$ saturation effect, i.e. size cannot be determined

T.E.M. Staab et al.,
Physica B 273-274 (1999) 501-504



Defects in electron-irradiated Ge

- Electron irradiation (2 MeV @ 4K) induces Frenkel pairs (vacancy - interstitial pairs)
- steep annealing stage at 200 K
- at high irradiation dose: divacancies are formed (thermally more stable)

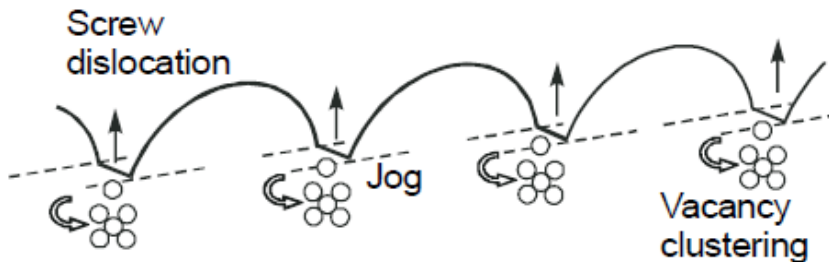


(Polity et al., 1997)

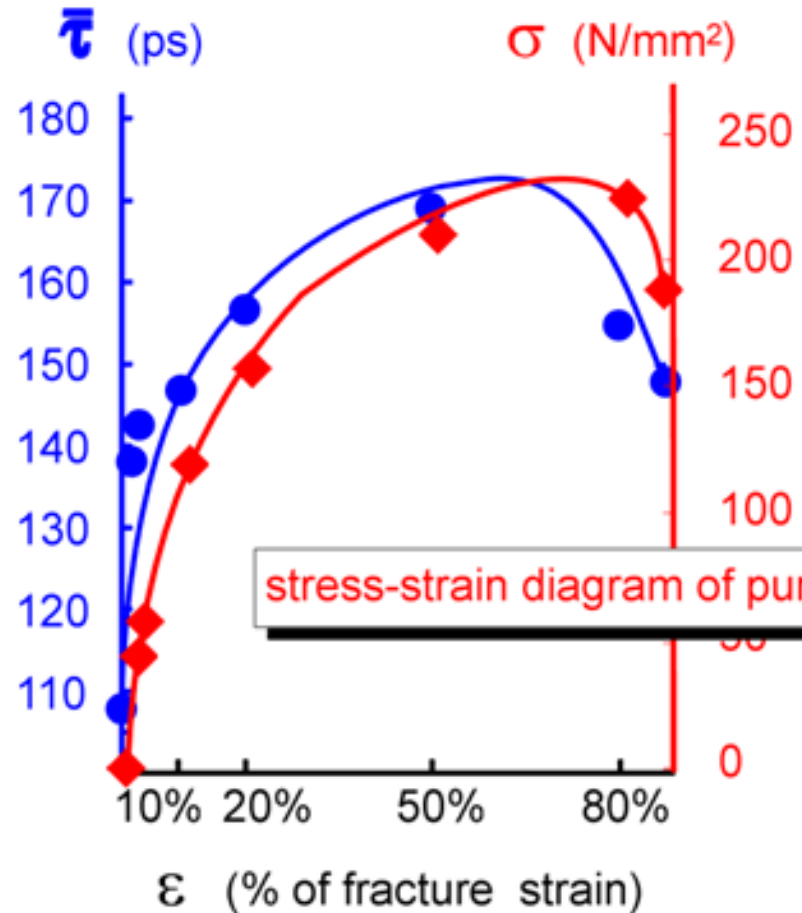


Defects in Iron after tensile Strength in Stress-Strain Experiment

- extensive study of defects in mechanically damaged iron and steel
- sensitive: detection of defects already in the elastic Hooke's range
- Vacancy cluster and dislocations are detectable in both cases
- small vacancy clusters are generated by jog dragging process



average positron lifetime in pure iron after tensile strain

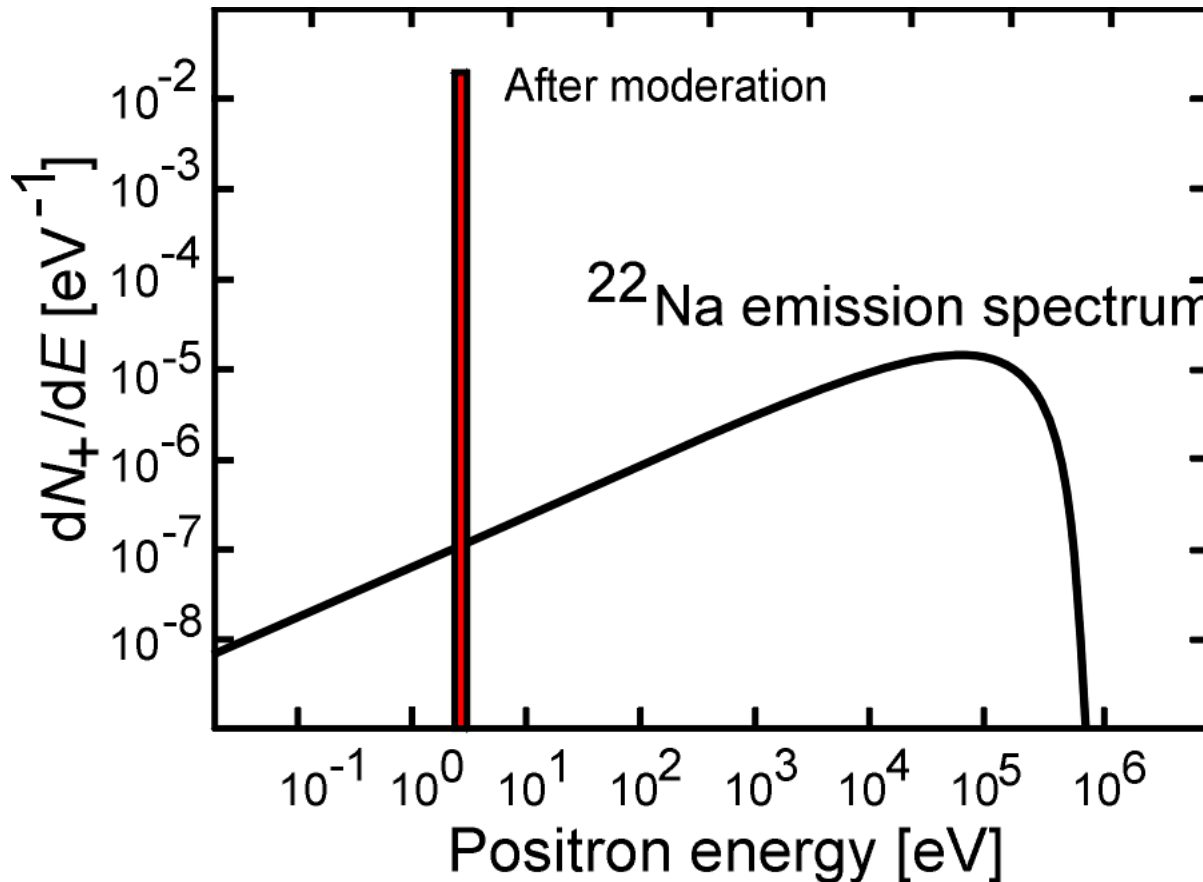


stress-strain diagram of pure iron

Somieski et al., J. Physique IV 5, C1/127-134 (1995)

