

Ehrenkolloquium für Herrn Prof. J.R. Niklas: Antimaterie in der modernen Materialforschung



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- Technik der Positronenannihilation
- Anwendungen
- Gepulste, intensive Positronenquelle EPOS an ELBE (FZ Dresden-Rossendorf)

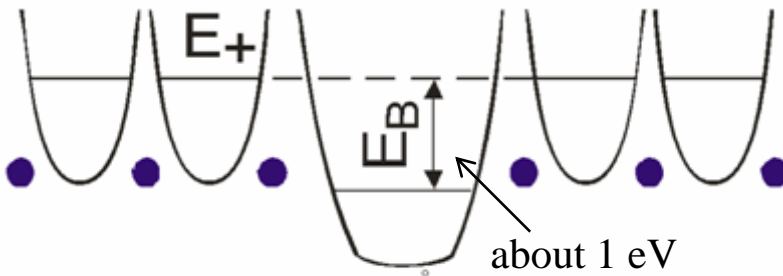
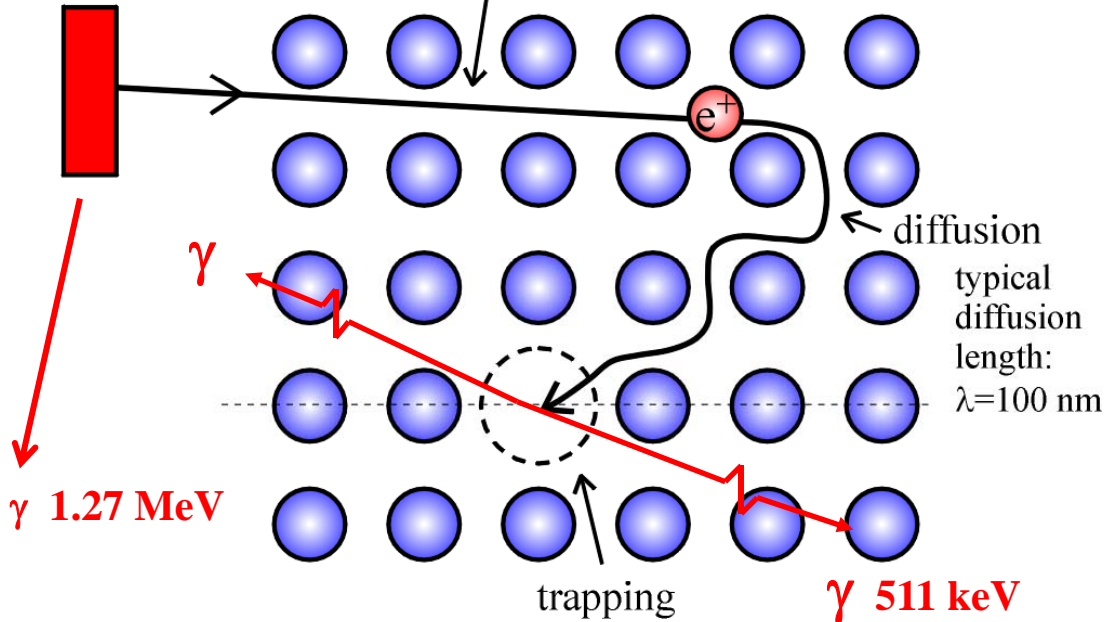


The positron lifetime spectroscopy

^{22}Na

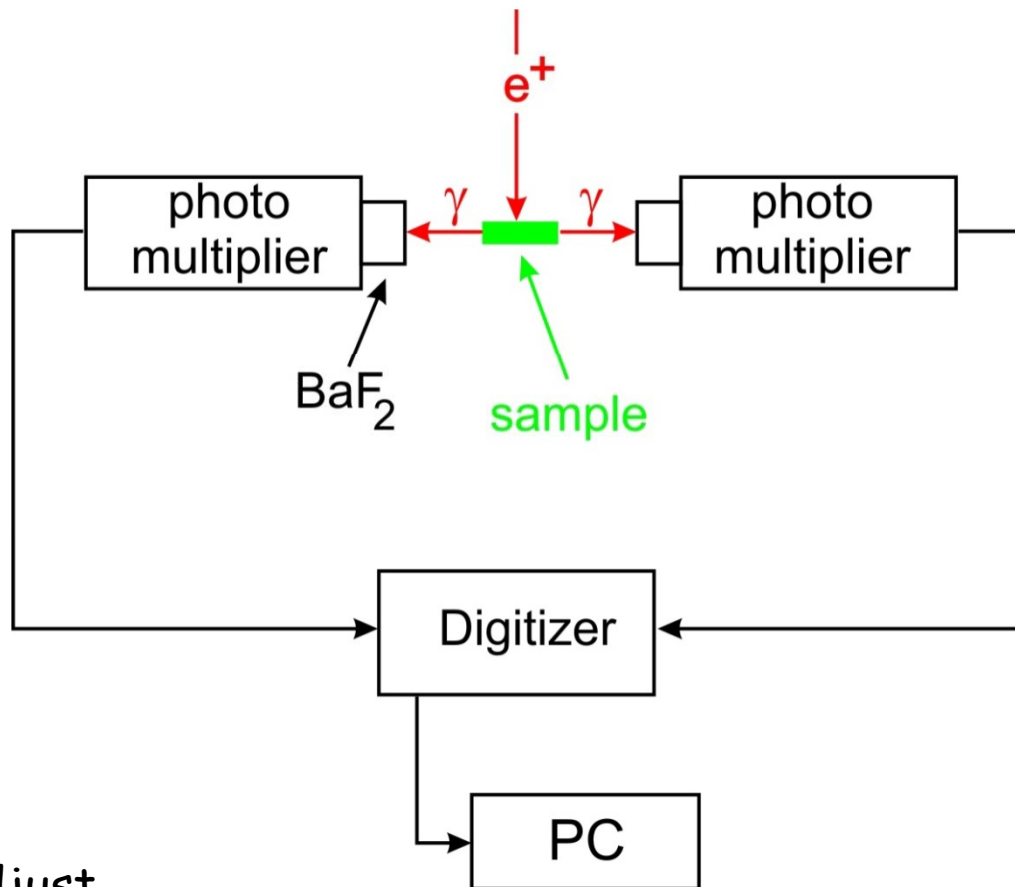
e^+ source

thermalization



- 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

Digital lifetime measurement



- simple setup
- nothing to adjust
- timing very accurate (accuracy 10^{-6})
- pulse-shape discrimination (suppress "bad pulses")
- each detector for start & stop (double statistics)

Screenshot of two digitized Anode Pulses

Fullscale: 1, Offset: 0.45, results in [240.976,-12.6829], ymax: 0.56375, dy: -253.659

zeroline: 0.0420833

Fraction Point: -0.127199

Minimum: -0.522192

Minimum at: 41.4432
Fraction Point At: 37.5605

Fullscale: 1, Offset: 0.45, results in [312.411,-16.4427], ymax: 0.434844, dy: -328.854

