

Die Positronenannihilation in der Materialforschung

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- Geschichtliches
- Techniken der Positronenannihilation
- Anwendungen
 - Kristalldefekte
 - weiche Materie
 - Porosimetrie
- Positronenmessplätze in Deutschland



Martin-Luther-Universität
Halle-Wittenberg

der Bundeswehr
Universität  München



Discovery of the Positron

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

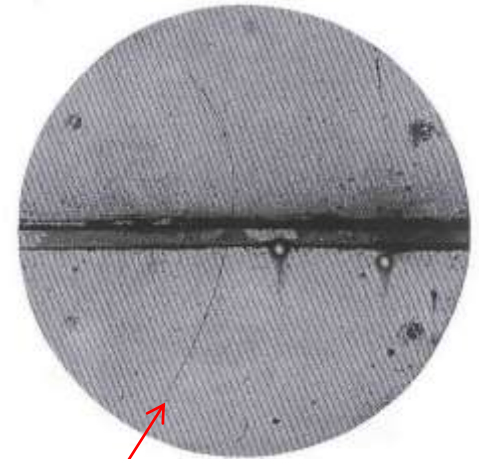


C.D. Anderson

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



P.A.M. Dirac



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



Electron structure of solids can be discovered

- during annihilation: conservation laws must be fulfilled (energy, momentum)
- positron cools down to thermal energies ->
- energy of annihilating electron-positron pair = energy of electron
- electron momentum distribution can directly be measured

MARCH 1 AND 15, 1942

PHYSICAL REVIEW

VOLUME 61

The Angular Distribution of Positron Annihilation Radiation

ROBERT BERINGER* AND C. G. MONTGOMERY

Sloane Physics Laboratory, Yale University, New Haven, Connecticut

(Received January 7, 1942)

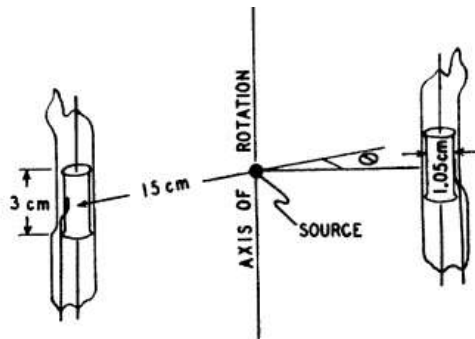
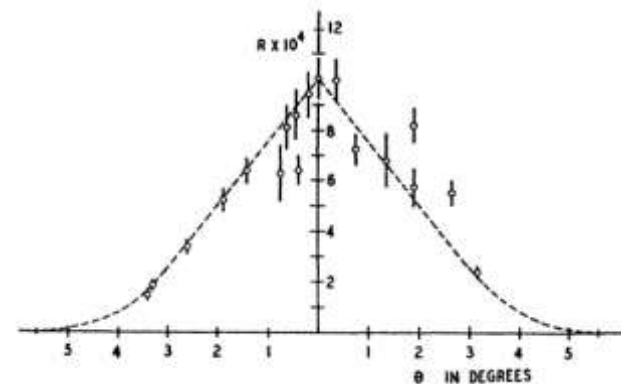
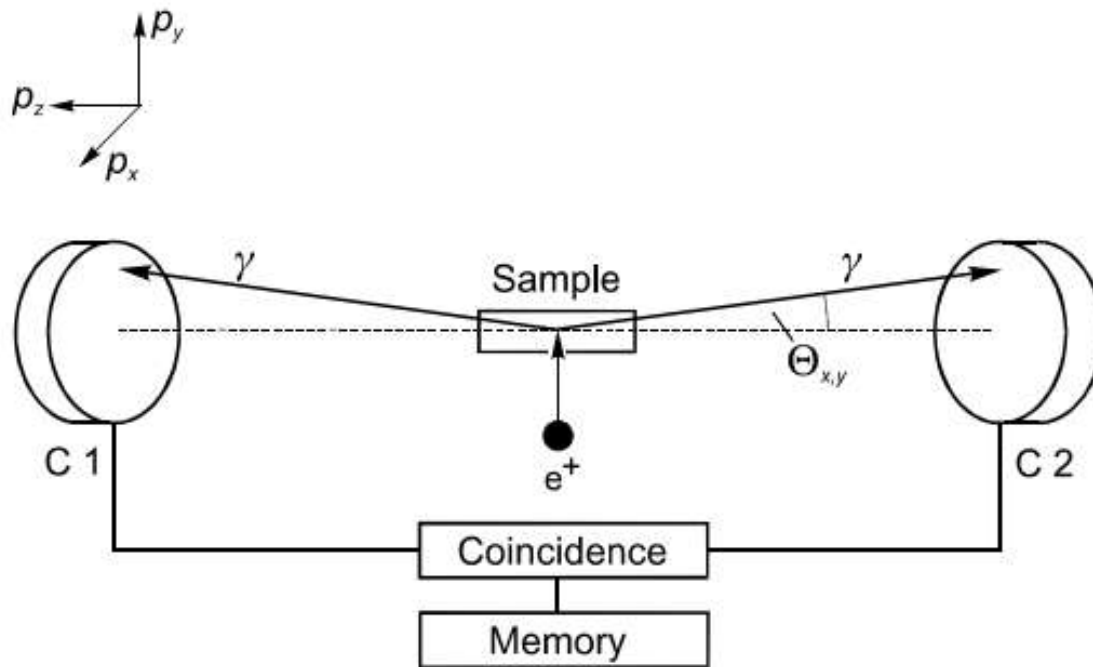


FIG. 1. Schematic arrangement of counters for observing coincidences from annihilation radiation.



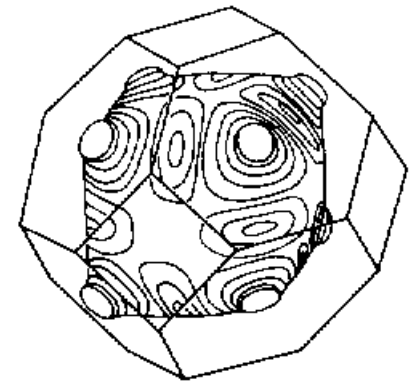
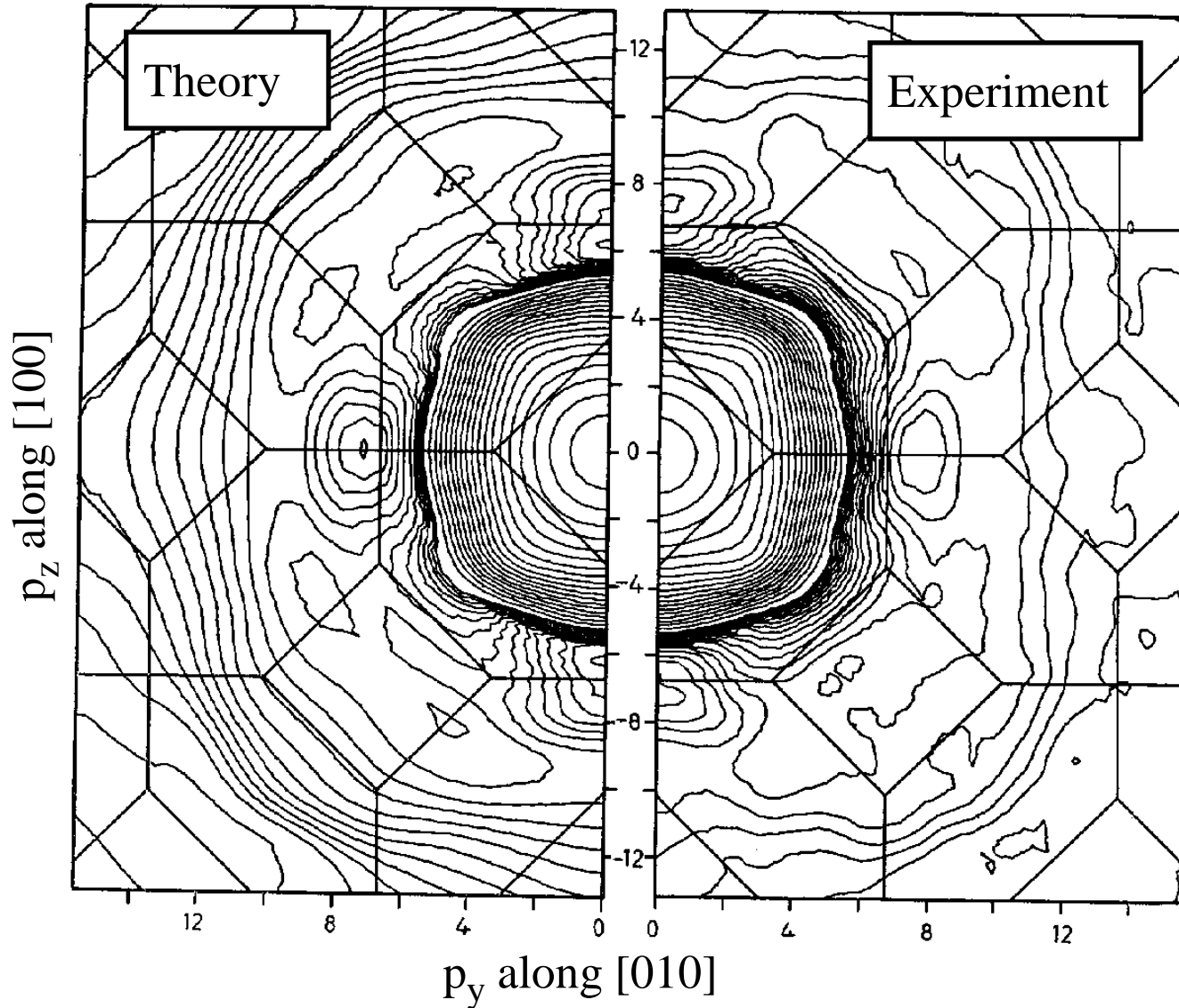
2D – ACAR (Angular Correlation of Annihilation Radiation)

- now: two-dimensional (position-sensitive) detectors
- measurement of single crystals in different directions:
- reconstruction of Fermi surface possible



$$N_c(\Theta_x, \Theta_y) = A_c \int_{-\infty}^{\infty} \sigma(\Theta_x m_0 c, \Theta_y m_0 c, p_z) dp_z$$

2D-ACAR of Copper



Fermi surface
of copper

(Berko, 1979)



Positrons are sensitive for Crystal Lattice Defects

- 1950...1960: in addition to ACAR -> 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