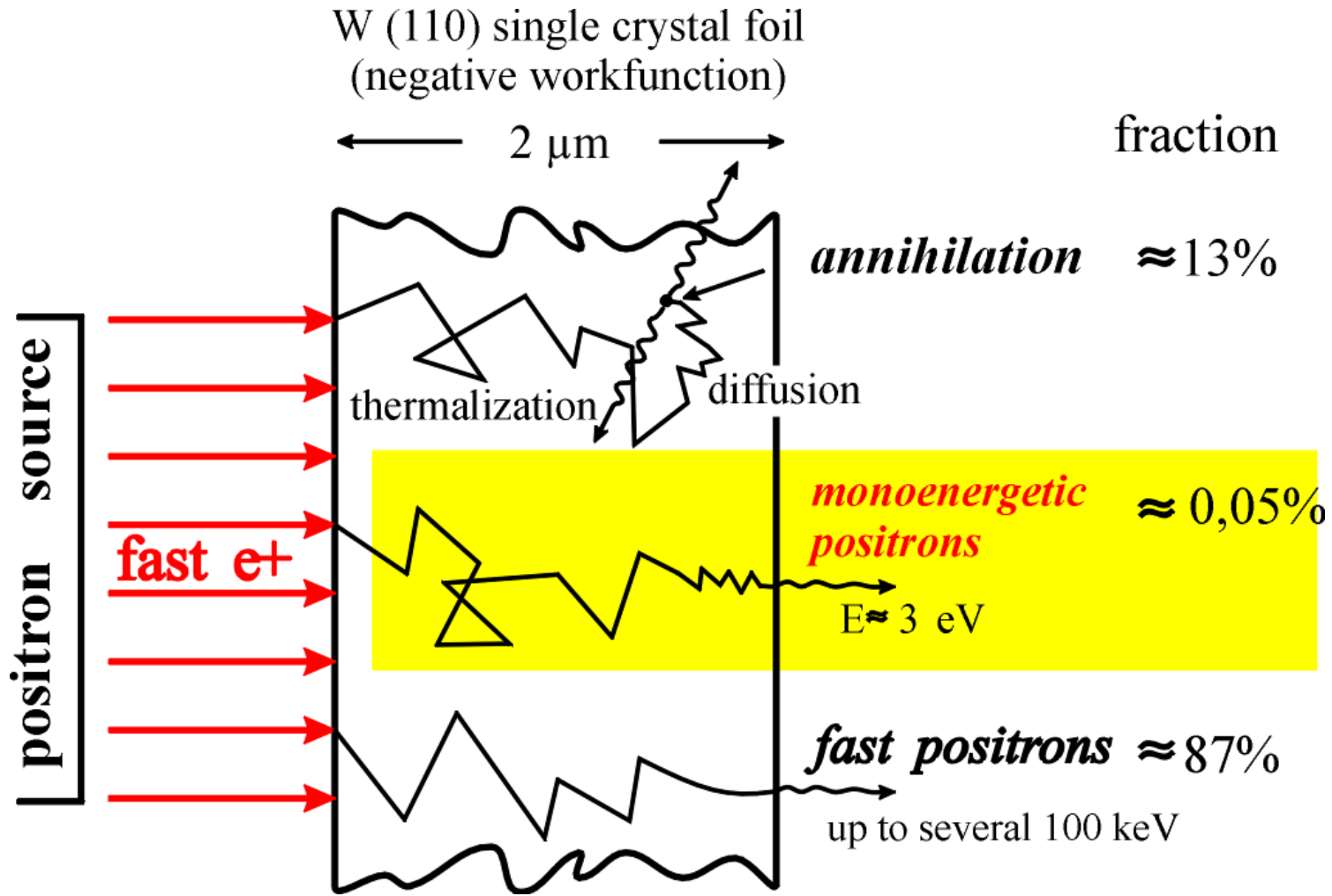
Moderation of Positrons

Mean implantation depth of un-moderated positrons from a ^{22}Na isotope source ($1/e$) for Si: $50\mu\text{m}$



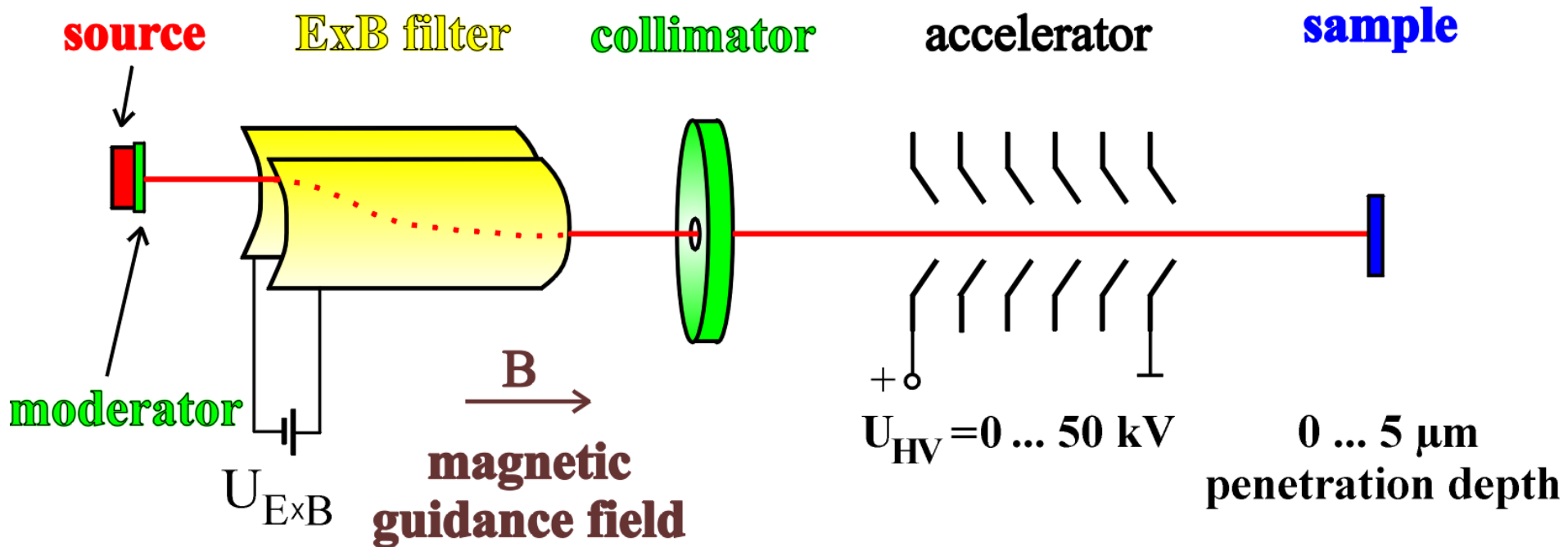
- broad β^+ positron emission spectrum
- deep implantation into solids
- not useful for study of defects in thin layers
- for defect depth profiling: moderation necessary
- monoenergetic positrons can be implanted to different depth