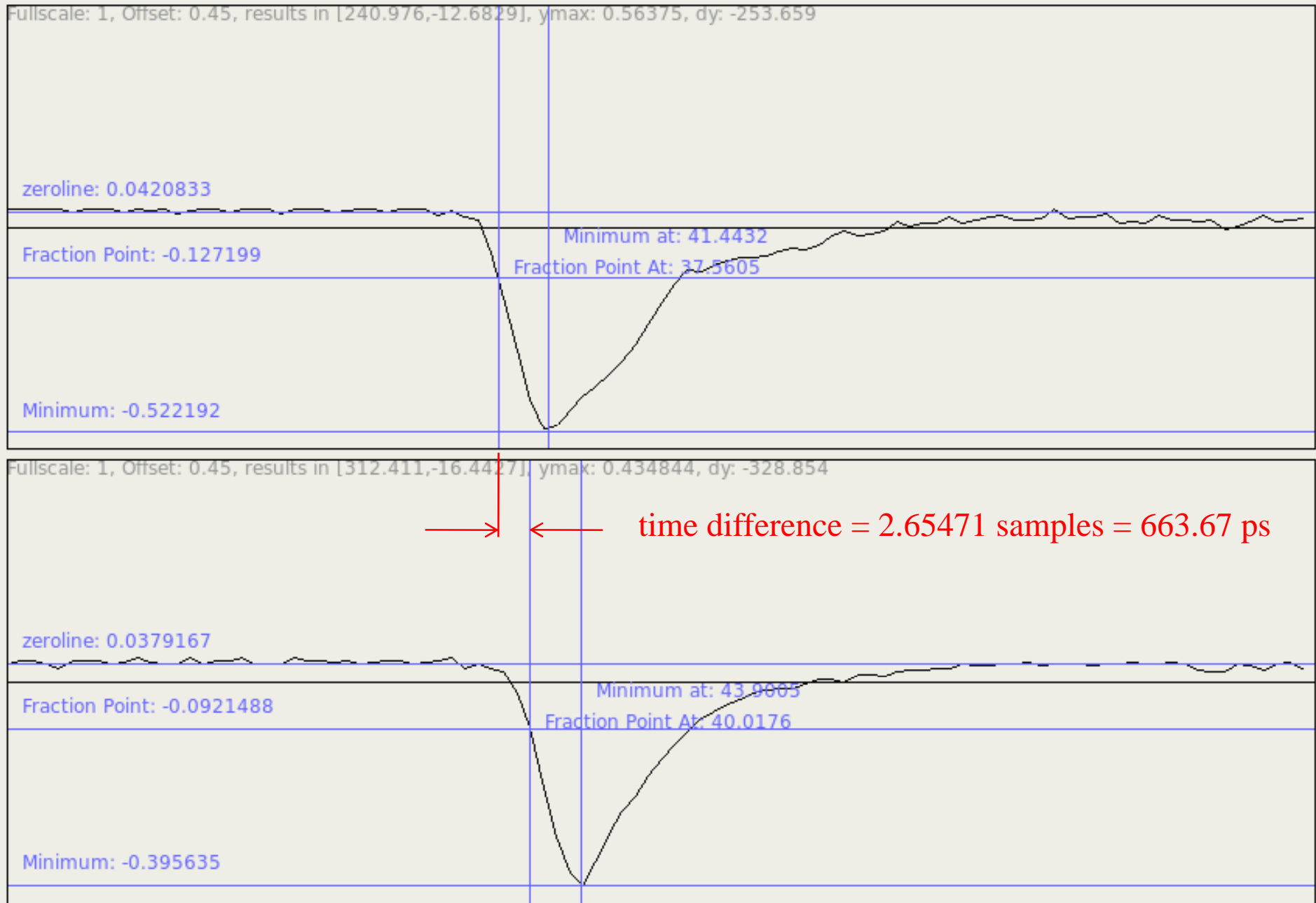
zeroline: 0.0379167

Fraction Point: -0.0921488

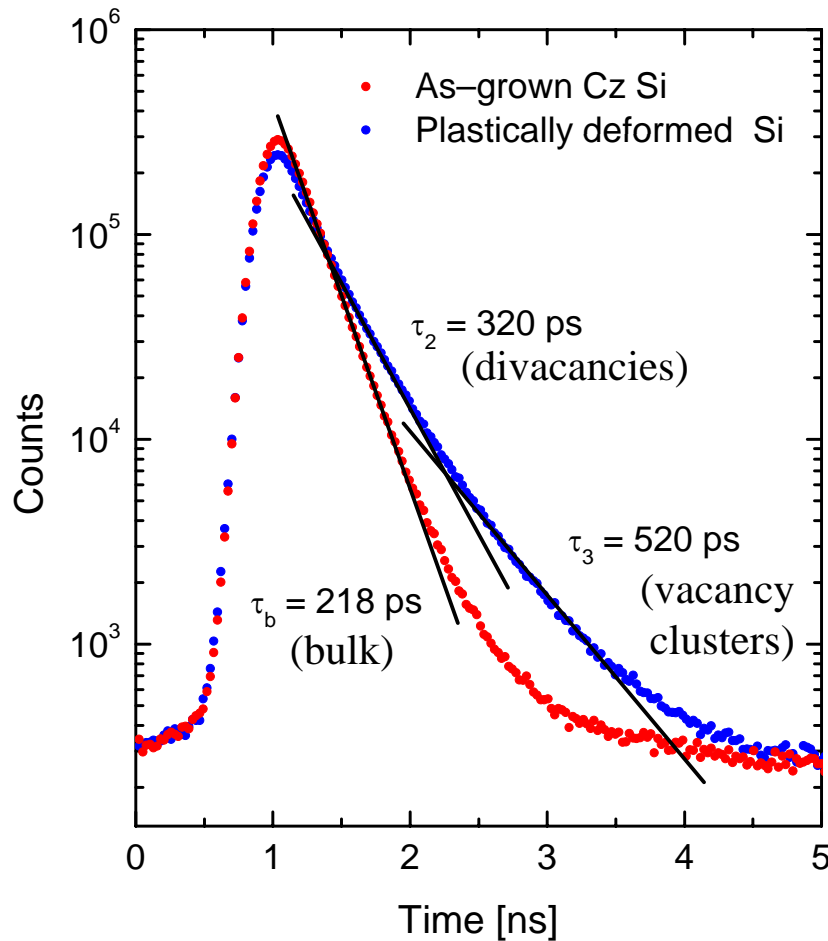
Minimum: -0.395635

Minimum at: 43.9885
Fraction Point At: 40.0176

time difference = 2.65471 samples = 663.67 ps



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

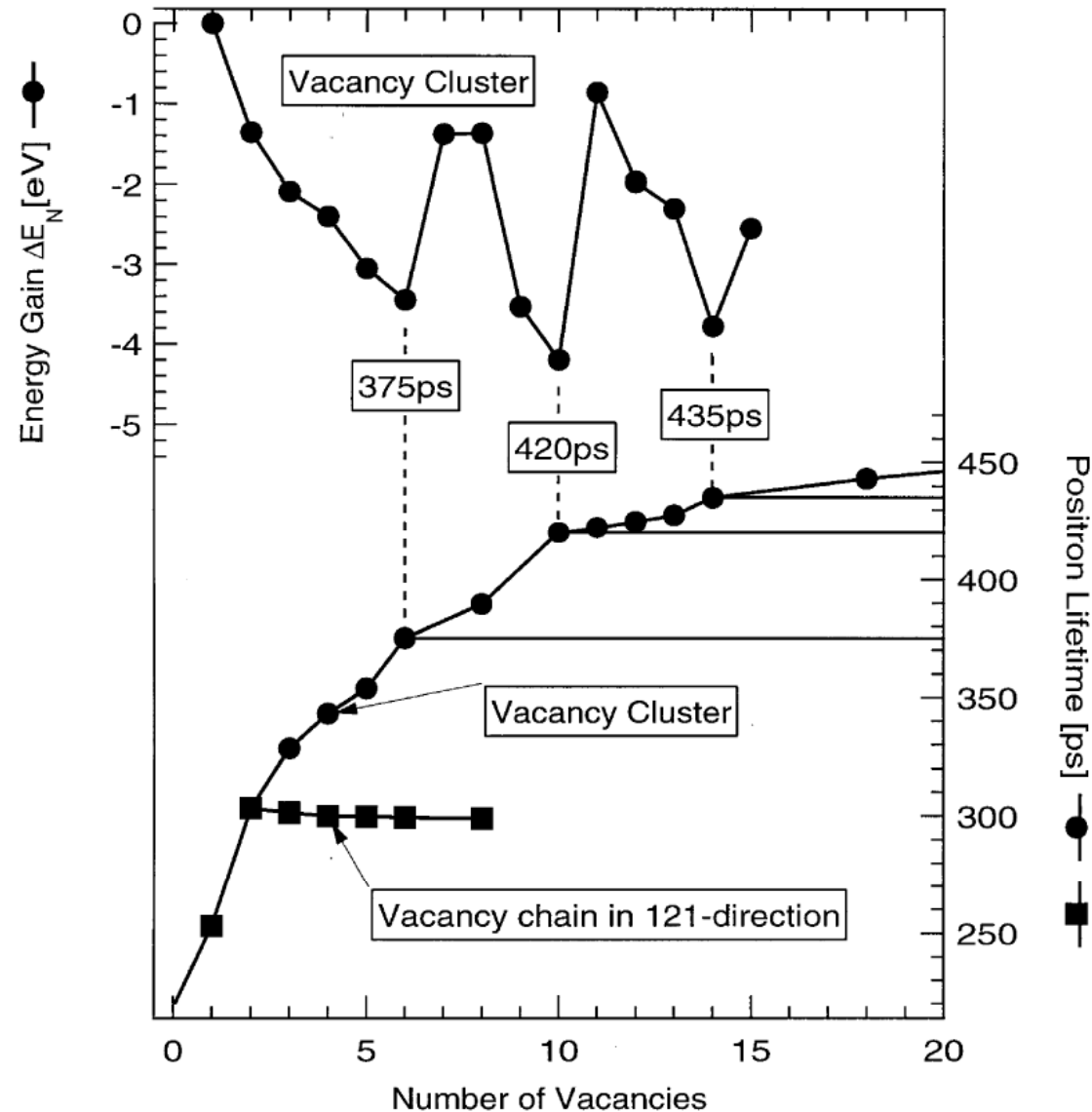


Detection Range of different Defects

Defect Type	Sensitivity range
	detection limit ... saturated trapping
neutral vacancies	$5 \times 10^{15} \dots 10^{19} \text{ cm}^{-3}$
dislocations	$10^8 \dots 5 \times 10^{11} \text{ cm}^{-2}$
precipitates (r=2 nm)	$10^{14} \dots 10^{17} \text{ cm}^{-3}$
grain boundaries	5 μm ... 200 nm (particle size)
microvoids (>50 atoms)	$10^{14} \dots 5 \times 10^{17} \text{ cm}^{-3}$