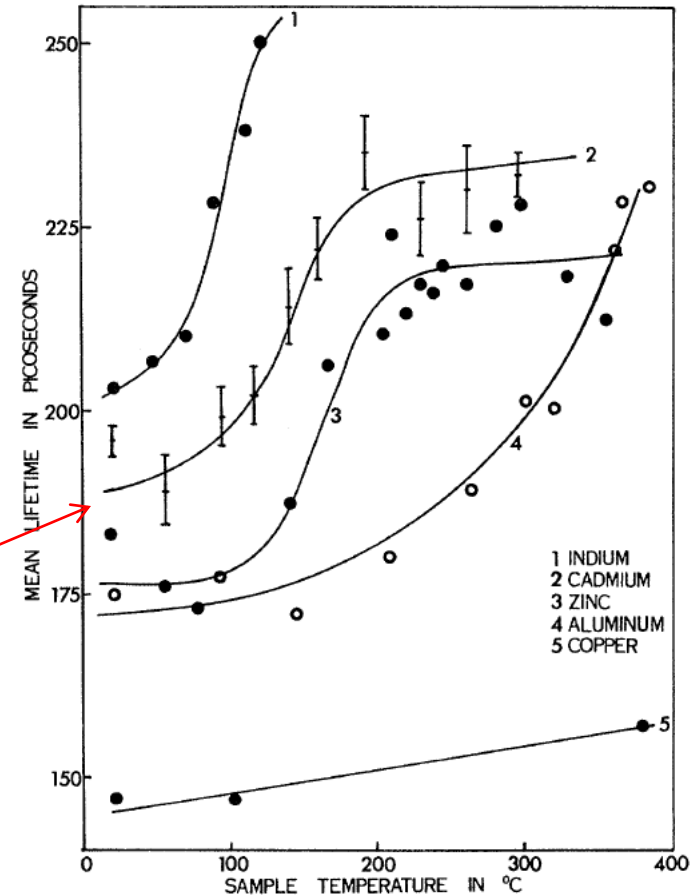


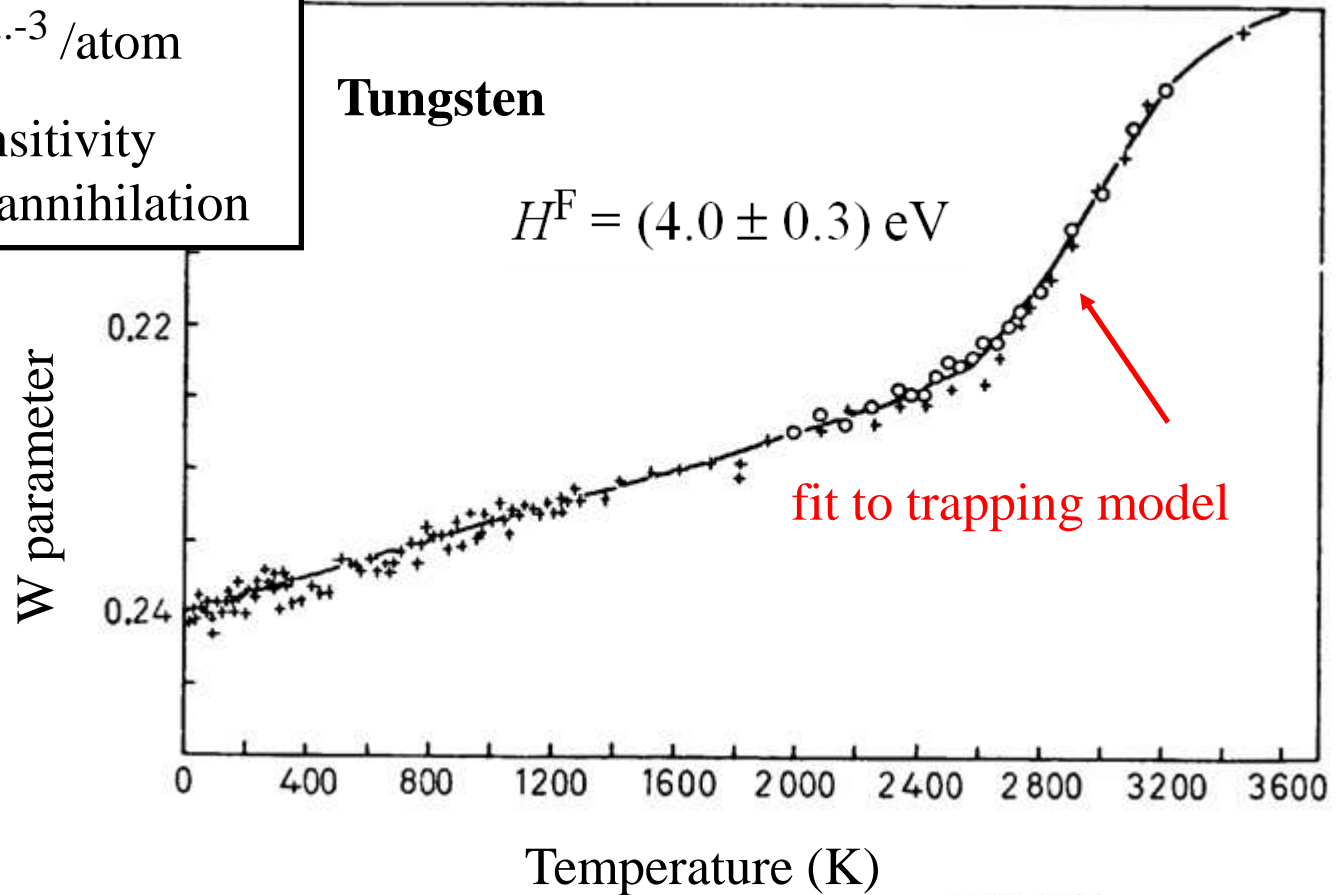
FIG. 1. Positron mean lifetimes in several metals as a function of temperature.



Vacancies in thermal Equilibrium

- Vacancy concentration in thermal equilibrium:
- in metals $H^F \approx 1...4$ eV \Rightarrow at T_m $[1v] \approx 10^{-4...-3}$ /atom
- fits well to the sensitivity range of positron annihilation

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



(Ziegler, 1979)



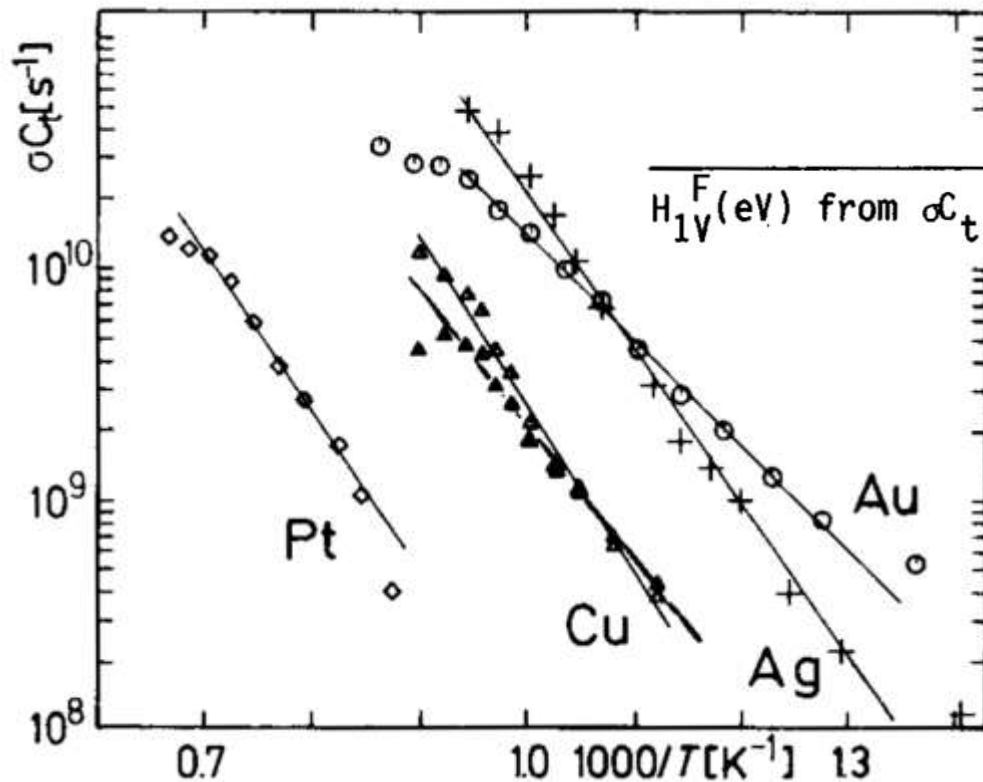
Determination of Vacancy Formation Enthalpy

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

H. E. Schaefer¹, W. Stuck¹, F. Banhart², and W. Bauer

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²Max-Planck-Institut für Metallforschung, Institut für Physik, Heisenbergstr. 1, D-7000 Stuttgart 80, Fed. Rep. of Germany



H_{1V}^F (eV) from σ_{C_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

VOLUME 25, NUMBER 2

15 JANUARY 1982

Vacancies and carbon impurities in α -iron: Electron irradiation

A. Vehanen, P. Hautojärvi, J. Johansson, and J. Yli-Kauppila

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P. Moser

Section de Physique du Solide, Département de Recherche Fondamentale,
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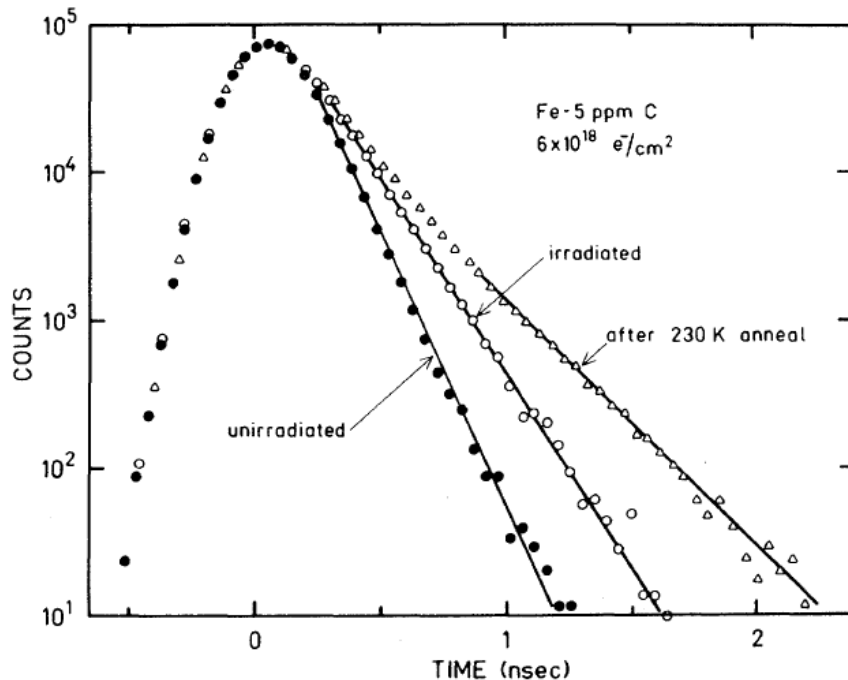


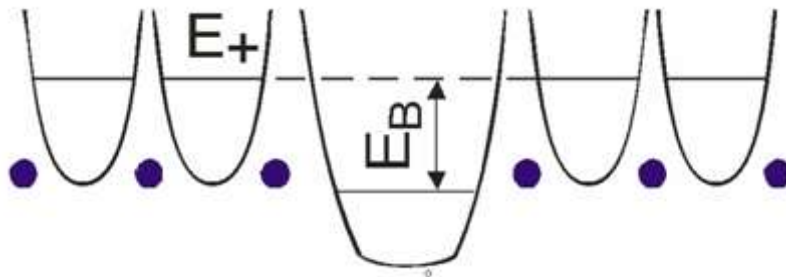
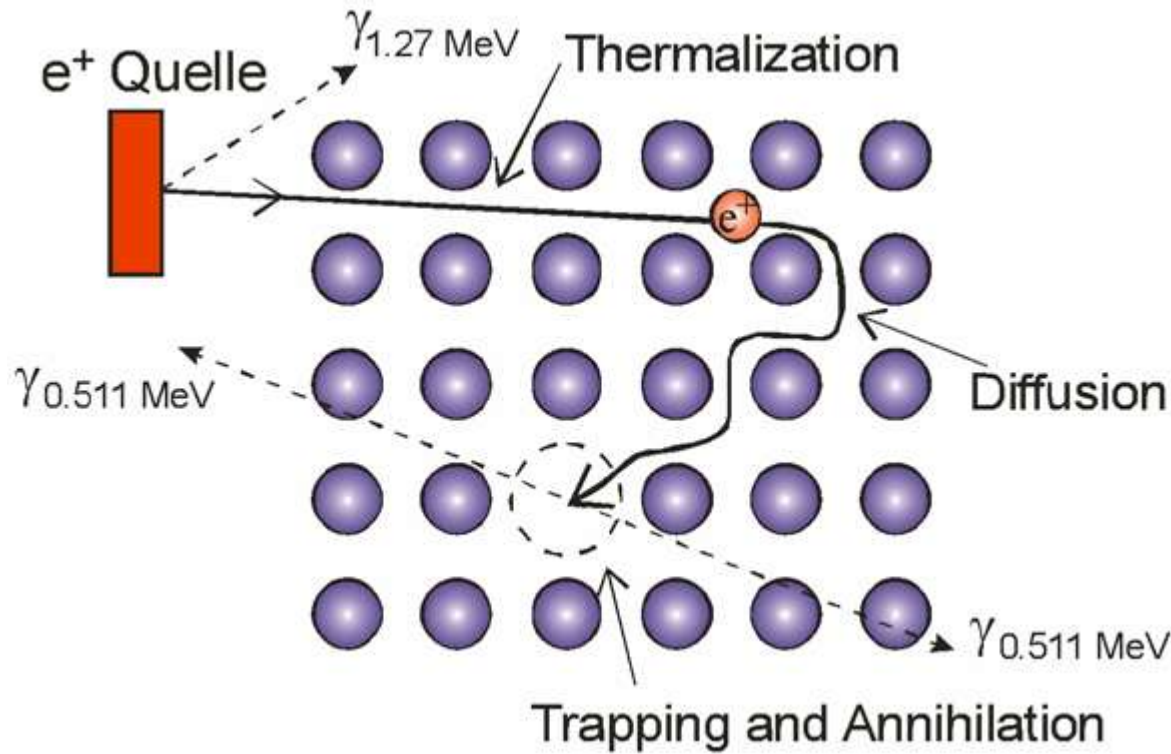
FIG. 1. Positron-lifetime spectra after source-background subtraction in electron-irradiated ($6 \times 10^{18} e^-/cm^2$) high-purity iron at various stages of isochronal annealing. The dramatic occurrence of a long-lifetime component after 230 K annealing is clearly visible.