Moderation of Positrons



moderation efficiency: $\approx 10^{-4}$

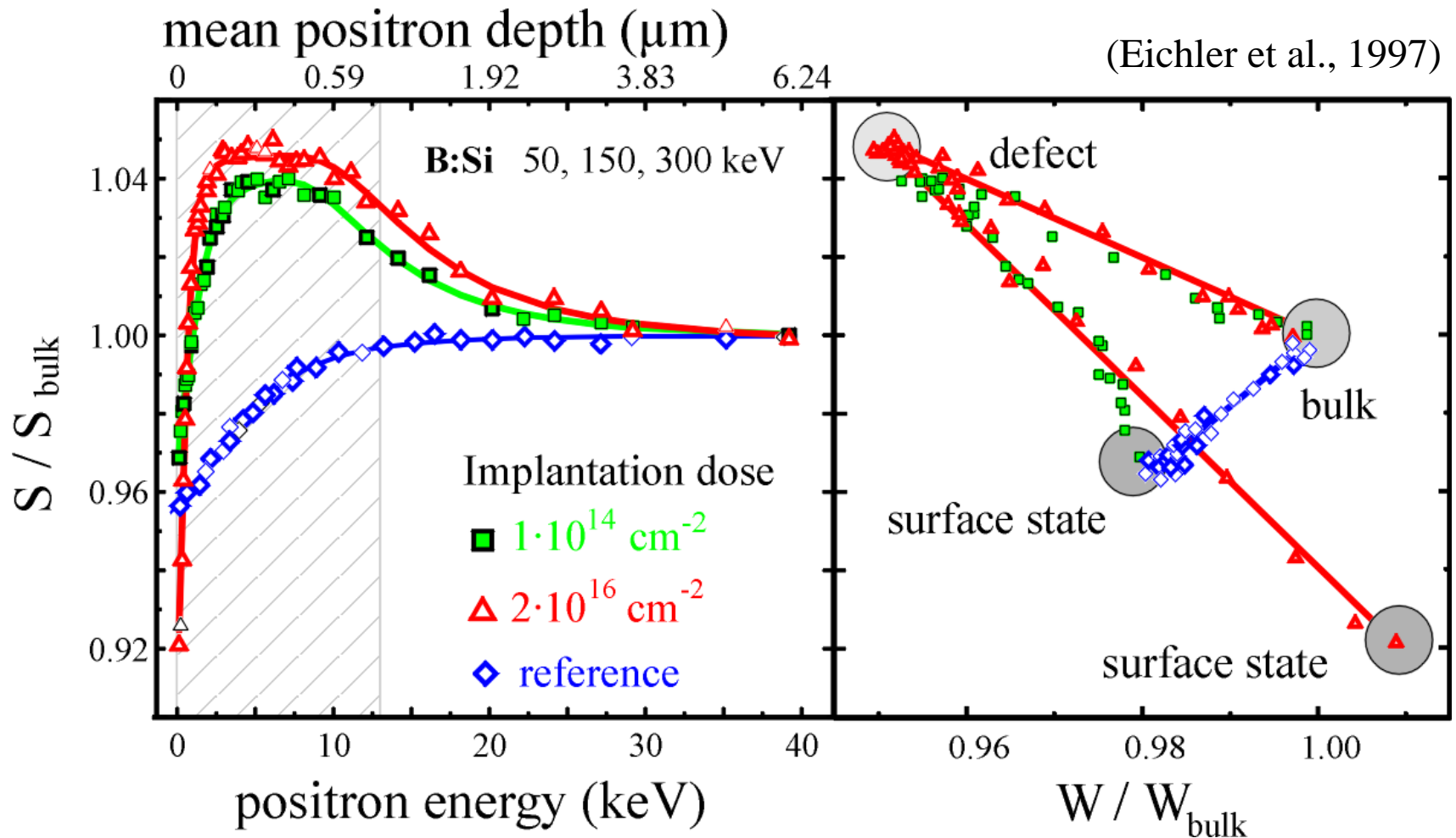
The Positron Beam System at Halle University



- positron lifetime measurement not any more possible
- way out: continuous beam must be chopped and bunched
- only two systems at intense sources available: FRM-II and Tsukuba

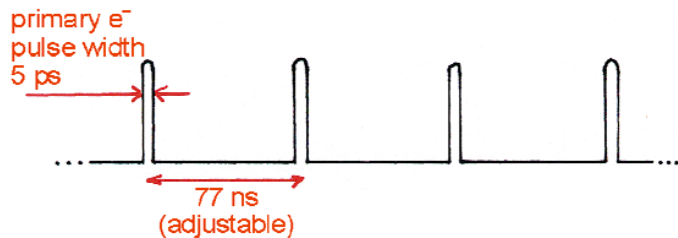
Defects in Si induced by Ion Implantation

- ion implantation is most important doping technique in planar technology
- main problem: generation of defects \Rightarrow positron beam measurements



EPOS = ELBE Positron Source

- ELBE -> electron LINAC (40 MeV and up to 40 kW) in Research Center Dresden-Rossendorf
- EPOS -> collaboration of Univ. Halle with FZD
- EPOS will be the combination of a positron lifetime spectrometer, Doppler coincidence, and AMOC
- User-dedicated facility
- main features:
 - high-intensity bunched positron beam ($E_+ = 0.5...30$ keV)
 - very good time resolution by using the unique primary time structure of ELBE
 - digital multi-detector array
 - fully remote control via internet by user



Concept of EPOS (ELBE Positron Source)

MePS

Monoenergetic Positron Spectroscopy

- Cave 111b / Lab 111d
- monoenergetic (slow) positrons
- pulsed system
- LT, CDBS, AMOC
- Still under construction

Information Depth:
0...5 μm

CoPS

Conventional Positron Spectroscopy

- LT, CDBS, AMOC
- using ^{22}Na foil sources
- He-cryostat
- automated system
- digital detector system