Theoretical Calculation of Vacancy Clusters in Si



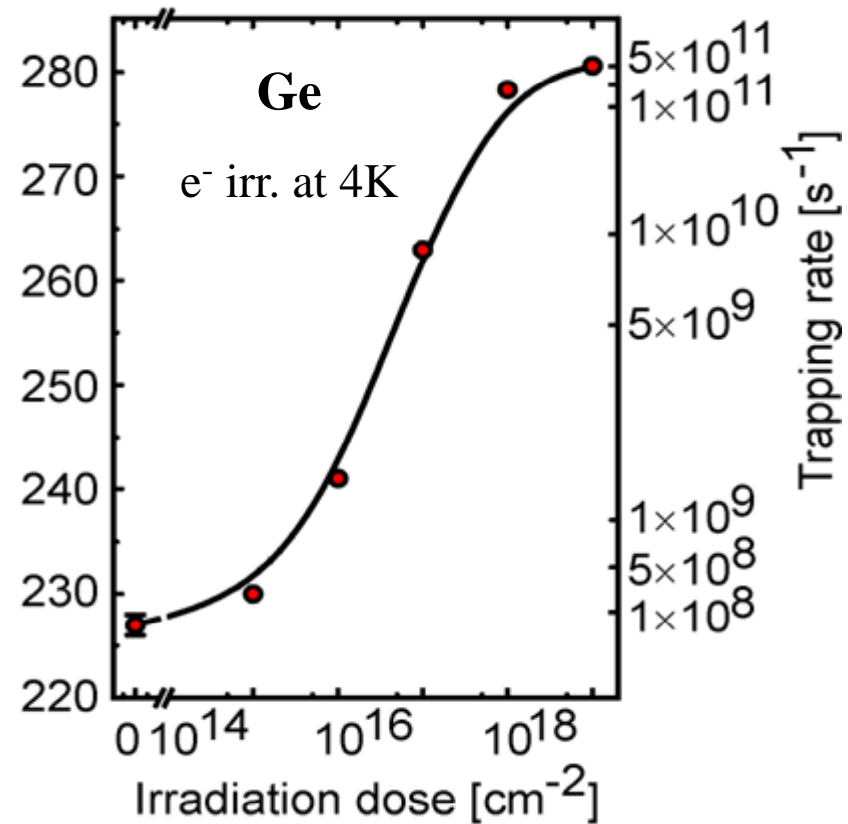
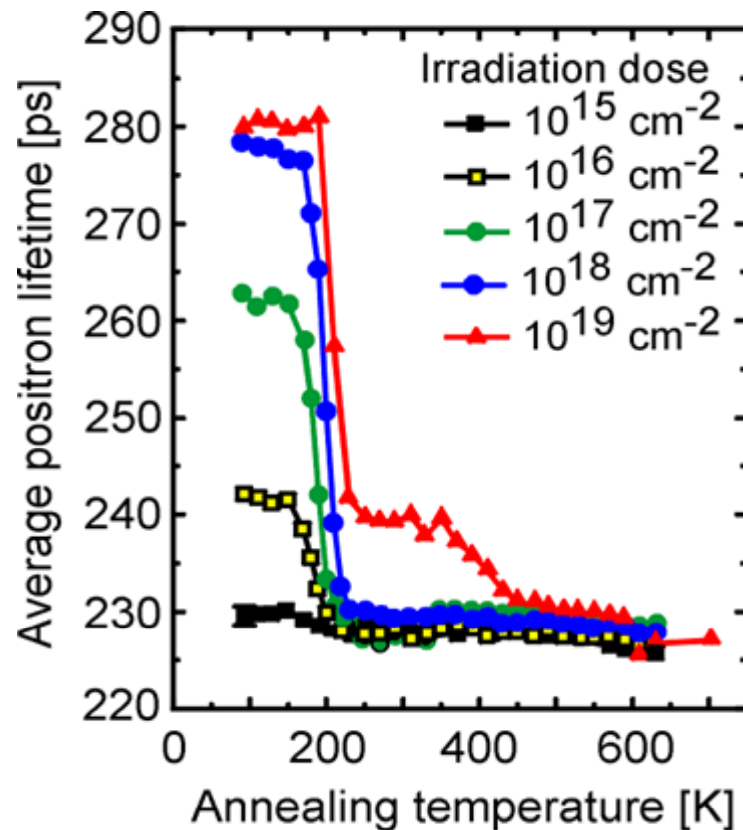
- there are cluster configurations with a large energy gain
- „Magic Numbers“ with 6, 10 und 14 vacancies
- positron lifetime increases distinctly with cluster size
- for $n > 10$ saturation effect, i.e. size cannot be determined

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 stable up to 400 K)

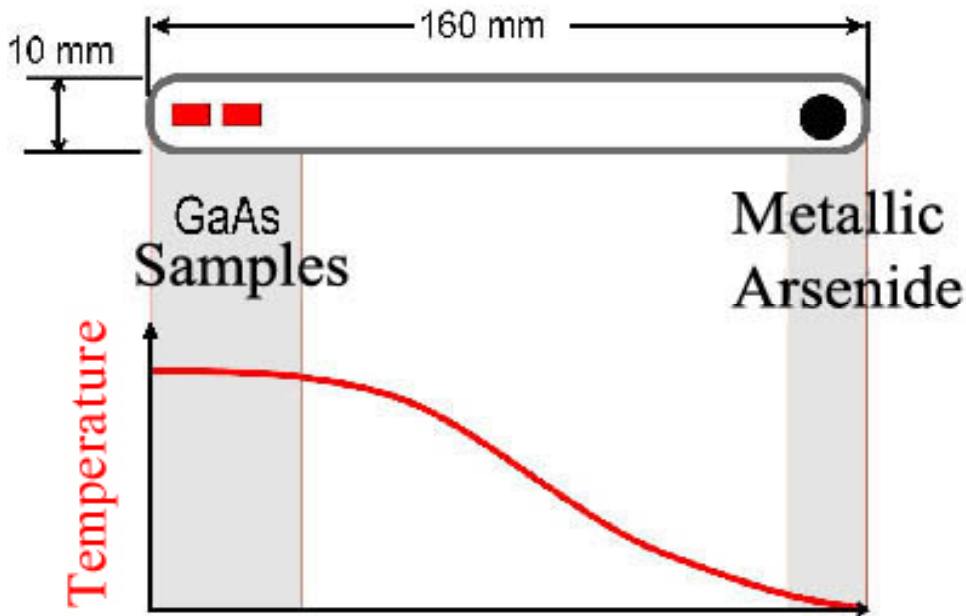


(Polity et al., 1997)



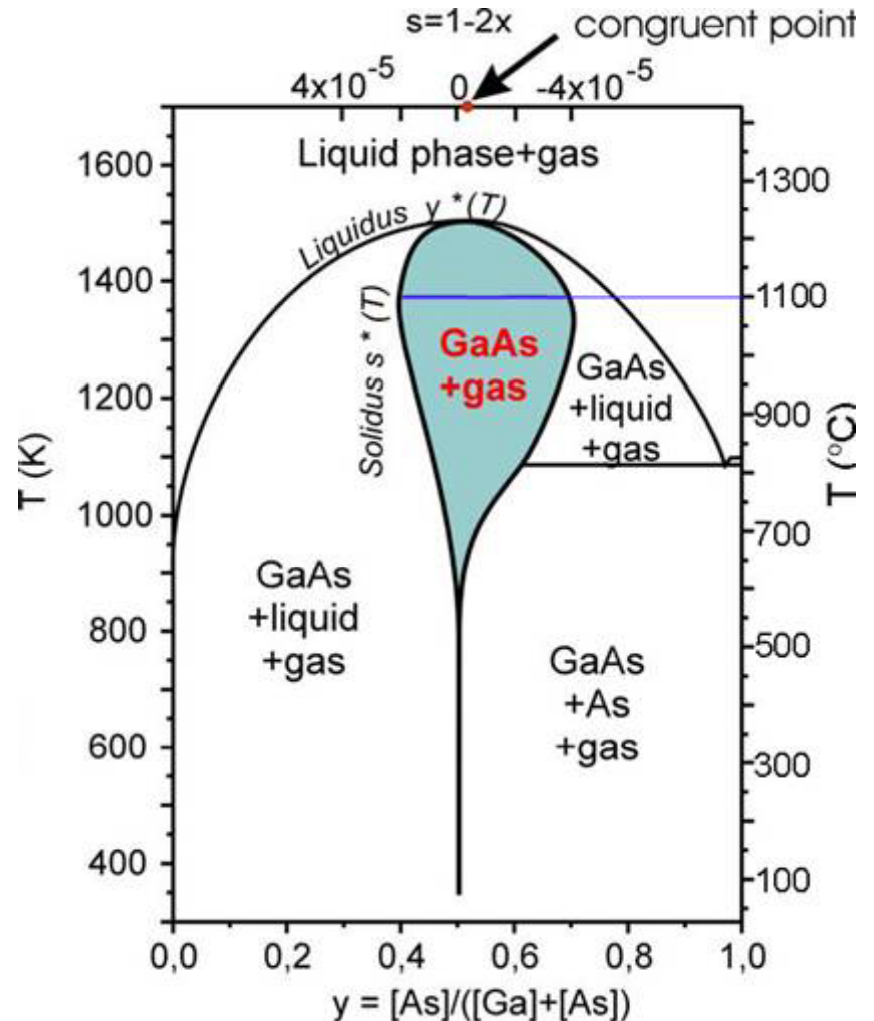
GaAs: annealing under defined As-partial pressure

- two-zone-furnace: Control of sample temperature **and** As partial pressure allows to navigate freely in phase diagram (existence area of compound)
- Measurements near equilibrium after quenching of samples