- 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

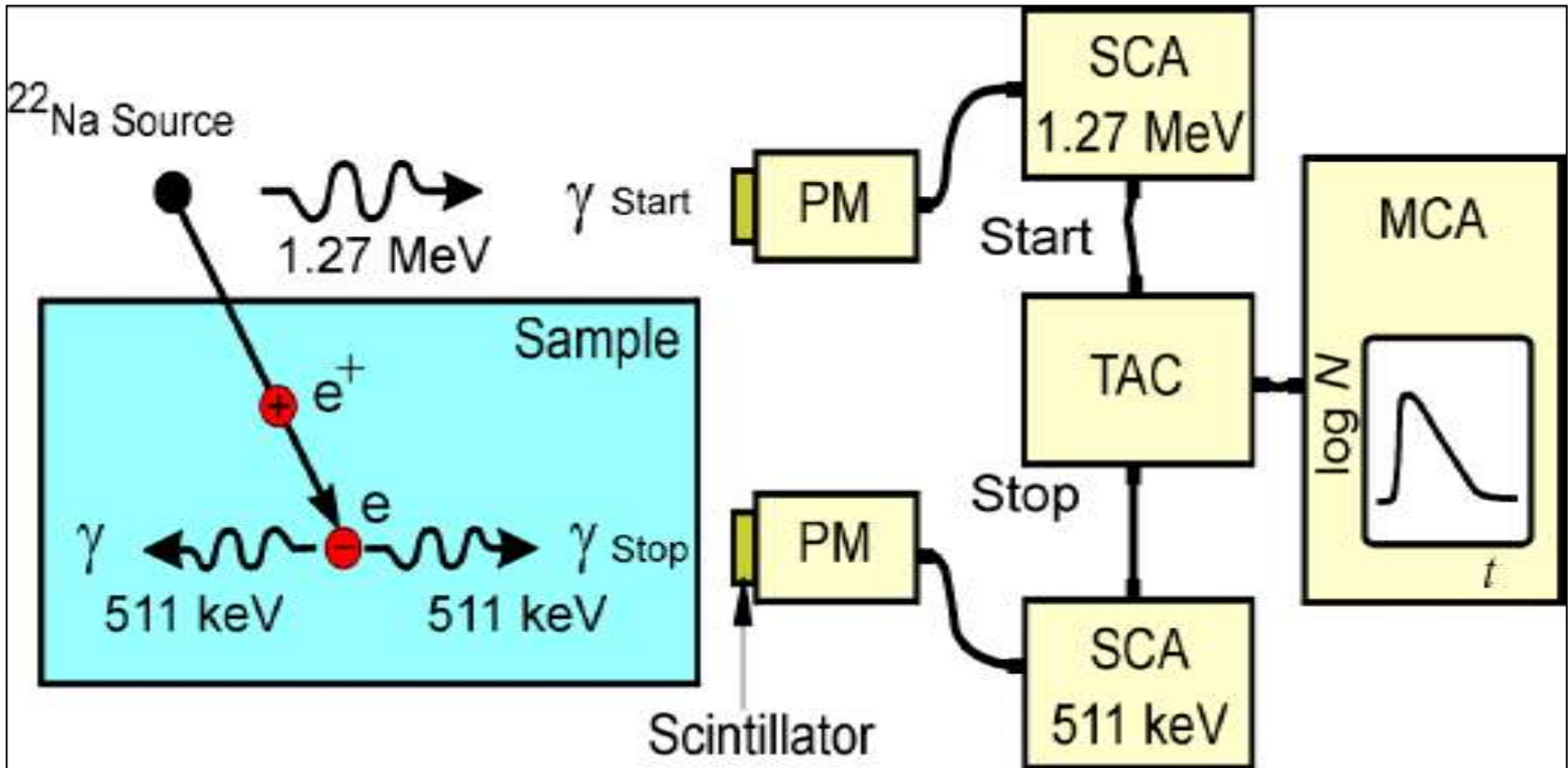
^{22}Na



- 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

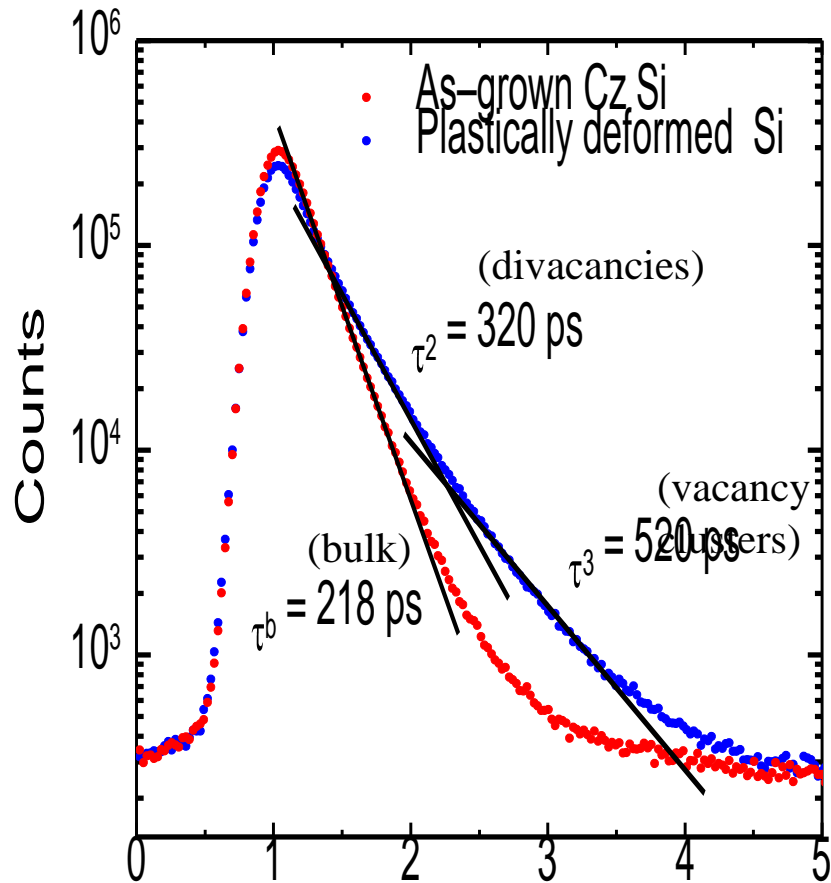


Positron lifetime spectroscopy



Positron lifetime: time between 1.27 MeV and 0.511 MeV quanta

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

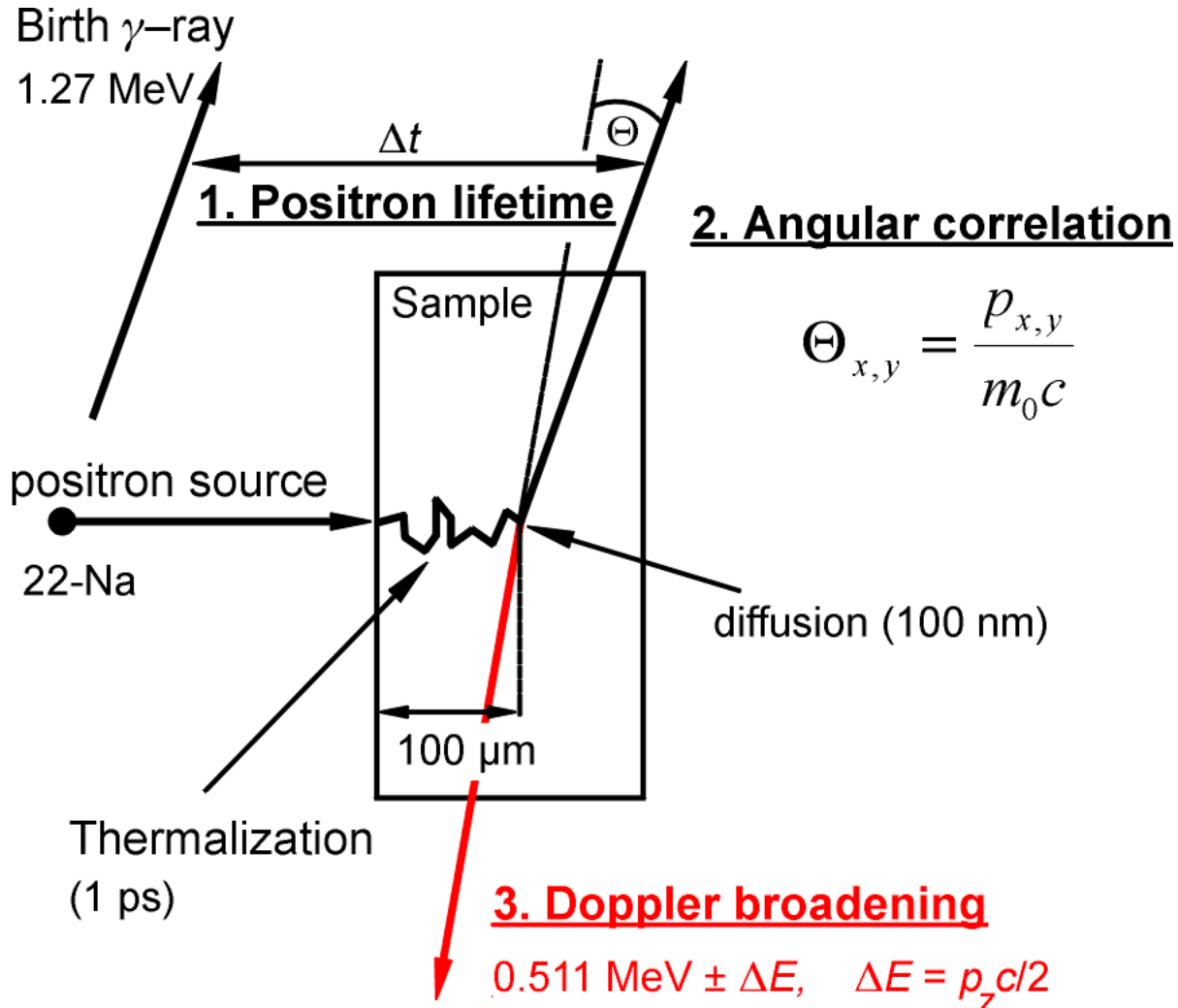
$$\kappa_d = \mu C_d = \frac{I_2}{I_1} \left(\frac{1}{\tau_b} - \frac{1}{\tau_d} \right)$$