Information Depth:
10...200 μm

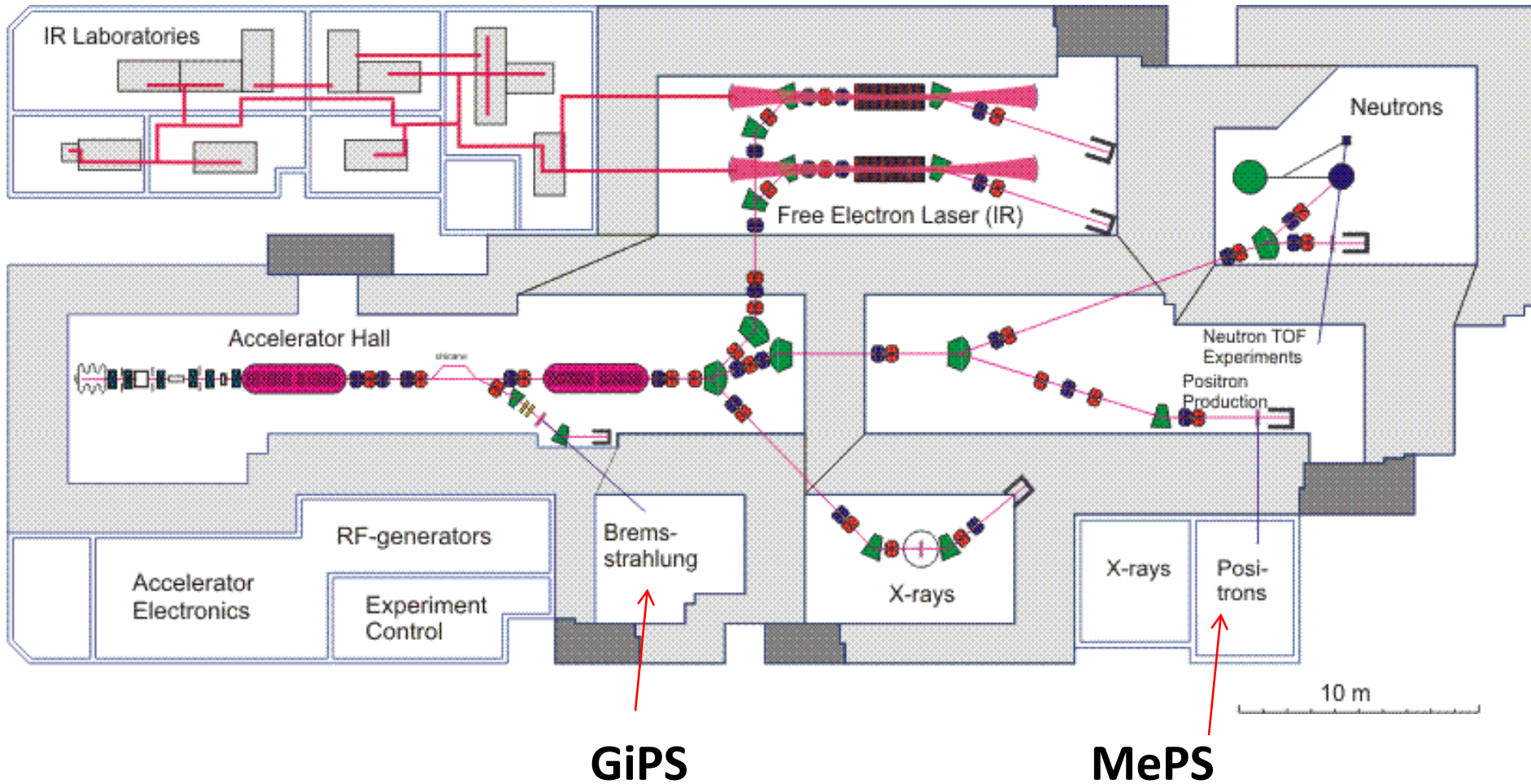
GiPS

Gamma-induced Positron Spectroscopy

- Cave 109 (nuclear physics)
- Positron generation by Bremsstrahlung
- Information in complete bulky sample (up to 100 cm^3)
- all relevant positron techniques (LT, CDBS, AMOC)

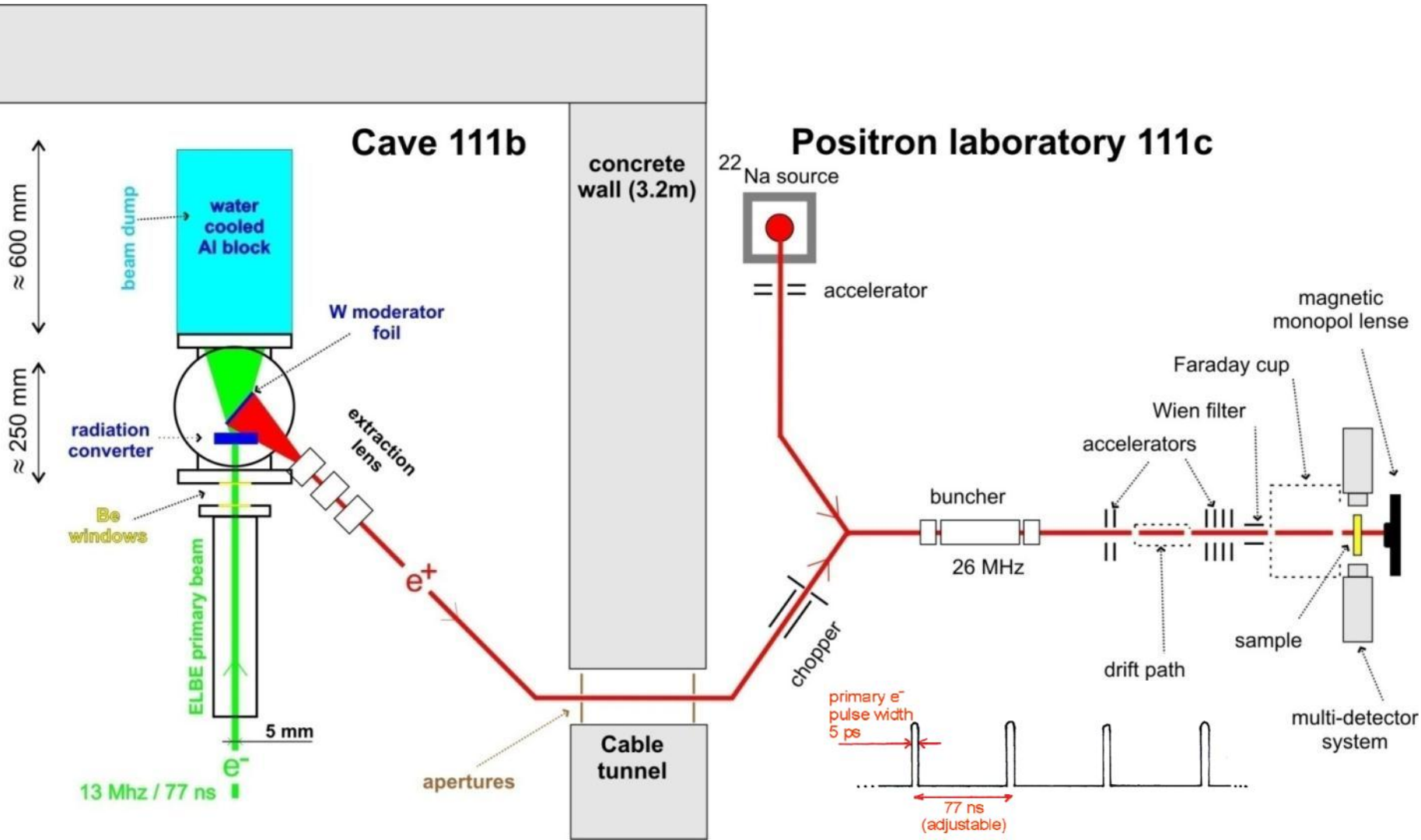
Information Depth:
0.1 mm ... 5 cm

Ground plan of the ELBE hall



Progress of Mono-energetic Positron Beam

- 40 MeV, 1 mA, 26 MHz repetition time in cw mode; lifetime, CDBS and AMOC with slow e^+
- Retain original time structure for simplicity and best time resolution