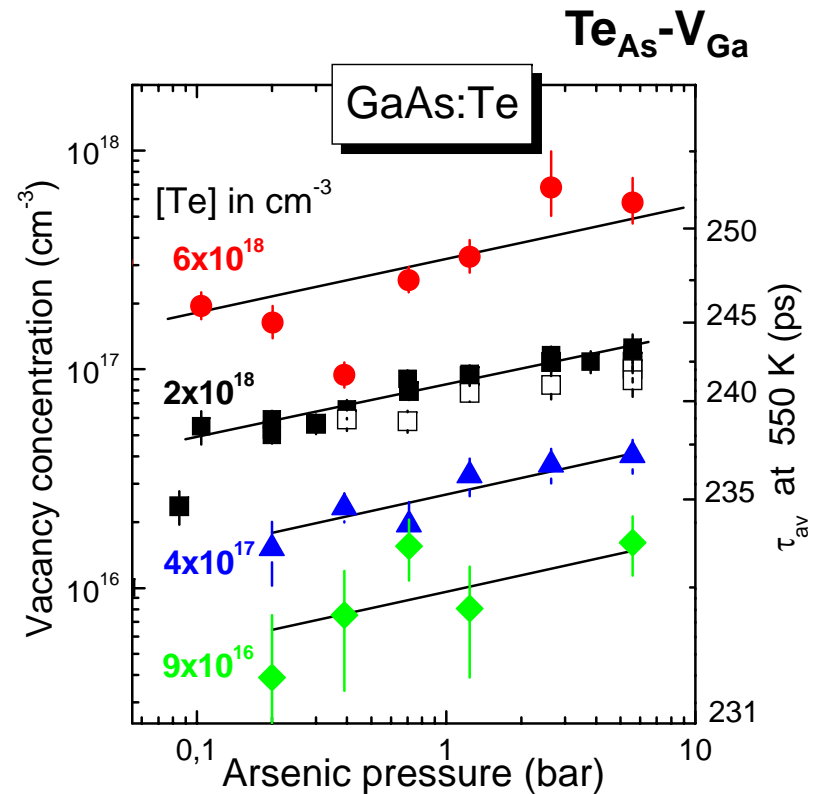
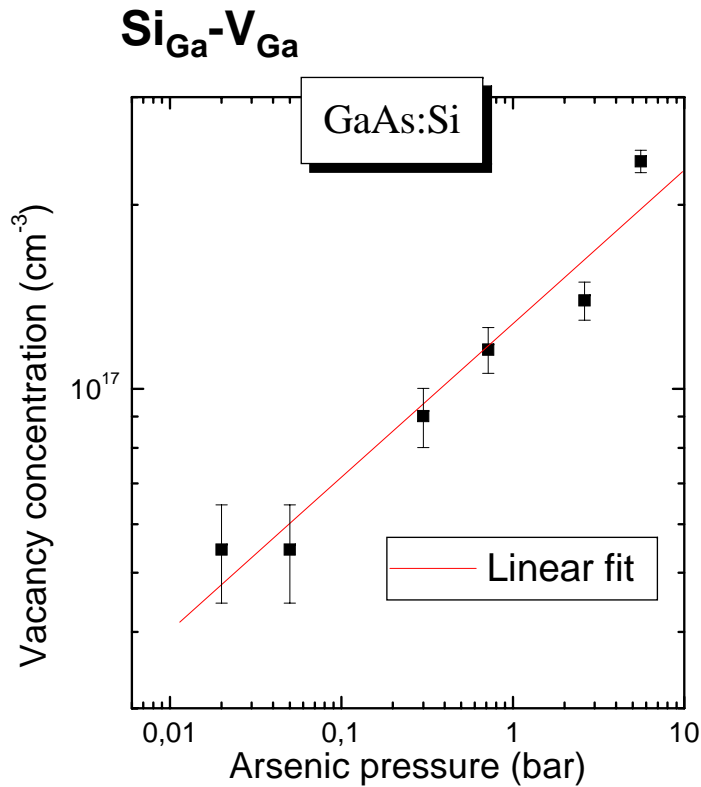
$T_{\text{sample}}: 1100^{\circ}\text{C}$

T_{As} : determines As-partial pressure



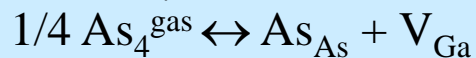
H. Wenzl et al., J. Cryst. Growth **109**, 191 (1991).

GaAs: Annealing under defined As pressure



*J. Gebauer et al.,
Physica B 273-274, 705 (1999)*

Thermodynamic reaction:



Mass action law:

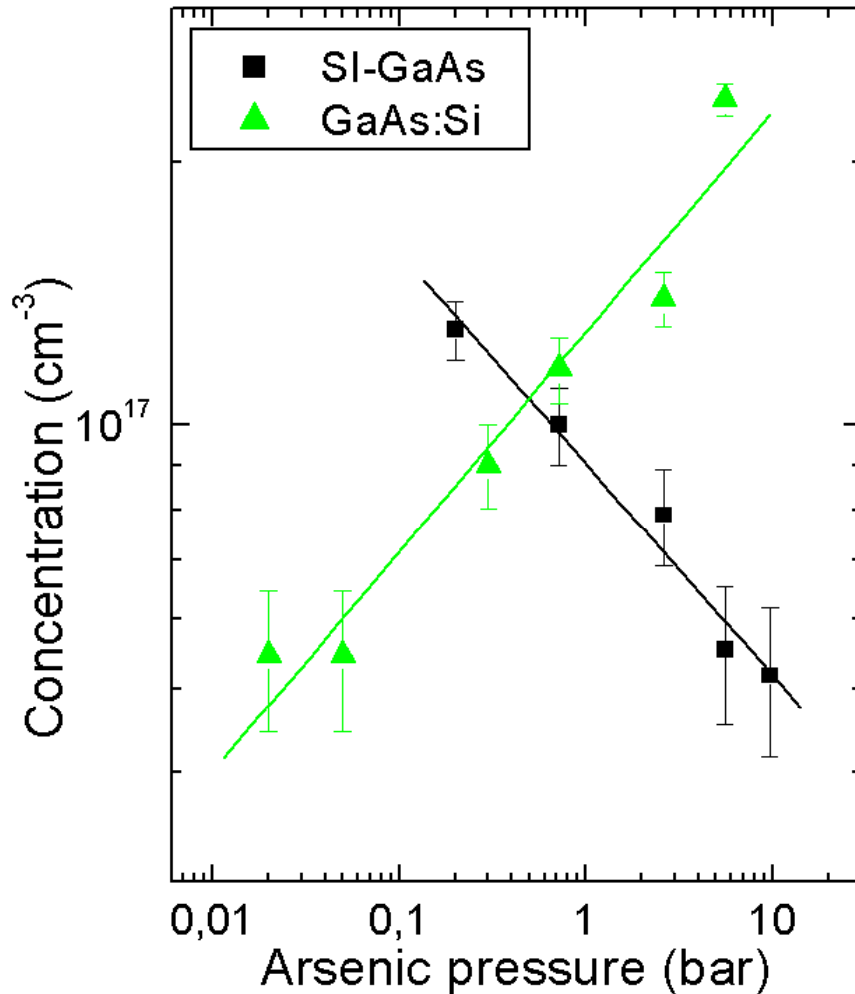
$$[\text{V}_{\text{Ga}}] = K_{\text{VG}} \times p_{\text{As}}^{1/4}$$

Fit: $[\text{V}_{\text{Ga}}\text{-Dopant}] \sim p_{\text{As}}^n$

$\rightarrow n = 1/4$



Comparison of doped and undoped GaAs



Bondarenko et al., 2003

Thermodynamic reaction:



Mass action law:

$$[\text{V}_{\text{As}}] = K_{\text{VAs}} \times p_{\text{As}}^{-1/4}$$

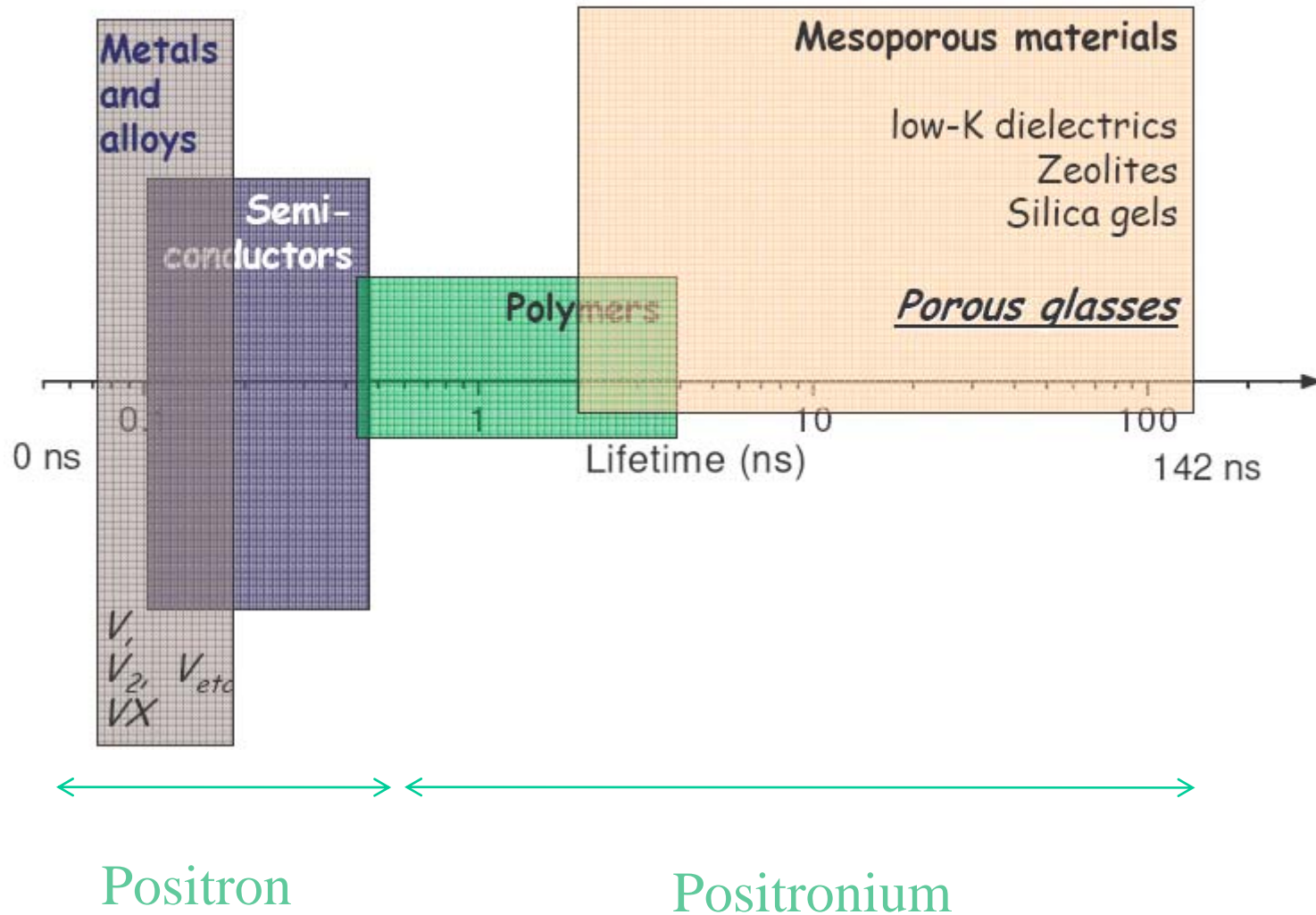
Fit: $[\text{V-complex}] \sim p_{\text{As}}^n$