trapping rate

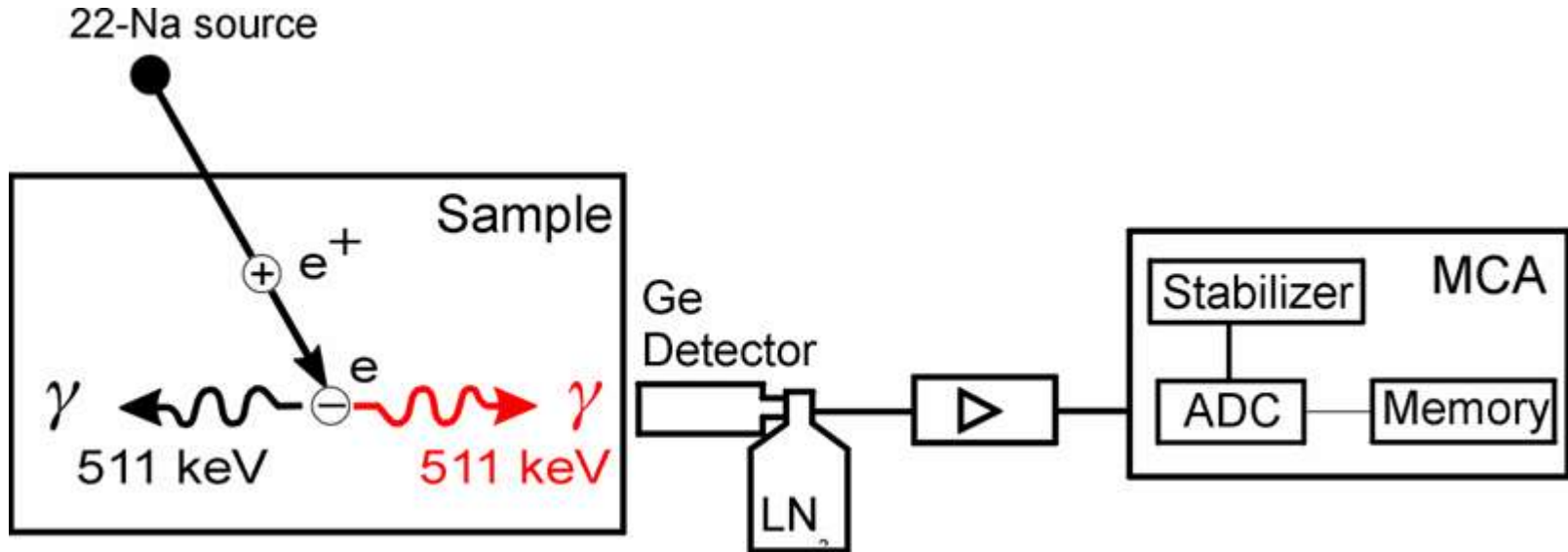
defect concentration



Doppler Broadening Spectroscopy

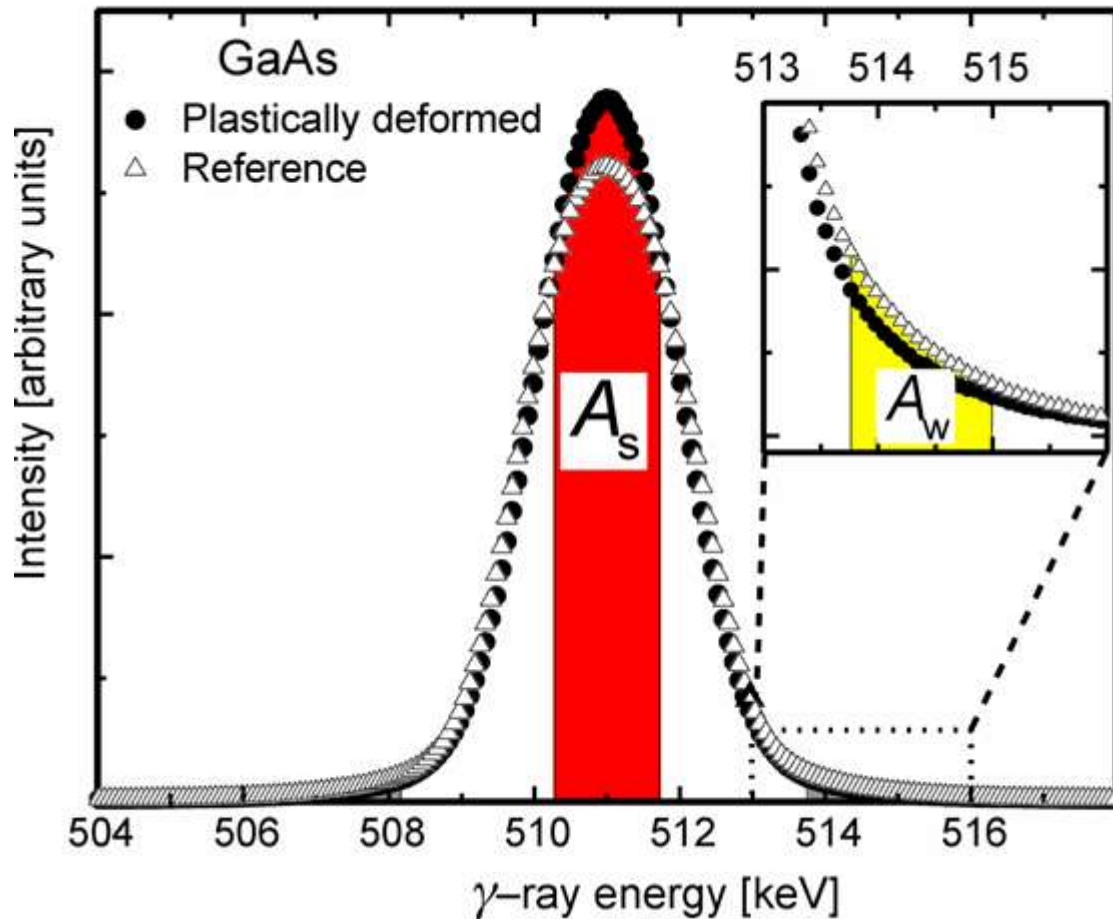


Measurement of Doppler Broadening



- electron momentum in propagation direction of 511 keV γ -ray leads to Doppler broadening of annihilation line
- can be detected by conventional energy-dispersive Ge detectors and standard electronics

Line Shape Parameters



S parameter:

$$S = A_S/A_0$$

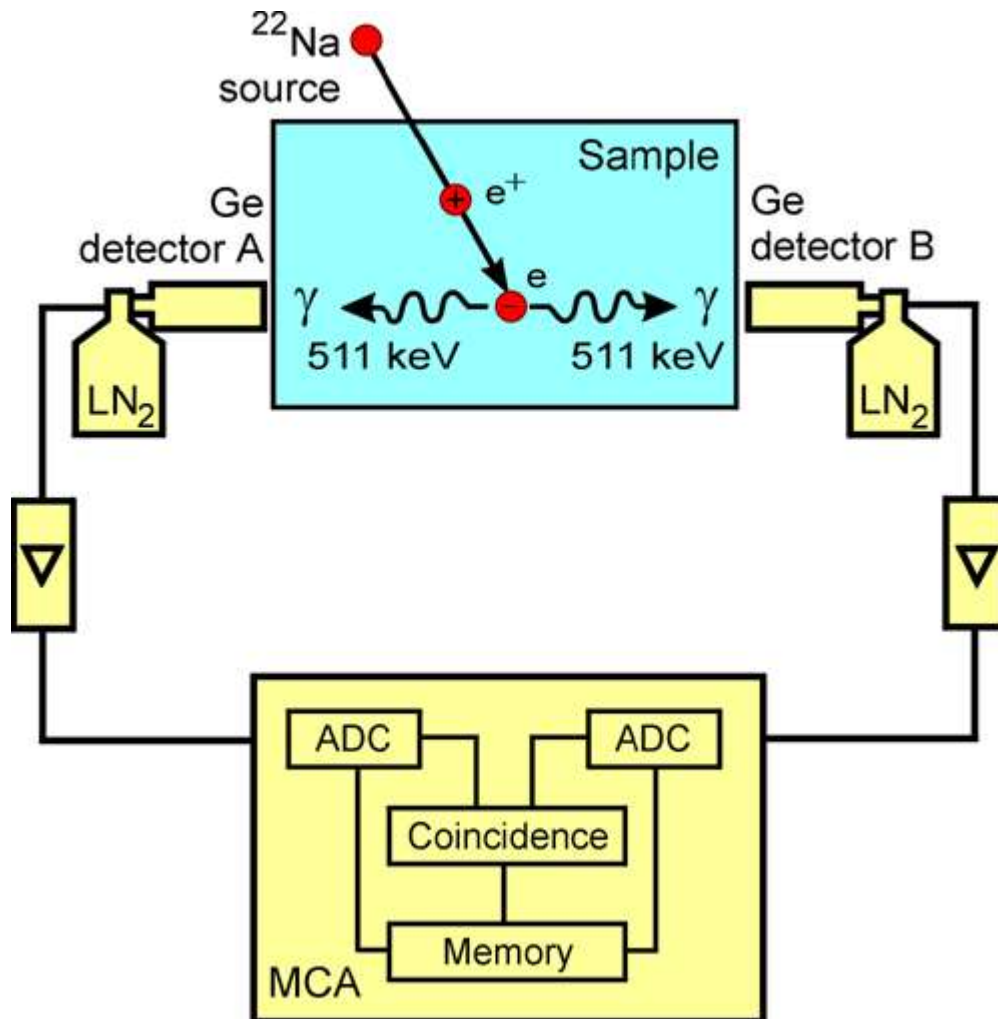
W parameter:

$$W = A_W/A_0$$

W parameter mainly determined by annihilations of core electrons (chemical information)



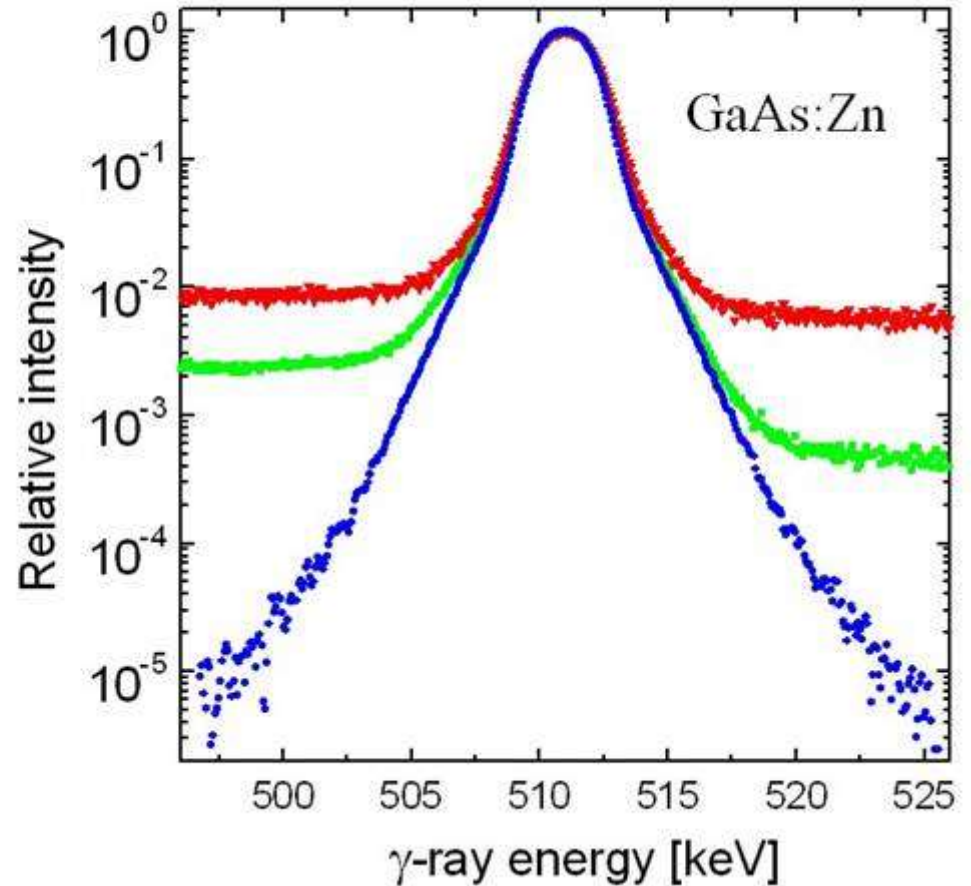
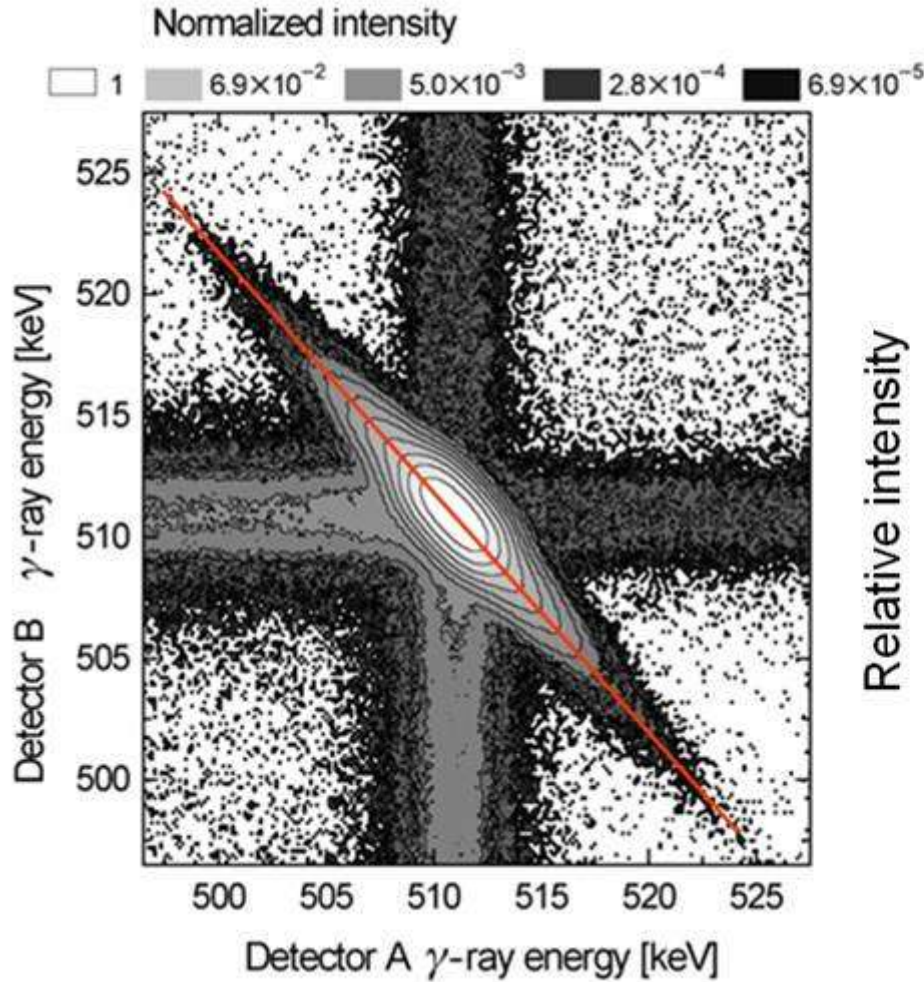
Doppler Coincidence Spectroscopy