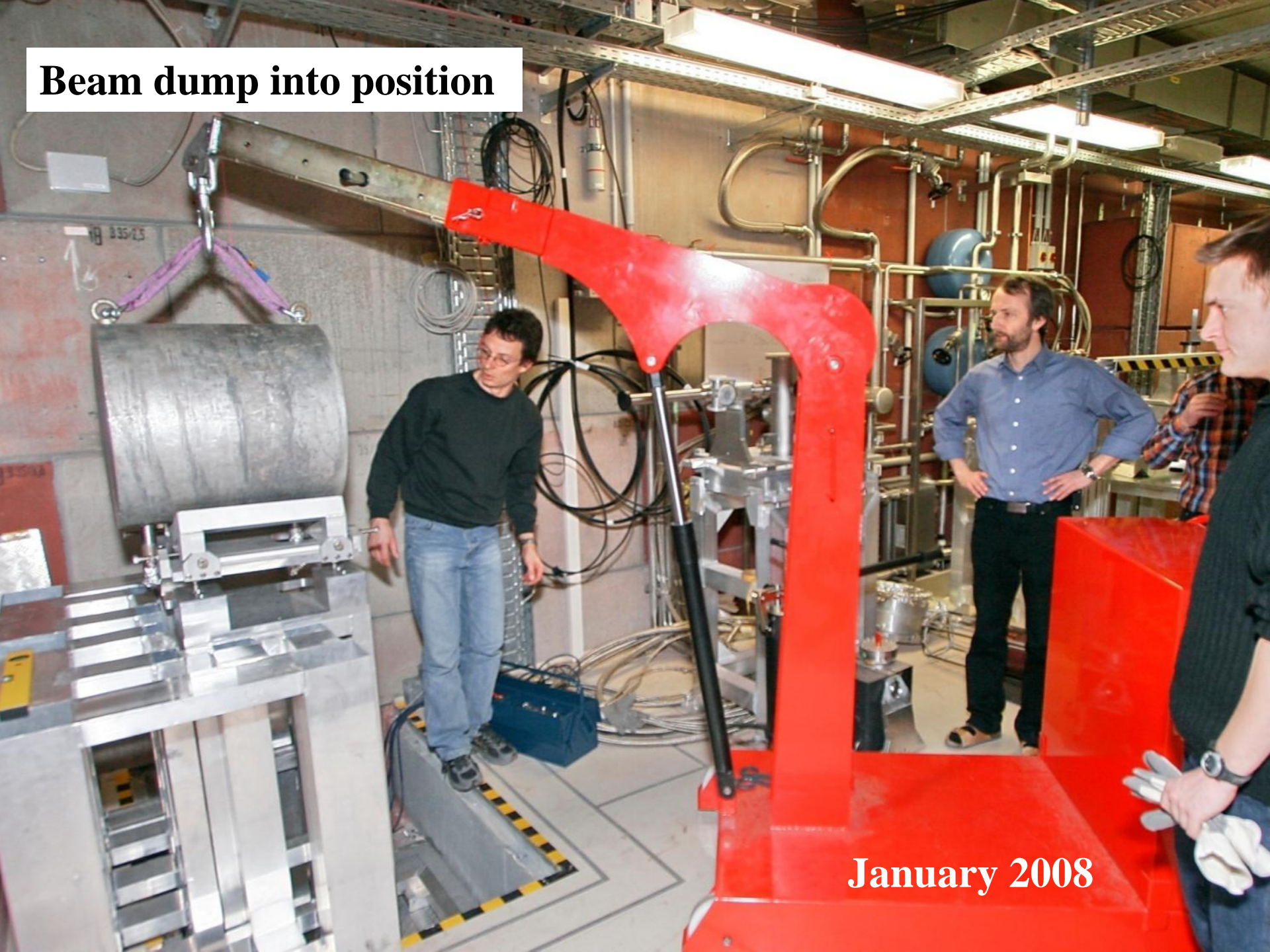


November 2007



Start of Mounting

Beam dump into position



January 2008

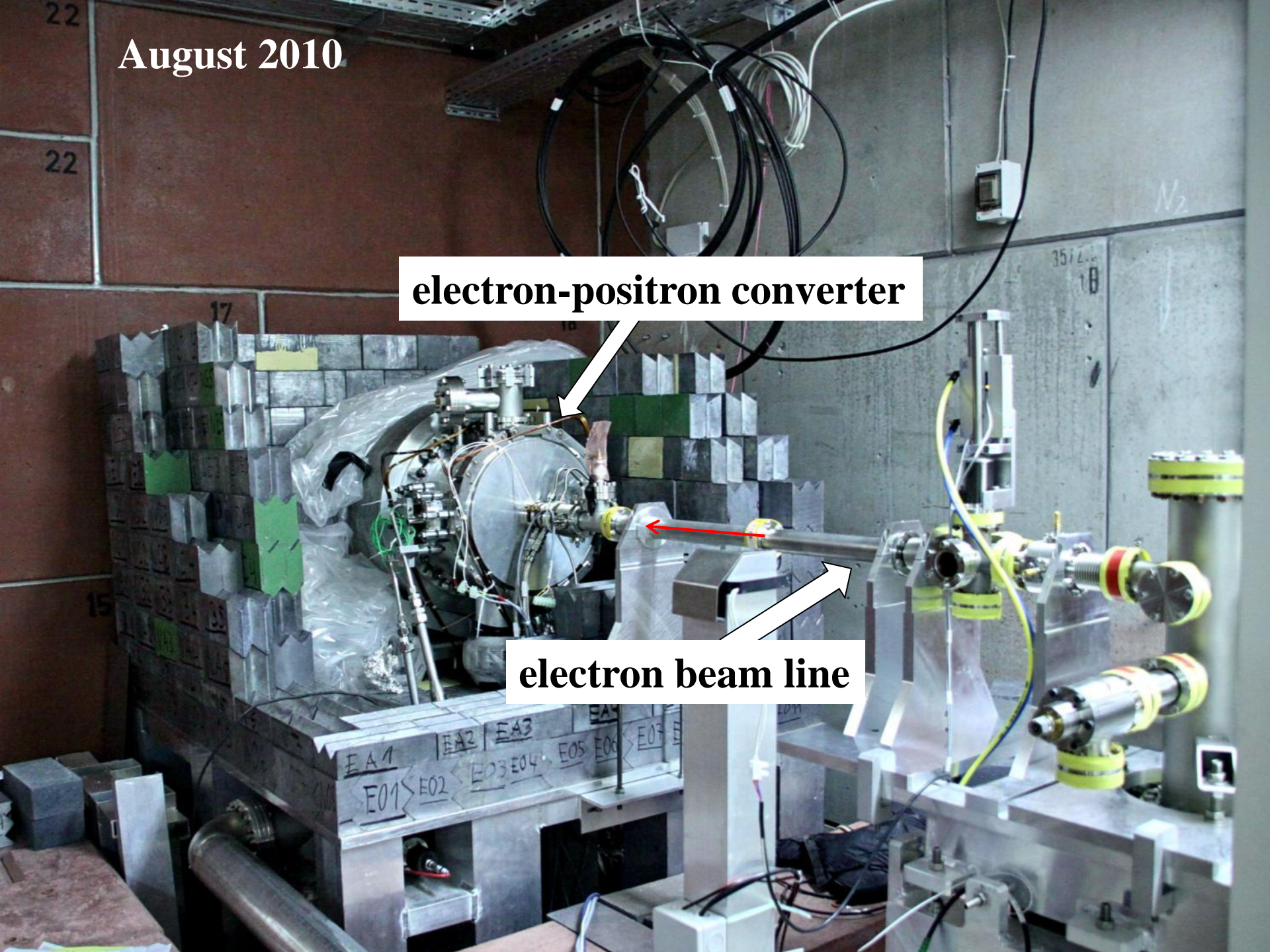
January 2009



22
22
17
15
August 2010

electron-positron converter

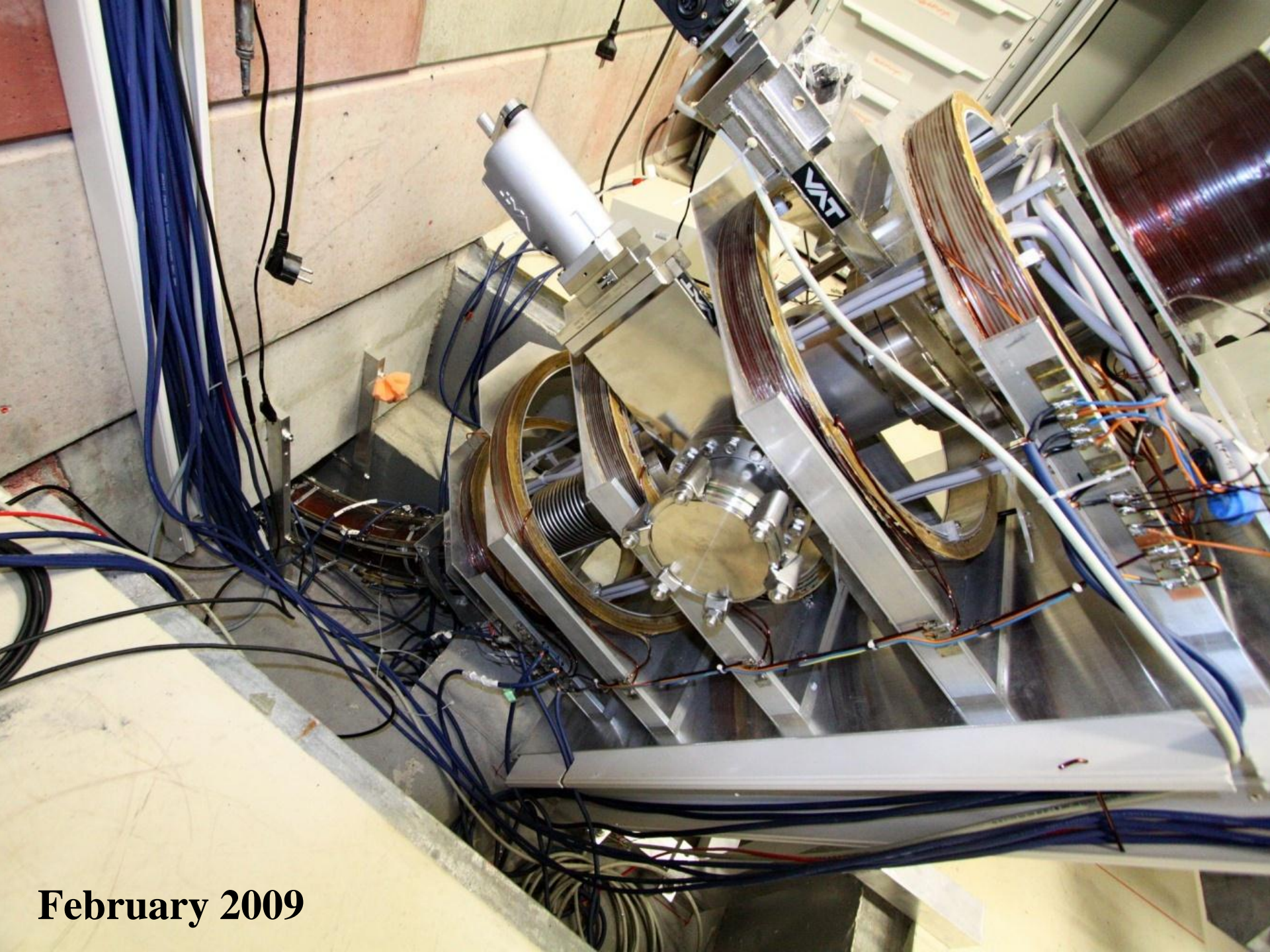
electron beam line



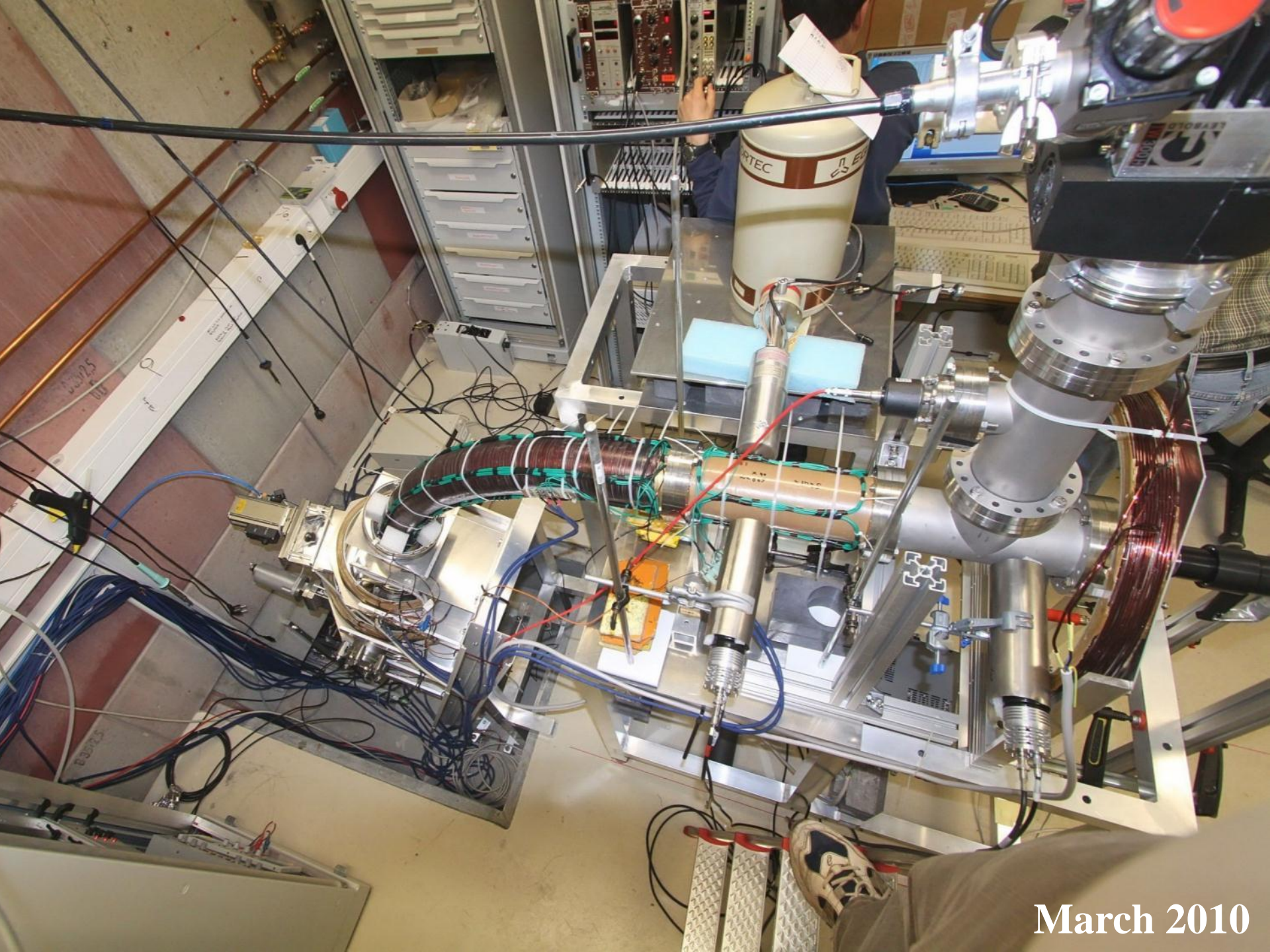
A photograph of a beamline test setup. The central focus is a circular, metallic component with a glowing green light source inside. This component is surrounded by a ring of small, circular ports. The entire setup is housed within a dark, industrial-looking enclosure. The text "Test of beamline with electrons" is overlaid in the upper right, and "July 2008" is overlaid in the bottom right.