→ $n = -1/4$

undoped GaAs: As vacancy



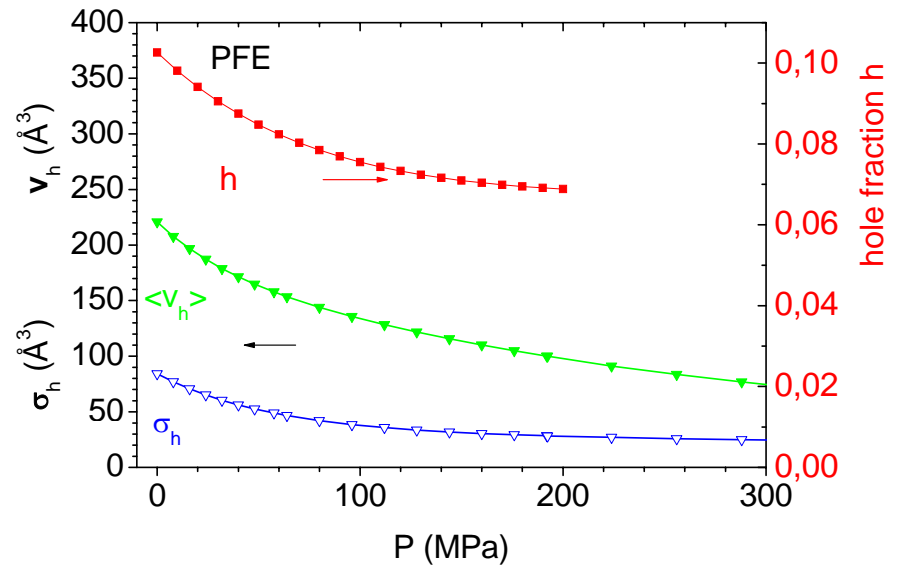
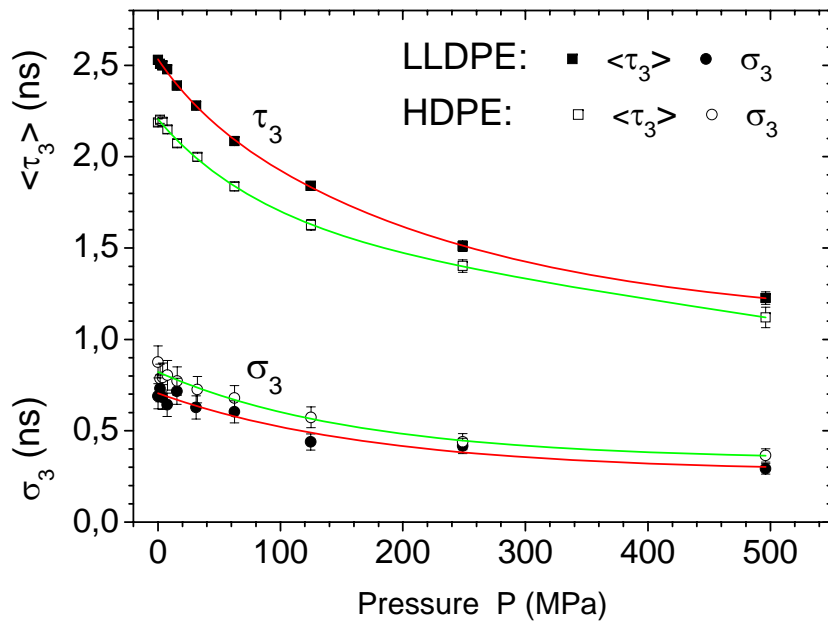
Typical Lifetimes



Polymers under Pressure

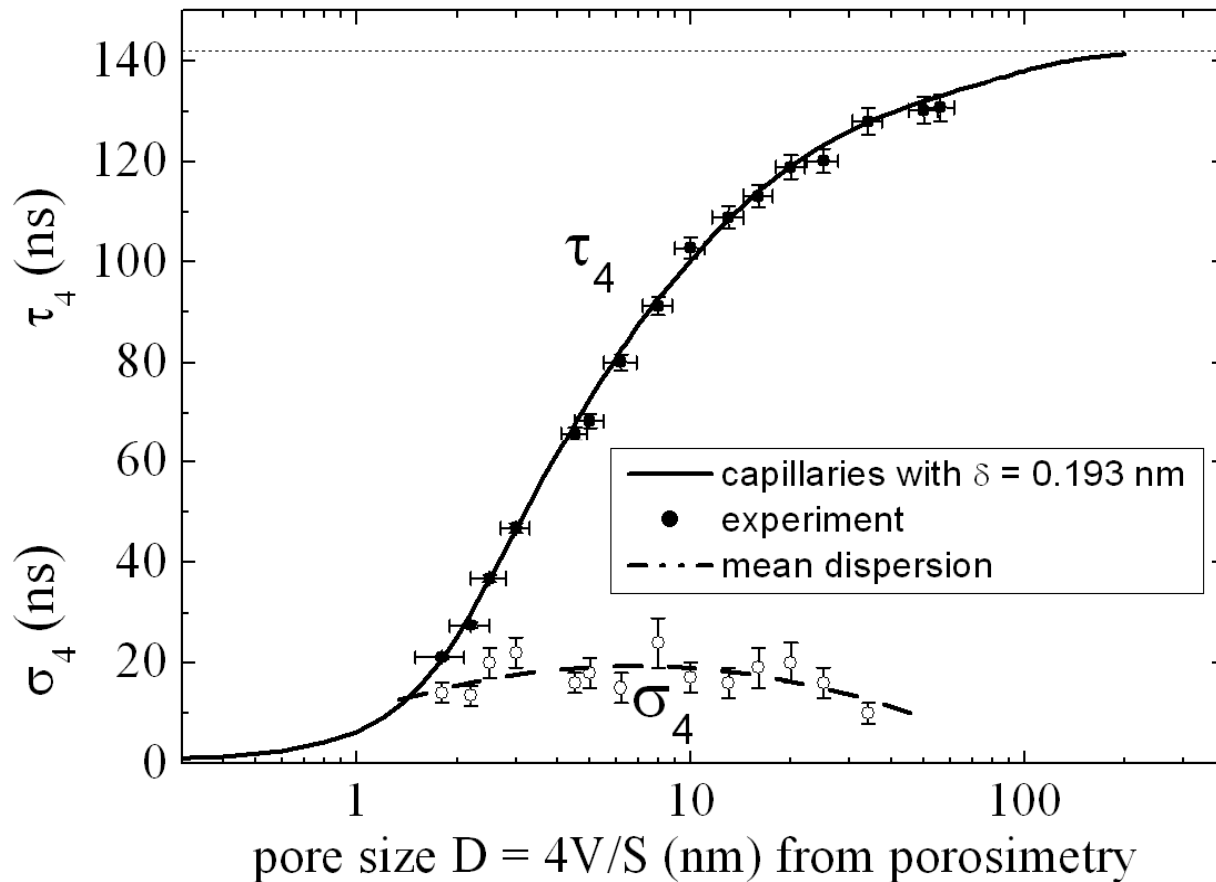
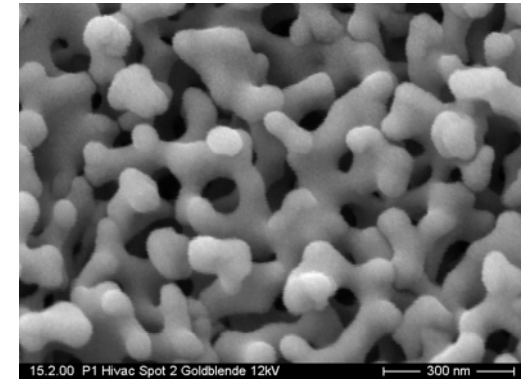
- The open volume V_h between polymer molecular chains can be determined from the ortho-positronium lifetime τ_3
- Also its size dispersion σ_h and the hole fraction h

LLDPE: low-density polyethylene HDPE: high-density polyethylene PFE: perfluorelastomer



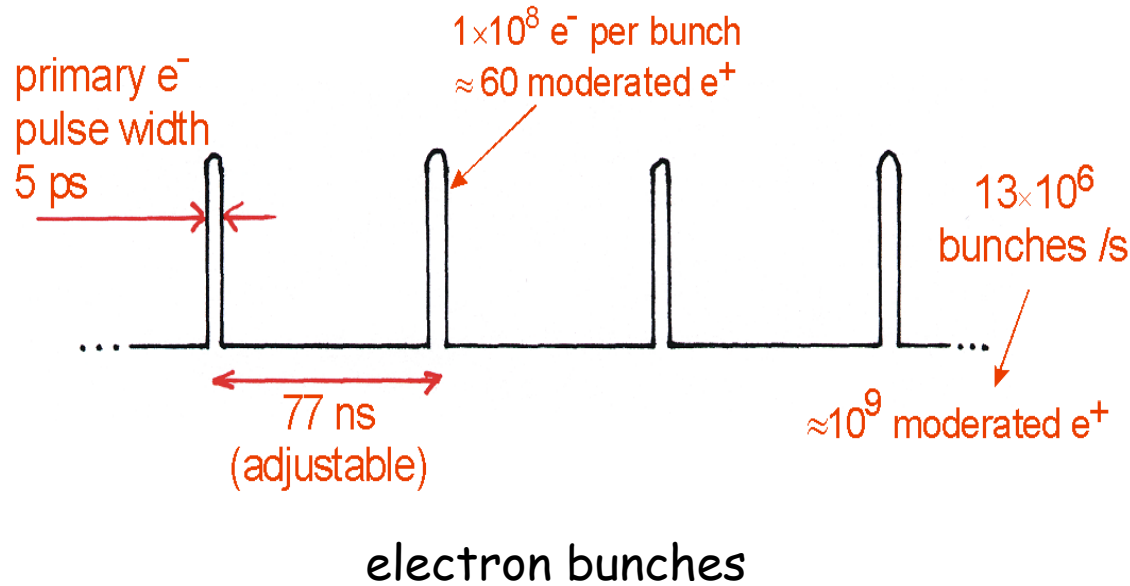
Porosimetry by Positronium

- Controlled pore glass: pore diameter 1...100 nm
- Best sensitivity of positrons: 1...10 nm
- Pore size distribution can be determined