- coincident detection of second annihilation γ reduces background
- use of a second Ge detector improves energy resolution of system



Doppler Coincidence Spectra

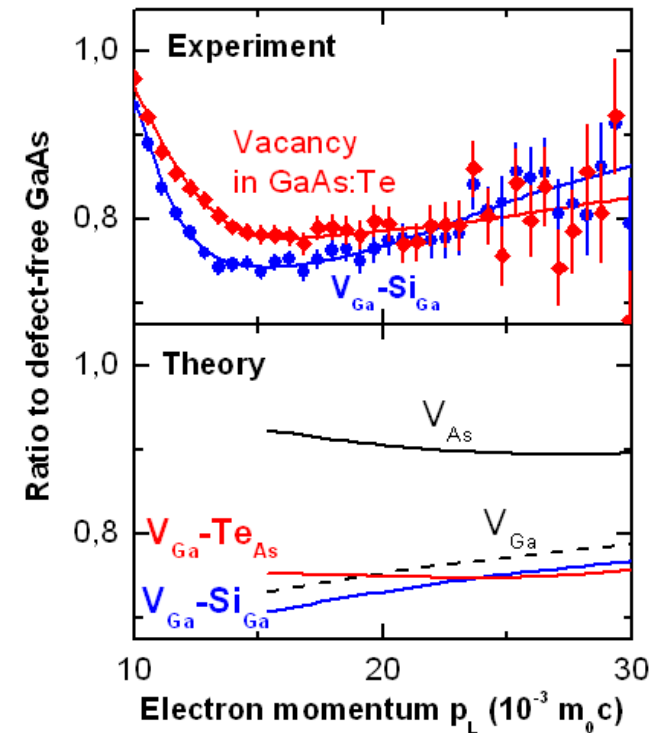
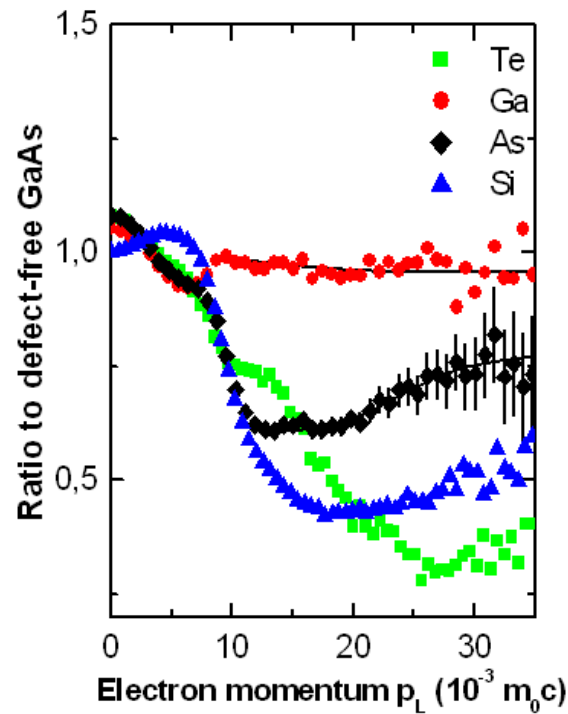
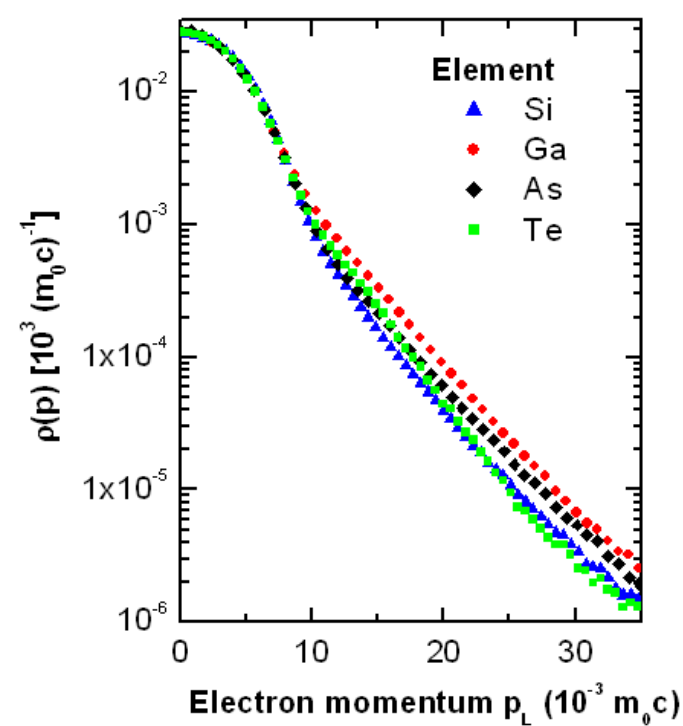


$$E_1 + E_2 = 2 m_0 c^2 = 1022 \text{ keV}$$



Doppler-Coincidence-Spectroscopy in GaAs

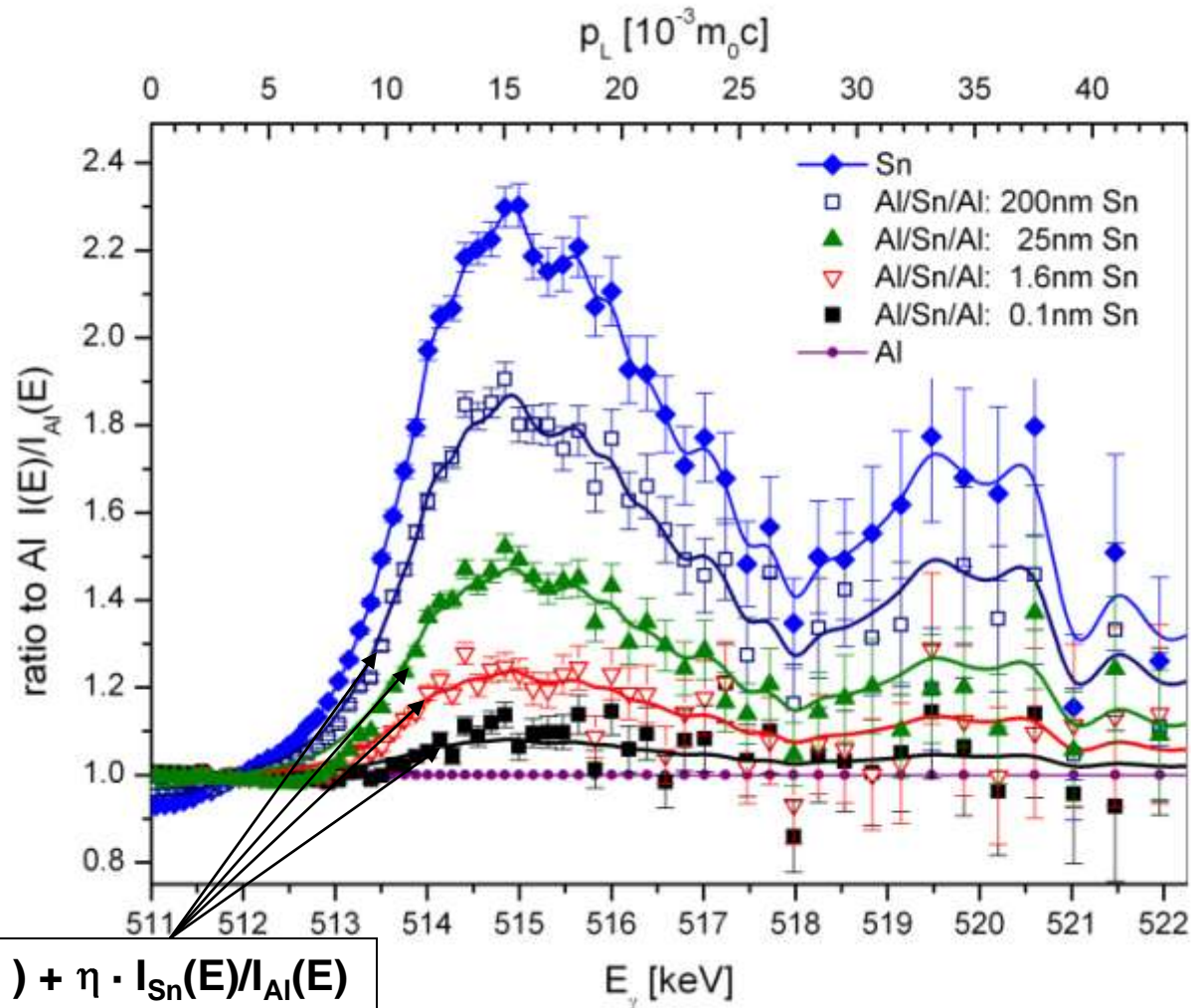
- Chemical sensitivity due to electrons at high momentum (core electrons)
- a single impurity atom aside a vacancy is detectable
- examples: $V_{Ga}-Te_{As}$ in GaAs:Te