Test of beamline with electrons

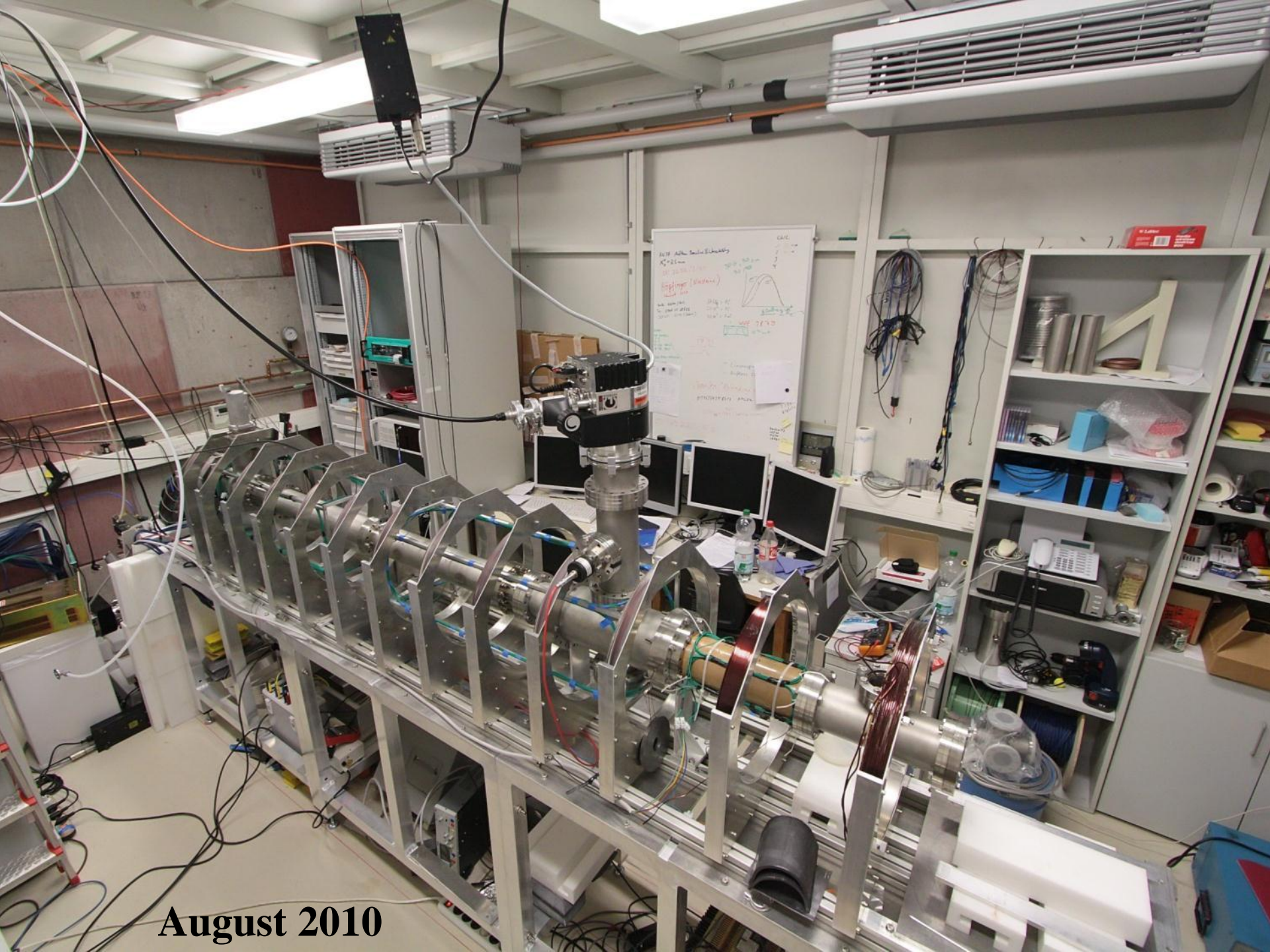
July 2008



February 2009

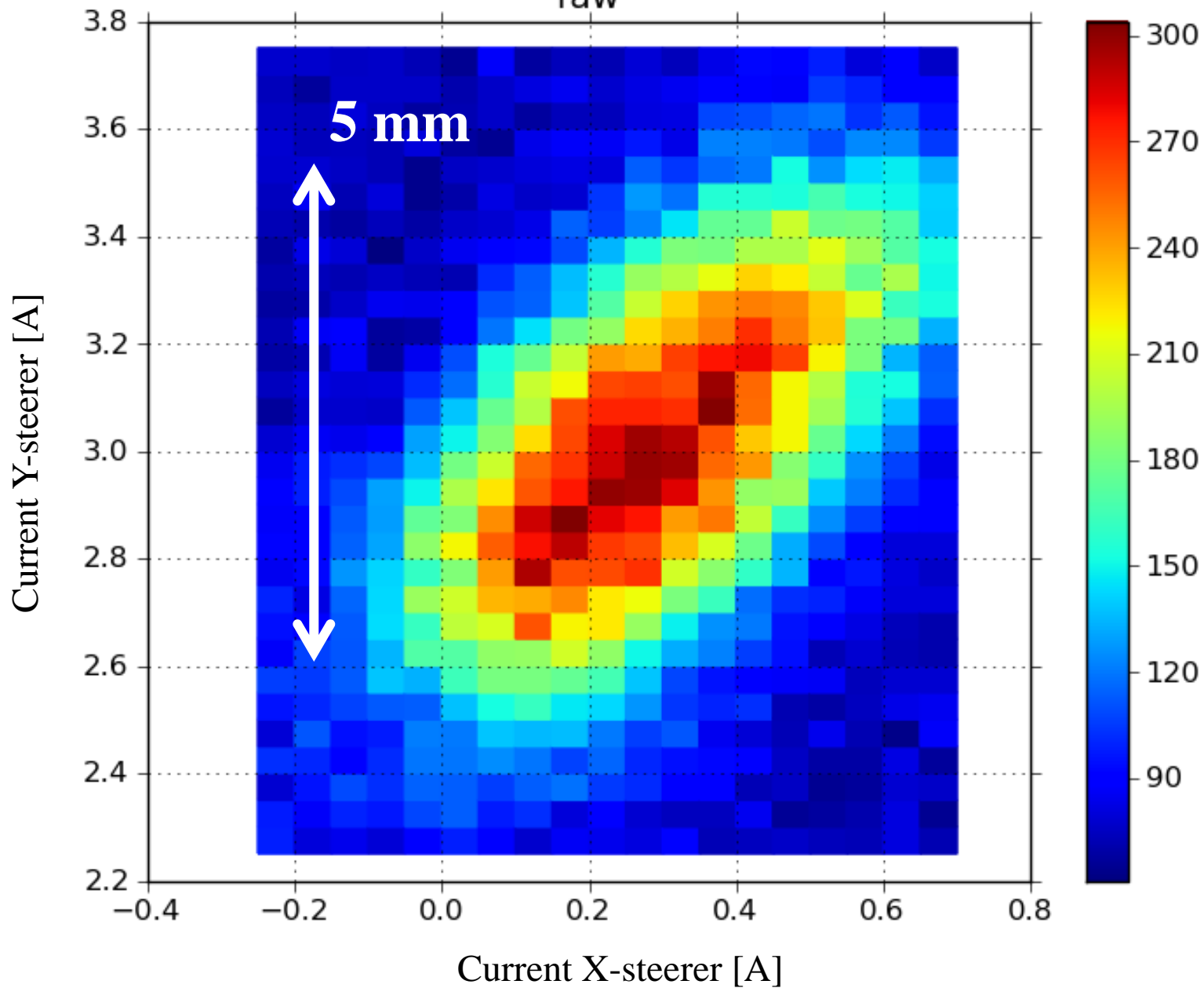


March 2010

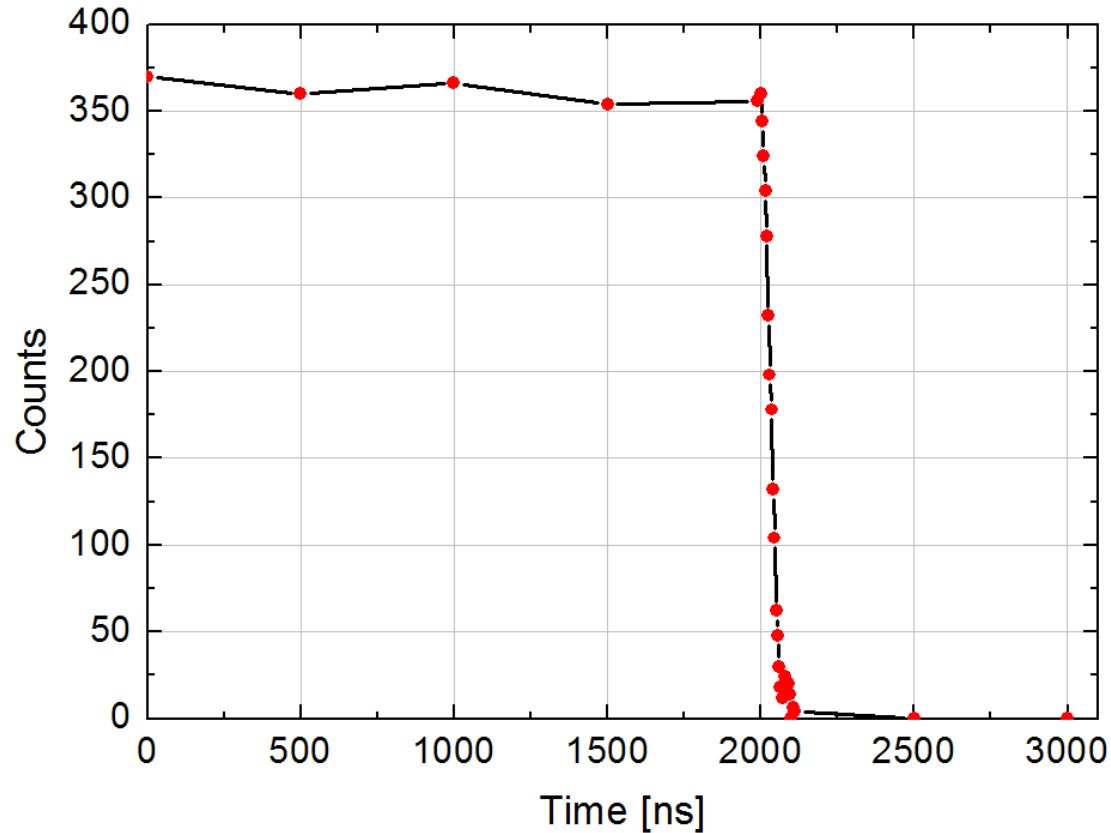


August 2010

raw

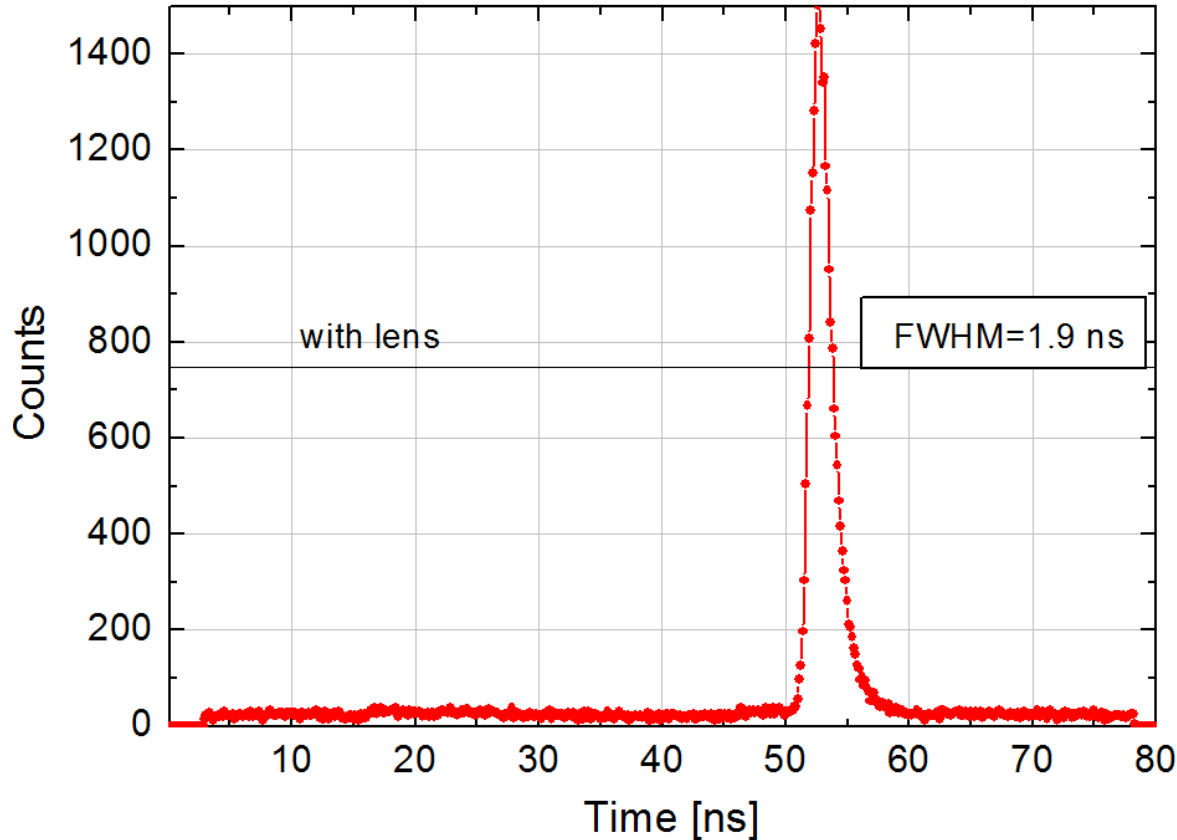


Measurement of Energy Distribution by retarding Grid



- electrostatic lens in action
- 2 apertures of 5mm were mounted in a distance of half a gyration length (63 mm)

GiPS: Gamma-induced Positron Spectroscopy



- using the double aperture: time structure very useful and according to former simulation
- still missing: Chopper signal - must be 2 ns / >500V / 13 MHz repetition frequency
- time width of < 50 ps expected with chopper and buncher

Positron Annihilation Spectroscopy: Applications

Variety of applications in all fields of materials science:

- bulk defects in semiconductors, ceramics and metals
- defect-depth profiles due to surface modifications (ion implantation; tribology)
- epitaxial layers (growth defects, misfit defects at interface, ...)
- soft matter physics (open volume; interdiffusion; ...)
- porosimetry 1...50 nm (e.g. low-k materials - highly porous dielectric layers)
- fast kinetics (e.g. precipitation processes in Al alloys; defect annealing; diffusion; ...)
- radiation resistance (e.g. space materials)
- many more ...