The EPOS positron source at Research Center Dresden-Rossendorf

- Radiation source ELBE = **E**lectron **L**inac with high **B**rilliance and low **E**mittance
- Primary electron beam (40 MeV x 1 mA = 40 kW)
- Main goal: Infrared Free-electron Lasers
- Very interesting time structure: cw-mode of short bunches with 13 MHz

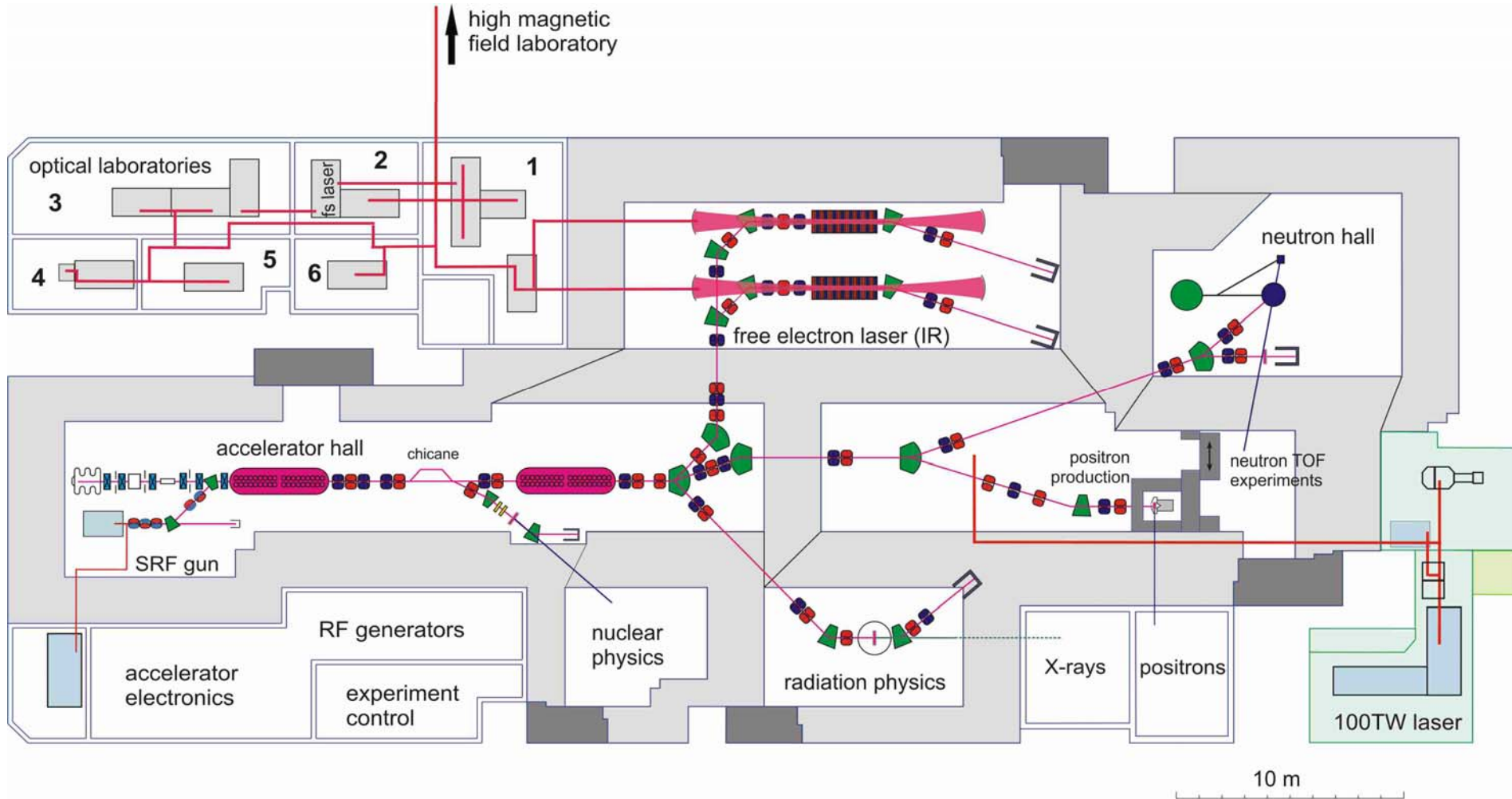


EPOS = ELBE Positron Source

- Intense, pulsed beam of slow (monoenergetic) positrons
- All relevant positron techniques for materials research (positron lifetime, Coincidence Doppler broadening, AMOC)
- EPOS is external facility of Martin-Luther-University Halle in collaboration with Research Center Dresden-Rossendorf (FZD)
- User-dedicated facility
- Remote controlled via internet
- Financing by University Halle, Land Sachsen-Anhalt, European Community, and FZD