J. Gebauer et al., Phys. Rev. B **60** (1999) 1464



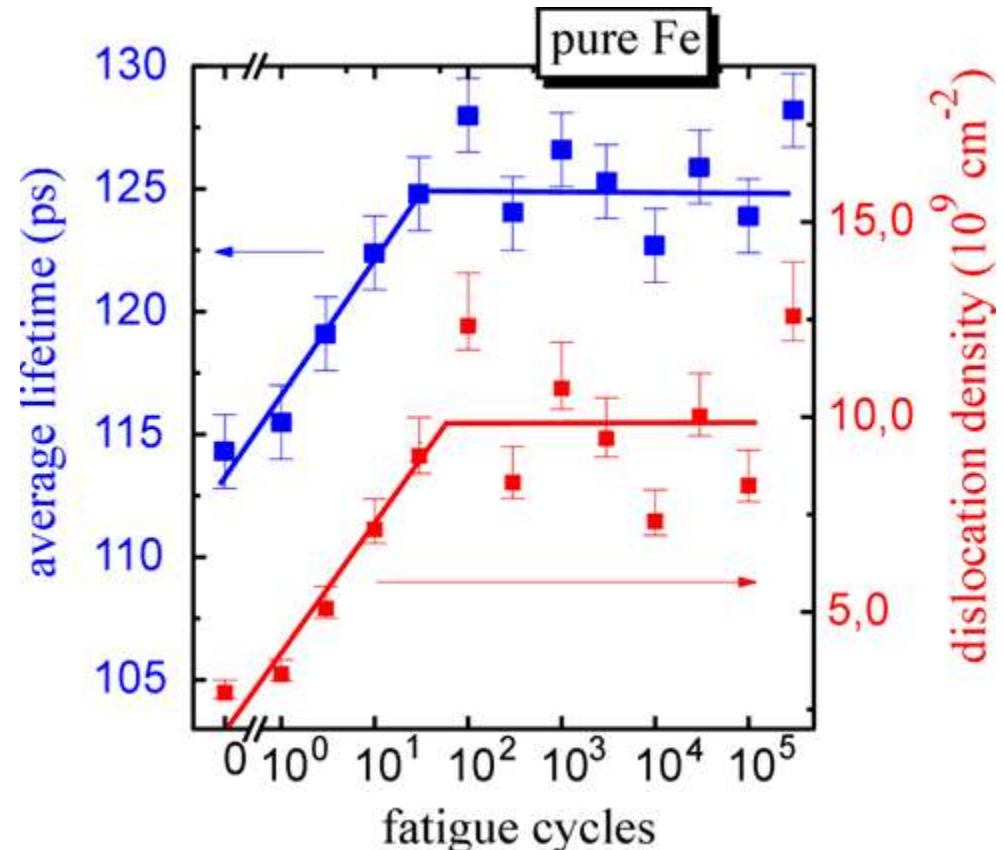
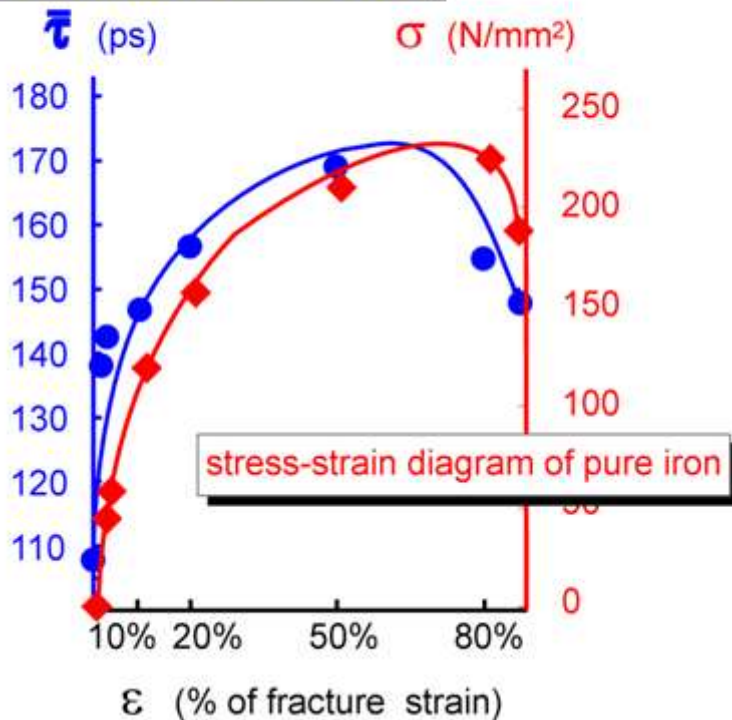
High Sensitivity of CDBS for thin Layers



Defects in Iron after tensile strength and fatigue treatment

- We performed an extensive study of defects in mechanically damaged iron and steel
- Positrons are very sensitive: detection of defects already in the Hooks range of the stress-strain experiment
- Vacancy cluster and dislocations are detectable in both cases

average positron lifetime in pure iron after tensile strain

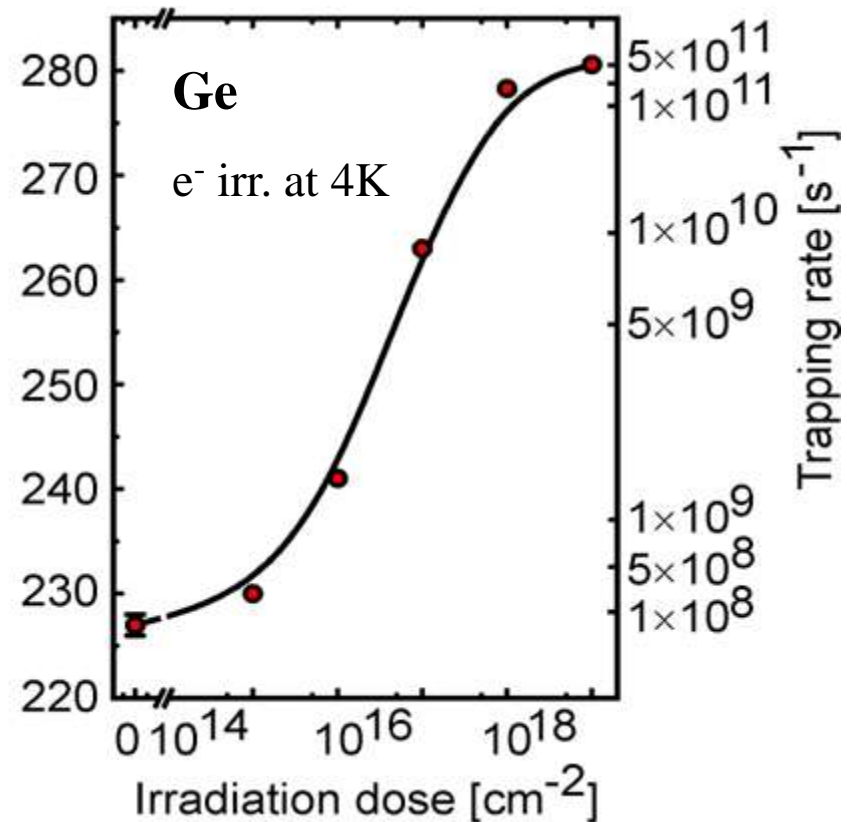
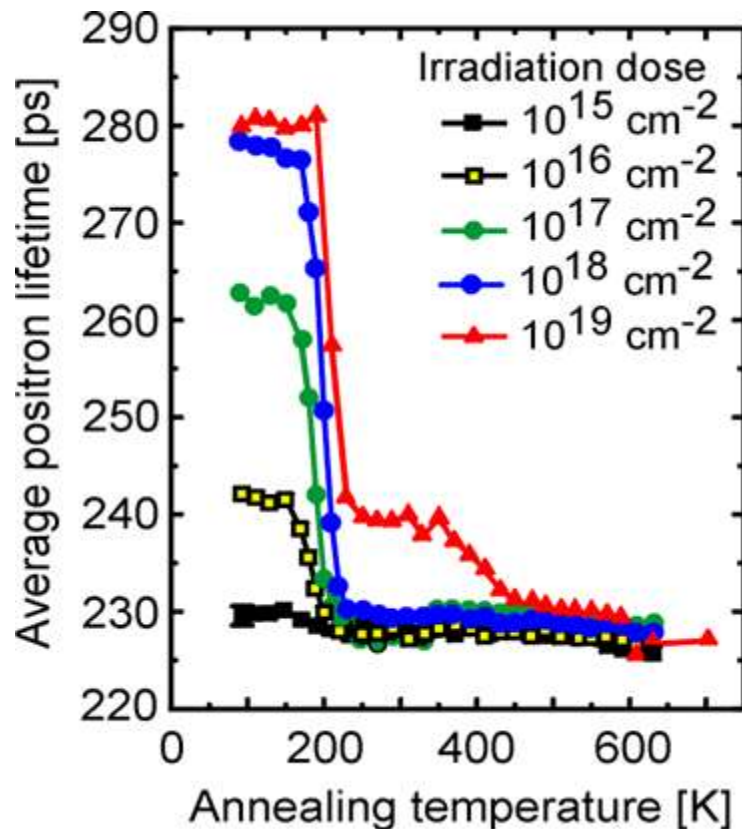


Somieski et al., J. Physique IV 5, C1/127-134 (1995)



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)

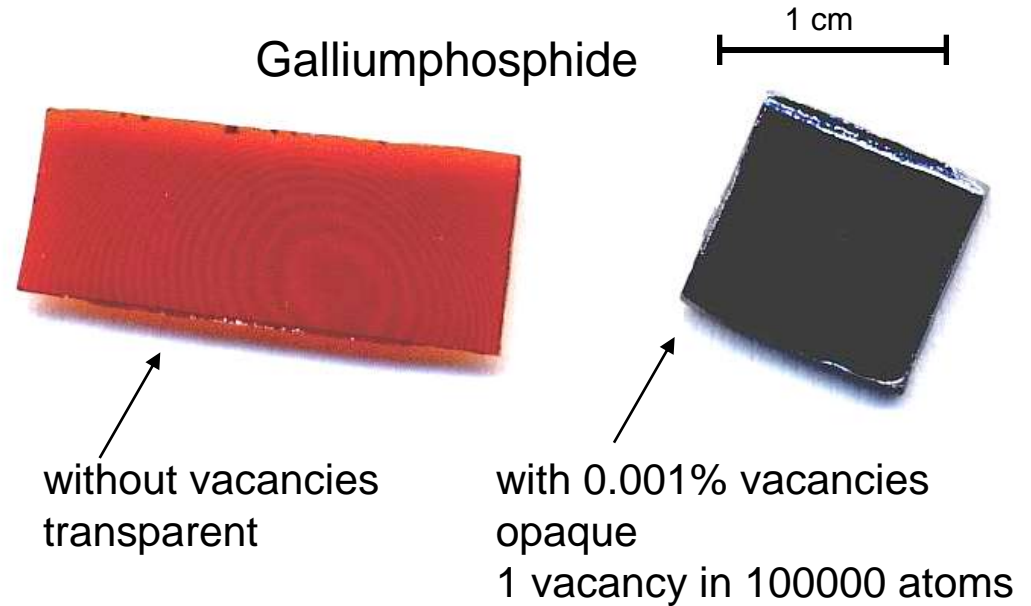


(Polity et al., 1997)



Point defects determine properties of materials

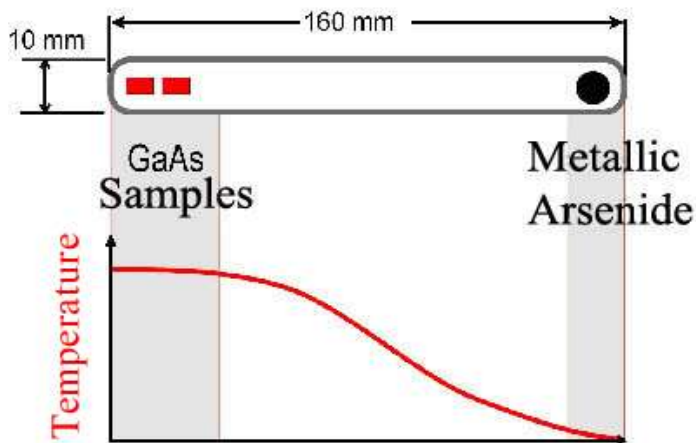
- Point defects determine electronic and optical properties



- Point defects are generated by irradiation (e.g. cosmic rays), by plastic deformation or by diffusion, ...
- Metals in high radiation environment -> formation of voids -> embrittlement

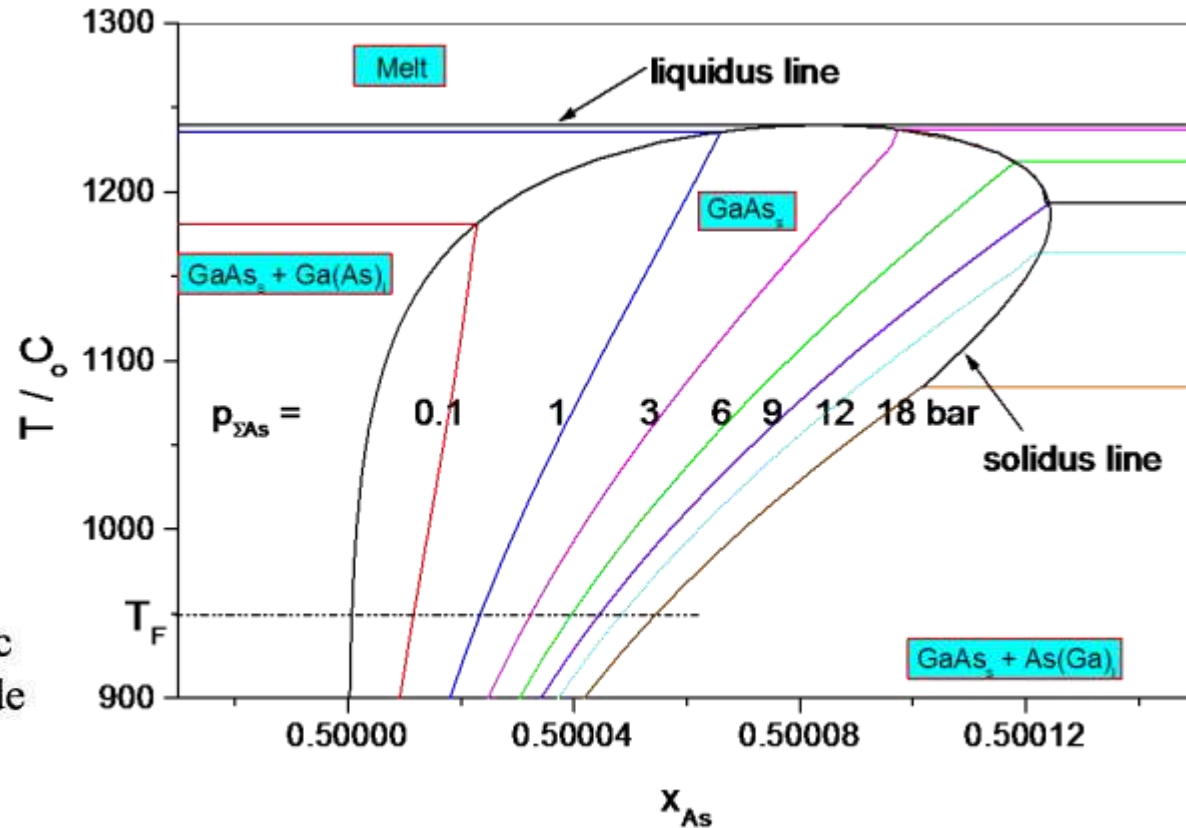
GaAs: annealing under defined As-partial pressure