ELBE Layout



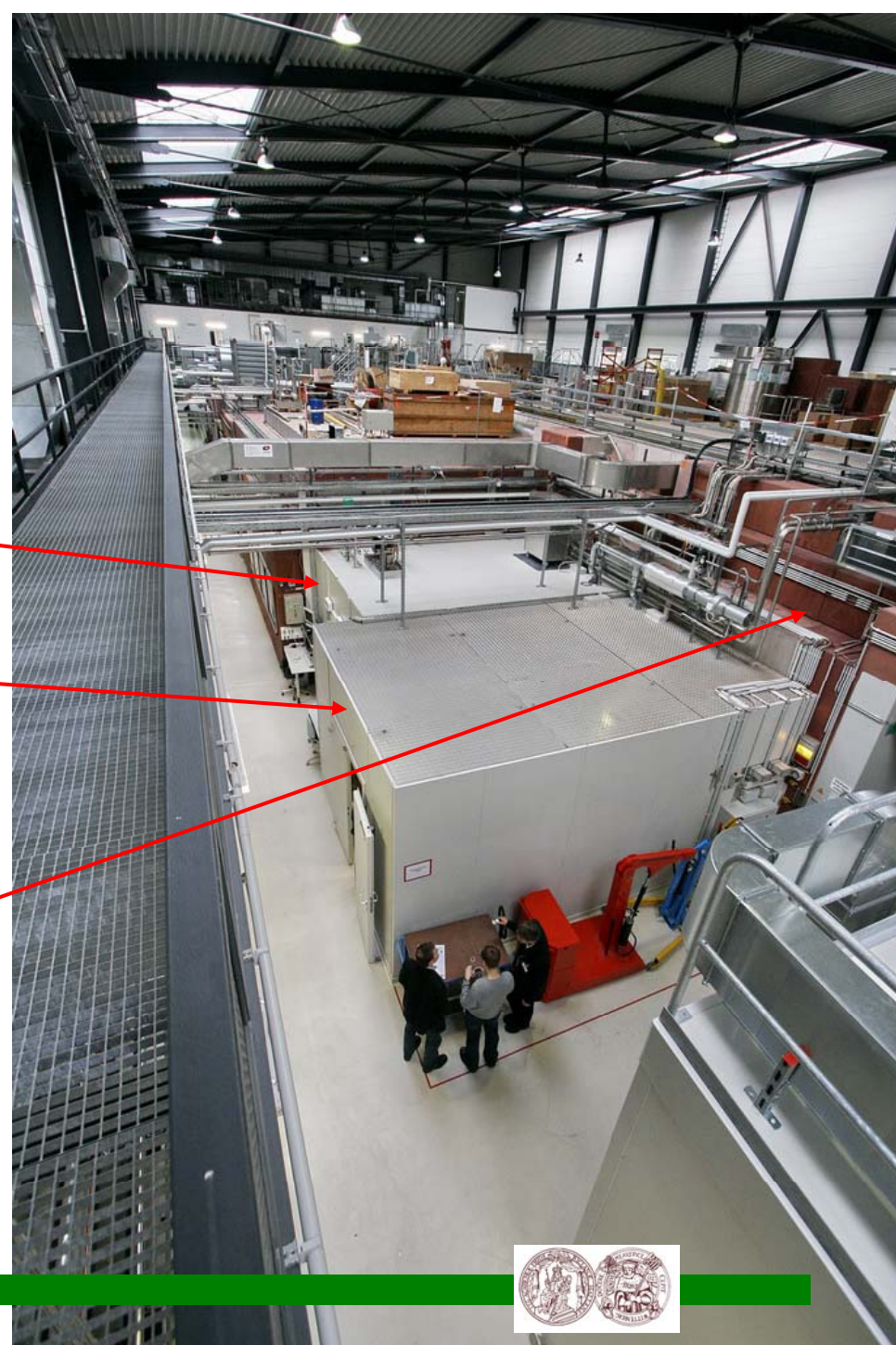
Positron Lab

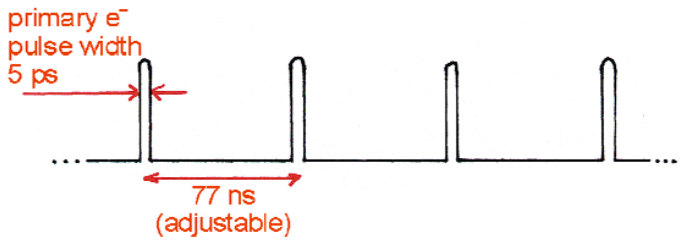
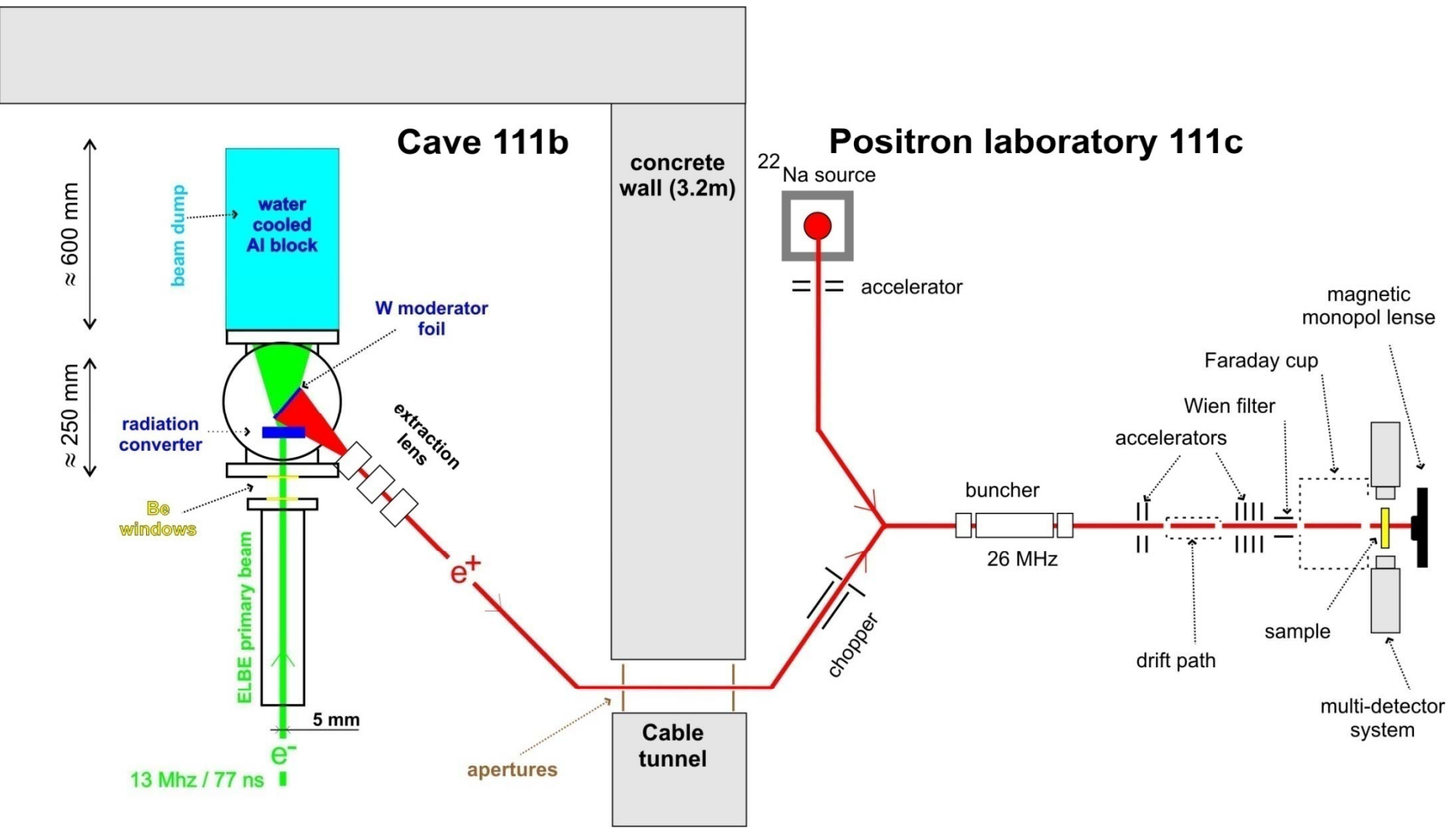
- positron lab in ELBE hall

X-ray Lab

Positron Lab

concrete screening of Cave 111b
(location of e^+ converter)



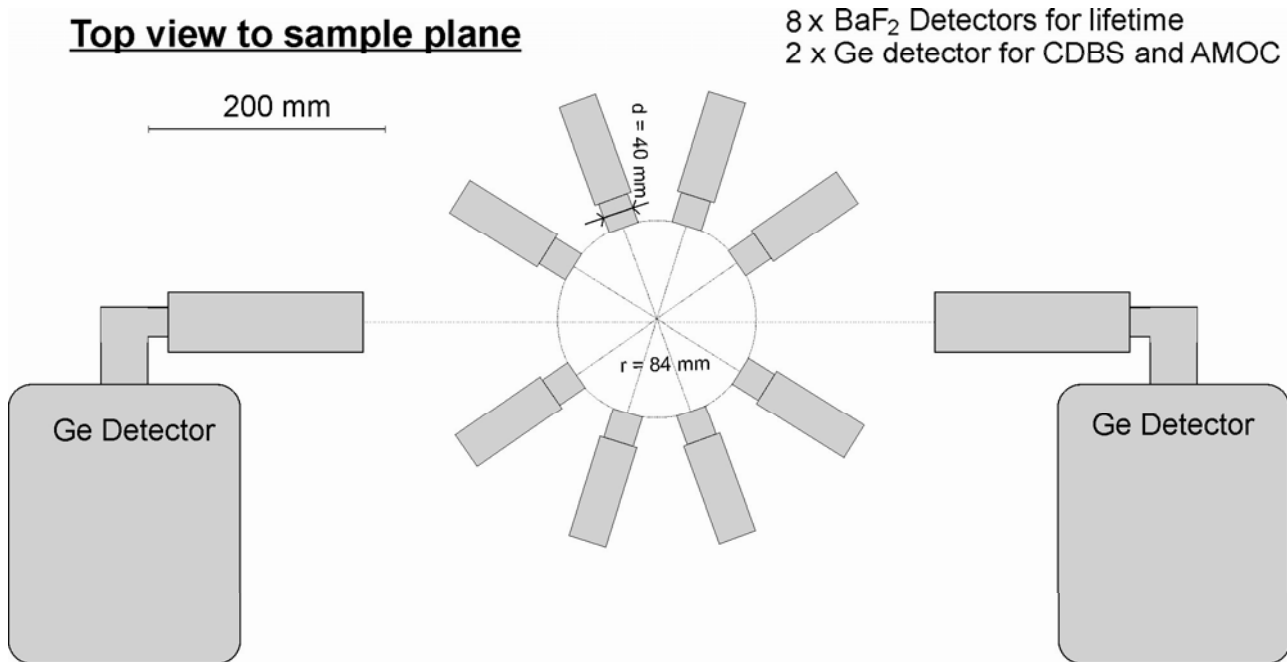


EPOS scheme



Detector system

- **3 experiments:** lifetime spectroscopy (8 BaF₂ detectors); Doppler coincidence (2 Ge detectors), and AMOC (1 Ge and 1 BaF₂ detector)
- **digital detection system:**
 - lifetime: almost nothing to adjust; time scale exactly the same for all detectors; easy realization of coincidence
 - Doppler: better energy resolution and pile-up rejection expected
 - pulse-shape discrimination improves spectra quality



First Projects of EPOS

- Irradiation-hard Steel of Fission Reactors (FZD)
- Defects in ZnO (FZD & Uni Leipzig; IKZ Berlin)
- Low-k dielectrics for microelectronics (AMD Saxony)
- Smart cut of wide band-gap semiconductors - GaN, ZnO, AlN (MPI Halle)
- Rp/2 effect in Si (FZD)
- Abnormal diffusion of Zn and Cu in III-V-compound semiconductors (Uni Halle)
- Pore size distribution on micro- and mesopores (Uni Halle)
- Defects in "dirty" Si for photovoltaic (Fraunhofer Inst. Halle)
- Thin polymer layers on top of metallic substrate (Uni Kiel)
- Hydrogen in defects in metals (Uni Prague, FZD)
- Defects after tribological treatment of surfaces (Academy Inst. Krakow)
- ...