- two-zone-furnace: Control of sample temperature **and** As partial pressure allows
- T_{As} : determines As-partial pressure
- navigate freely in phase diagram (existence area of compound)



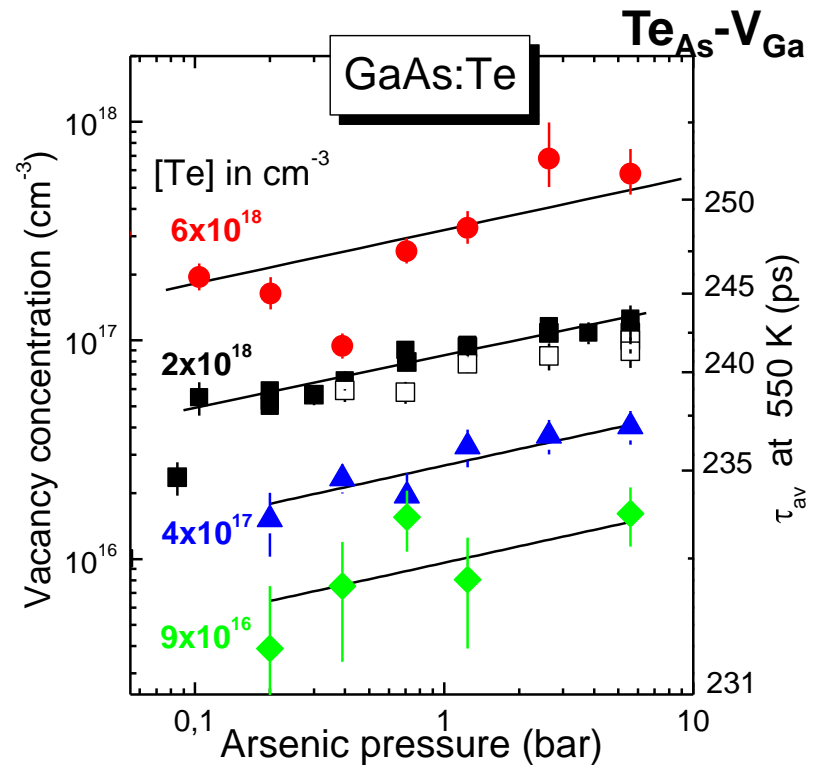
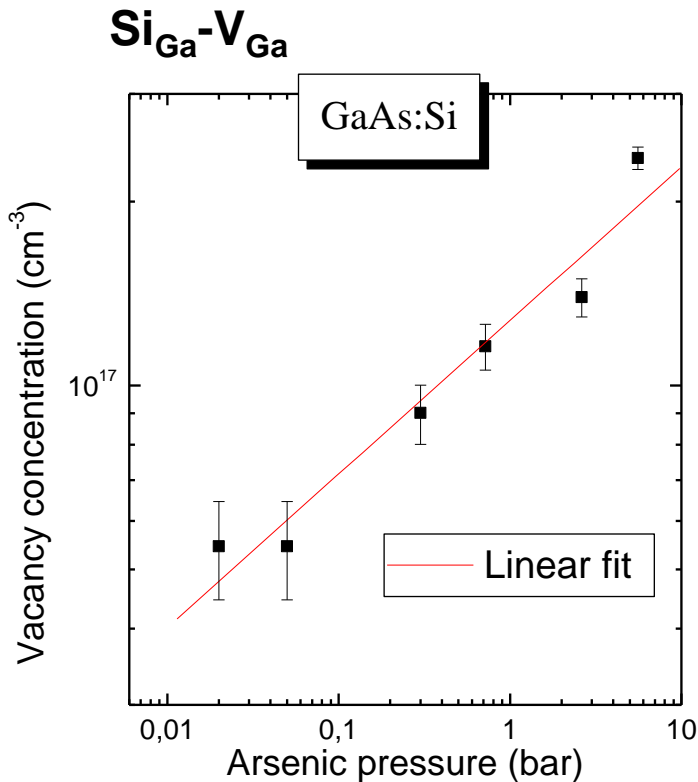
$T_{\text{sample}}: 1100^\circ \text{C}$

Equilibrium Phase Diagram of GaAs

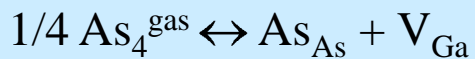


Jurisch, Wenzl; 2002

GaAs: Annealing under defined As pressure



Thermodynamic reaction:



Mass action law:

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

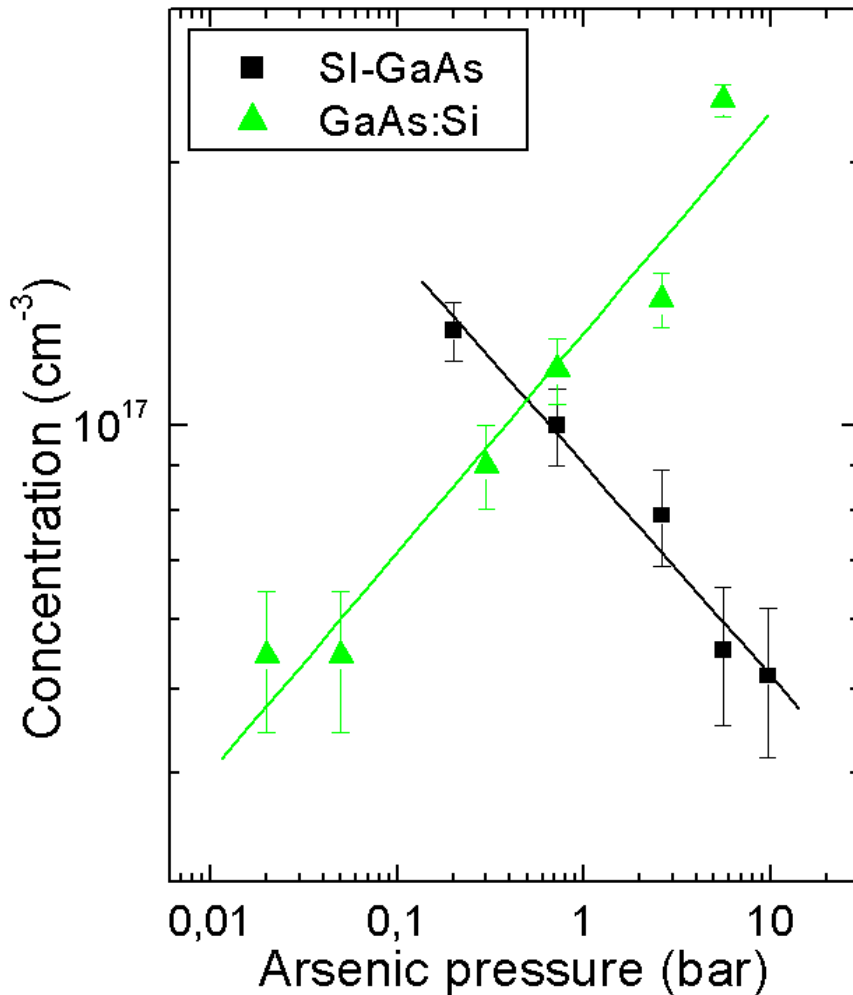
*J. Gebauer et al.,
Physica B 273-274, 705 (1999)*