View flange

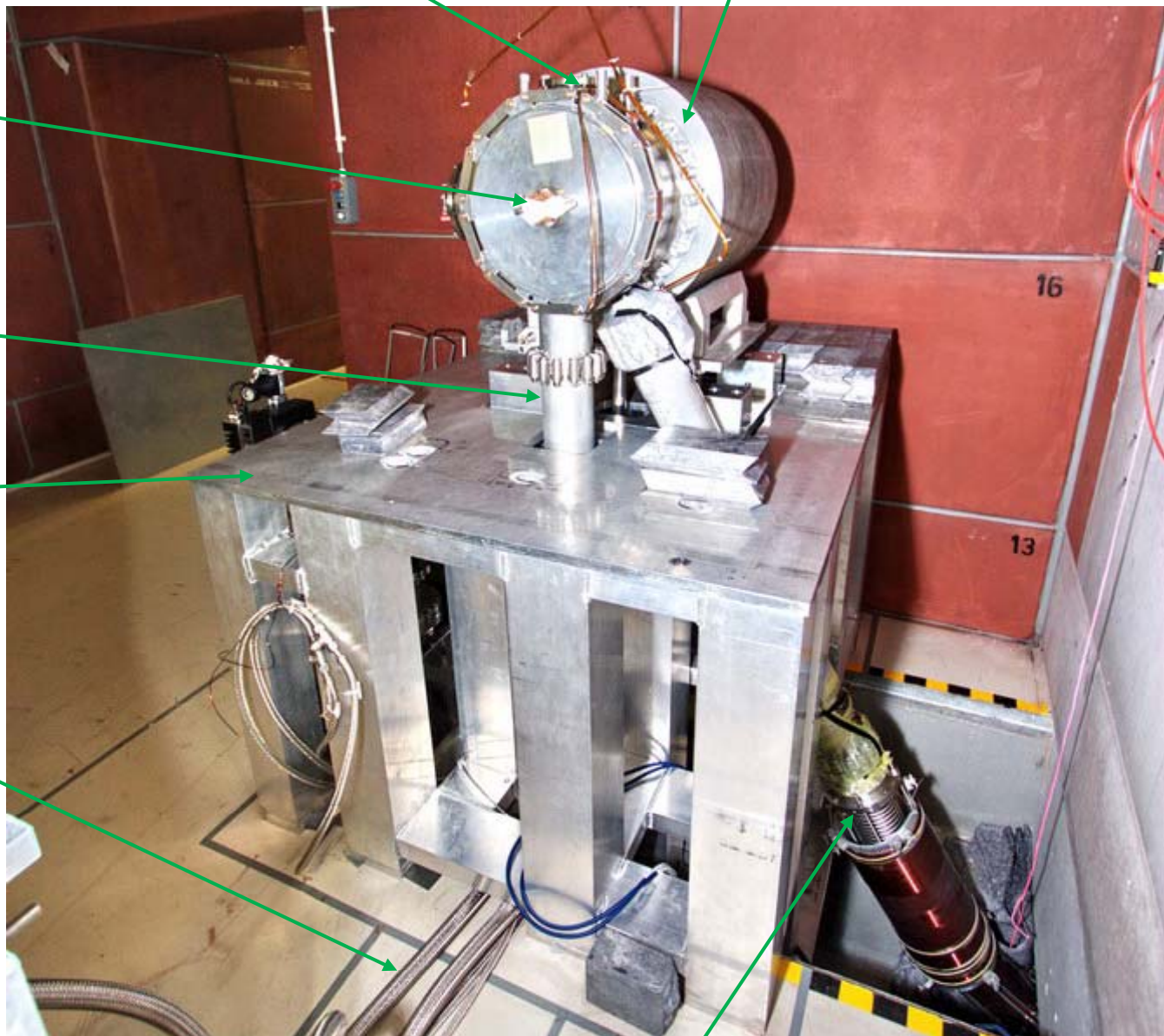
Beam dump

Be entrance window

to vacuum system

Pb stand

Water cooling pipes

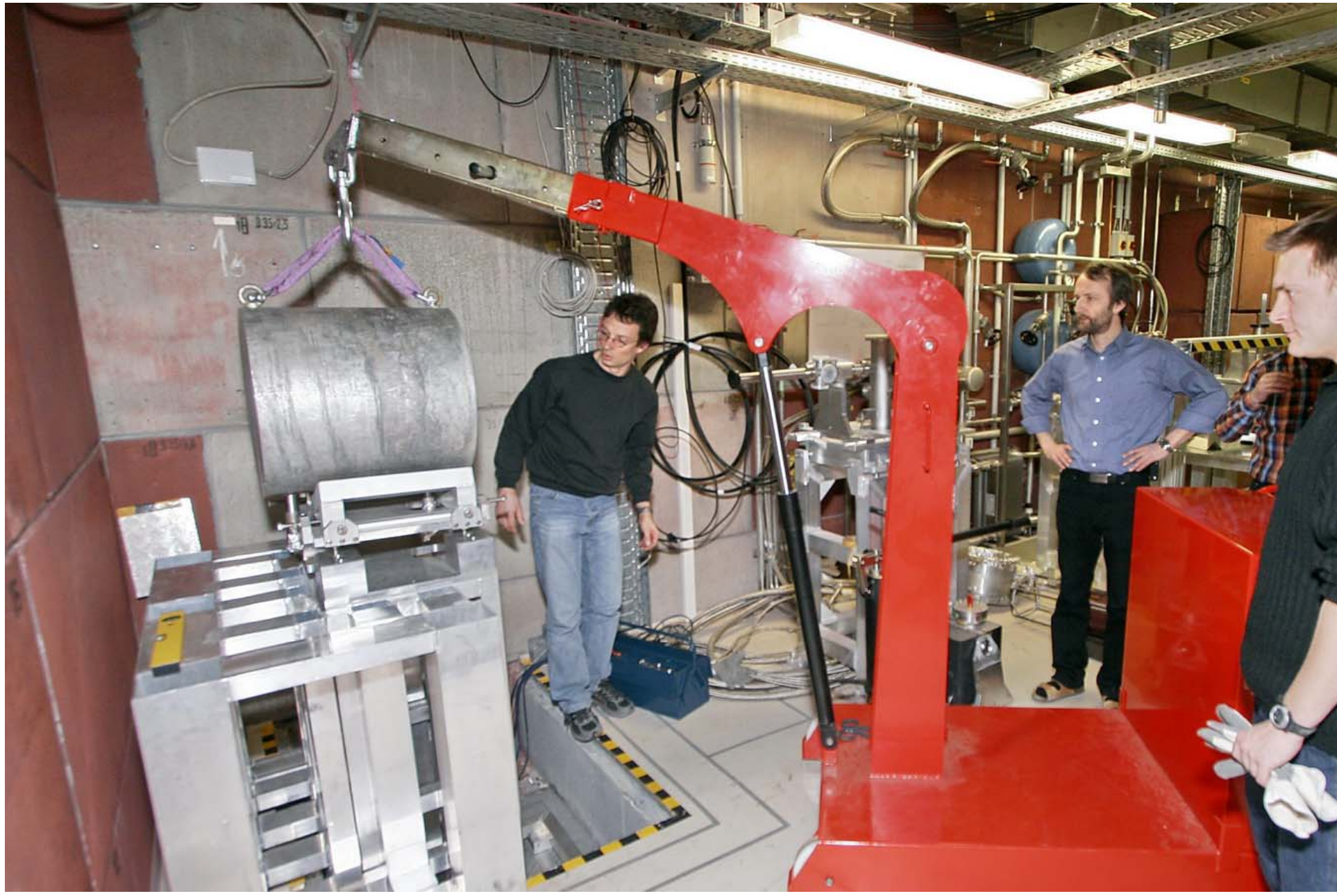


Beam extraction to Laboratory

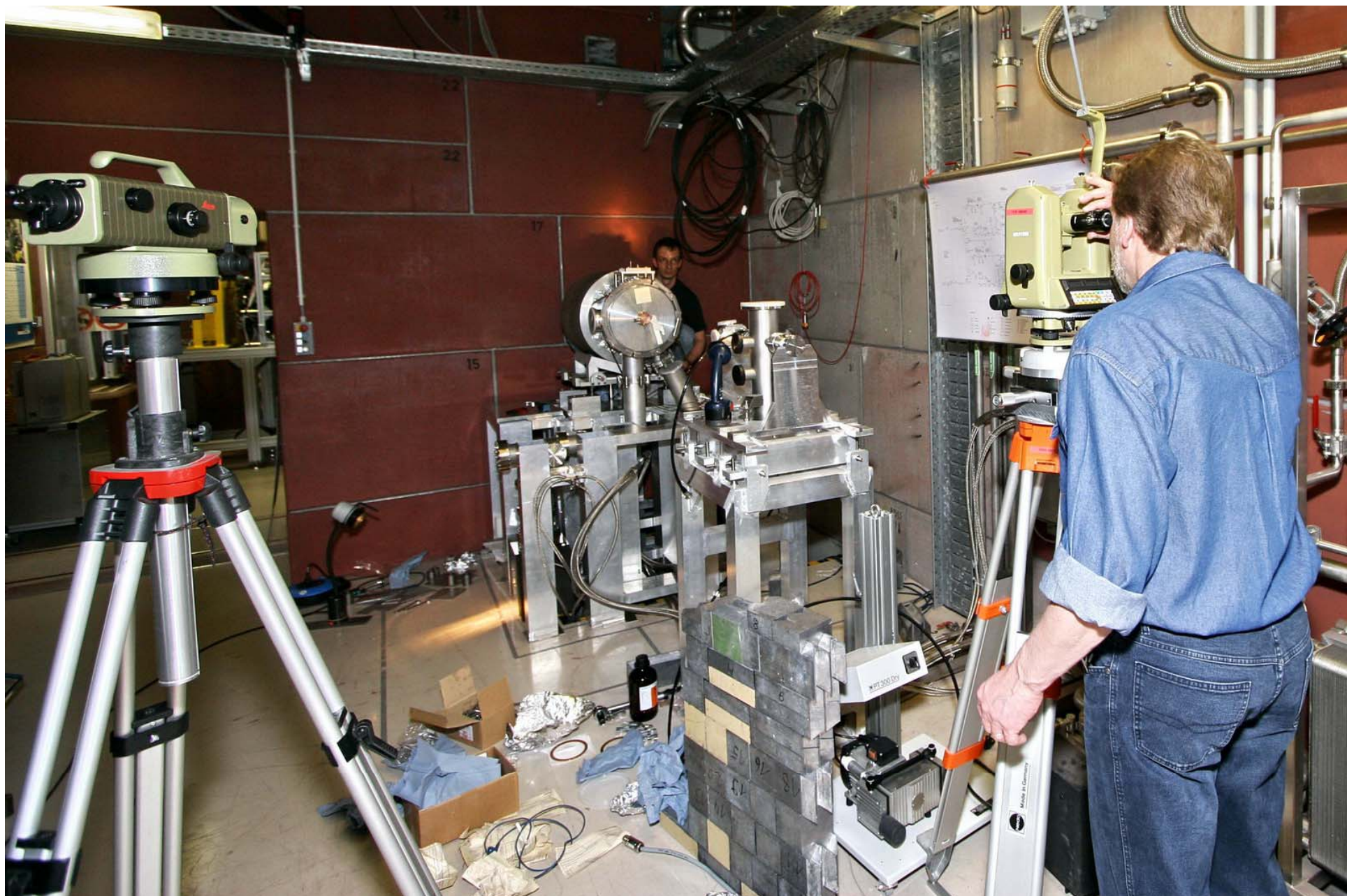
Beam line through cable channel under 3.2 m concrete



Electron beam dump is put onto the stand



Adjustment of Be windows: deviation 0.17mm (y-direction) and 0.10 mm (x-direction)



Pb screening almost complete



Heavy concrete radiation screening started