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

→ $n = 1/4$



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

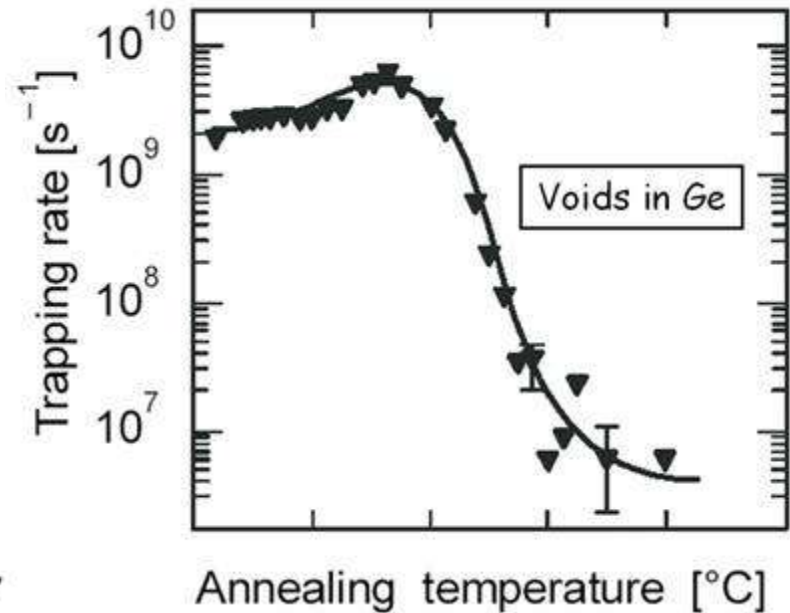
$$\rightarrow n = -1/4$$

undoped GaAs: As vacancy

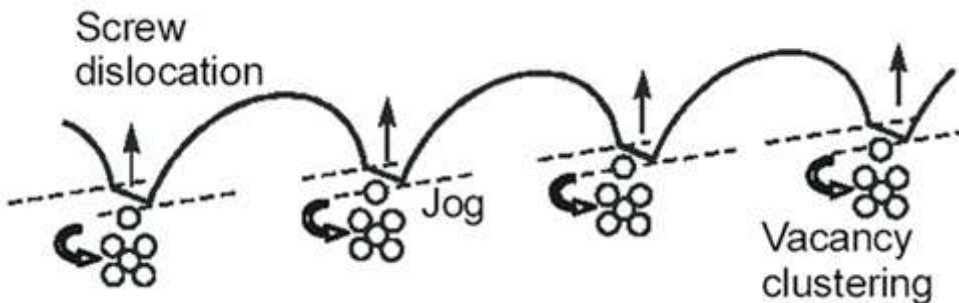


Vacancy clusters in semiconductors

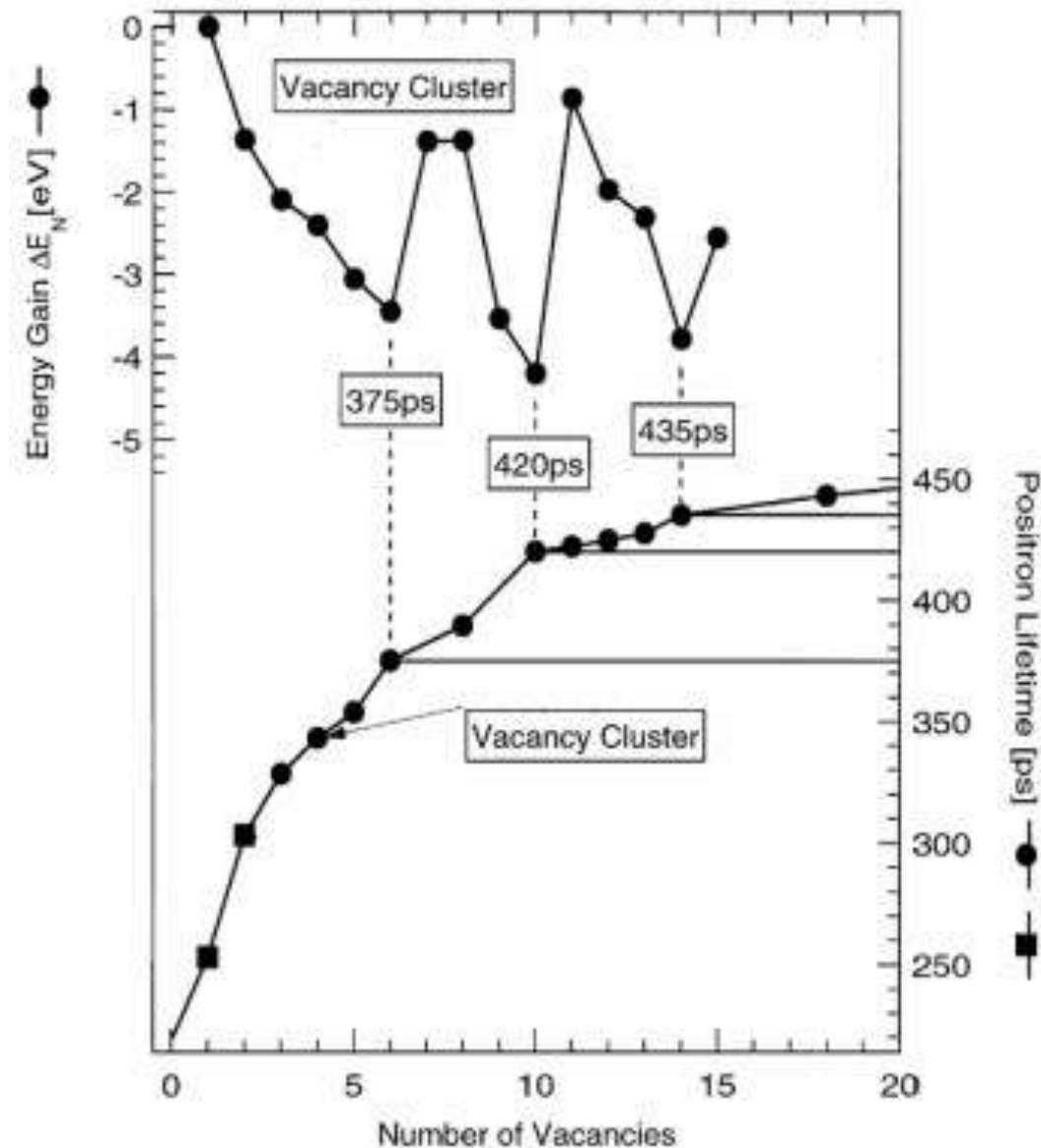
- vacancy clusters were observed after neutron irradiation, ion implantation and plastic deformation
- due to large open volume (low electron density) -> positron lifetime increases distinctly
- example: plastically deformed Ge
- lifetime: $\tau = 525$ ps
- reason for void formation: jog dragging mechanism
- trapping rate of voids disappears during annealing experiment



Krause-Rehberg et al., 1993



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



Vacancy clustering during defect annealing

- electron irradiated Fe
- clustering in early stage can be observed
- very sensitive: formation of divacancies and small clusters ($n < 10$)

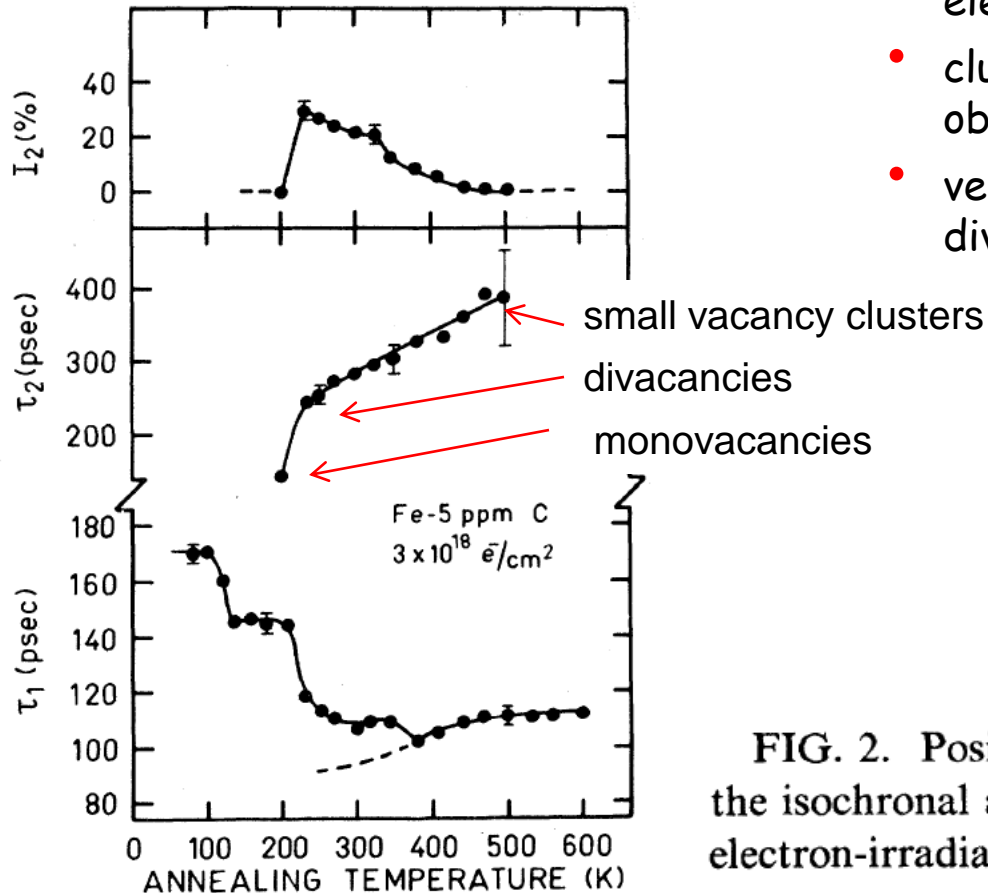
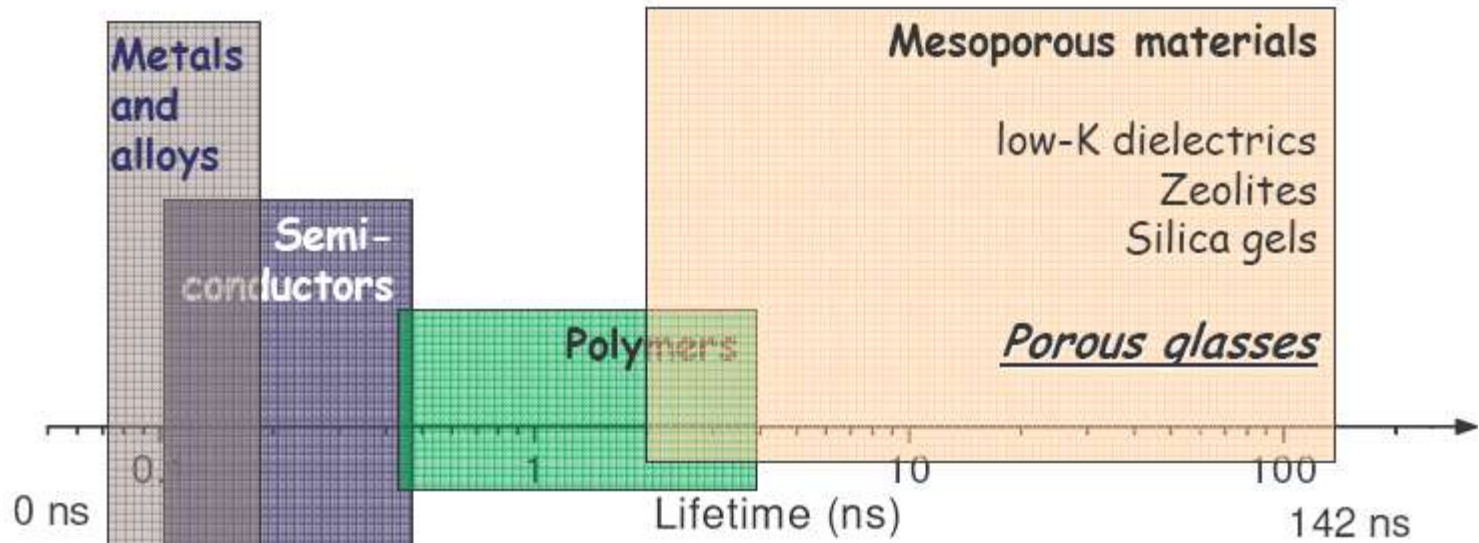


FIG. 2. Positron-lifetime parameters as a function of the isochronal annealing temperature in the low-dose electron-irradiated pure iron.



Typical Lifetimes



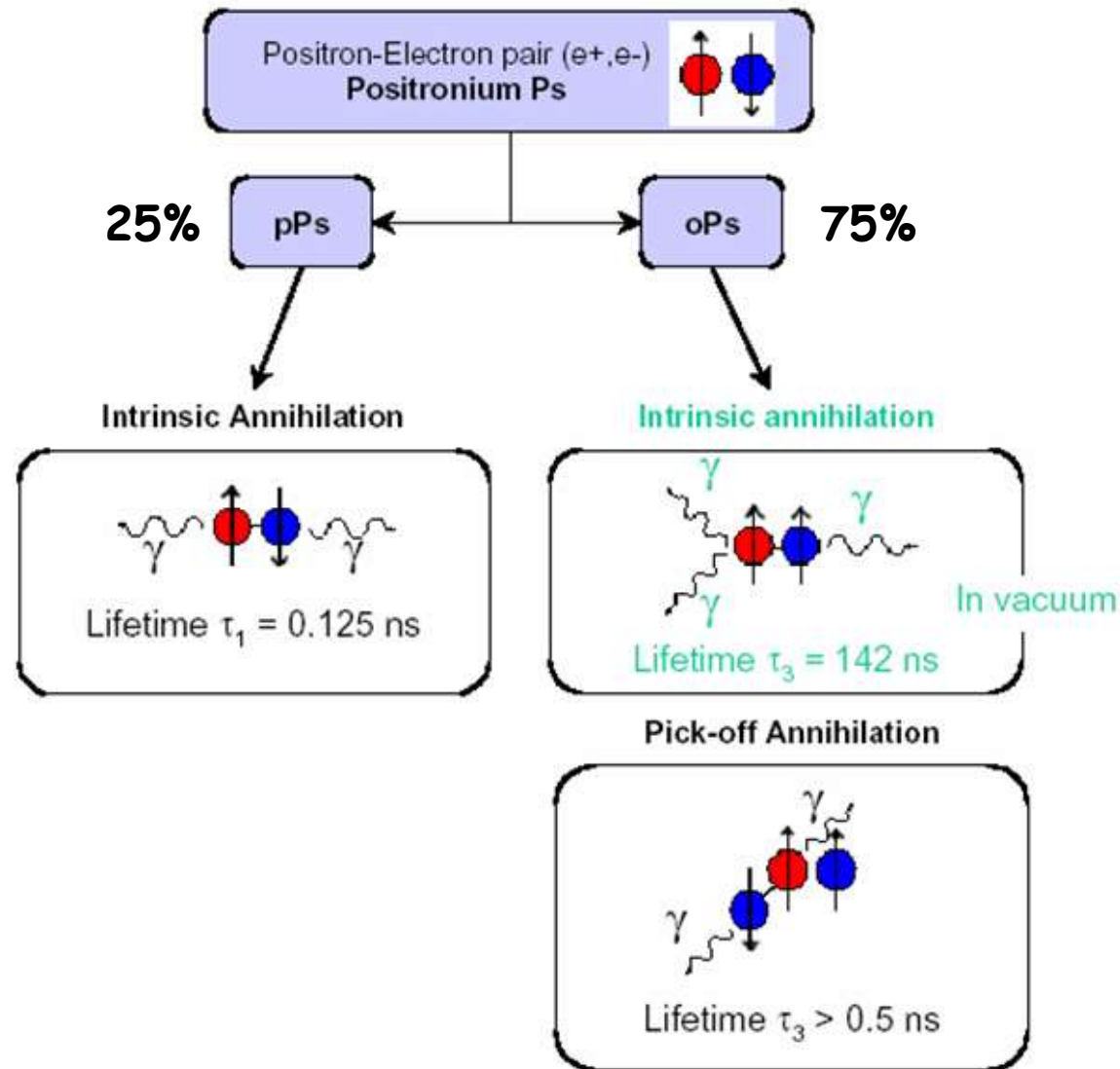
Positron

Positronium



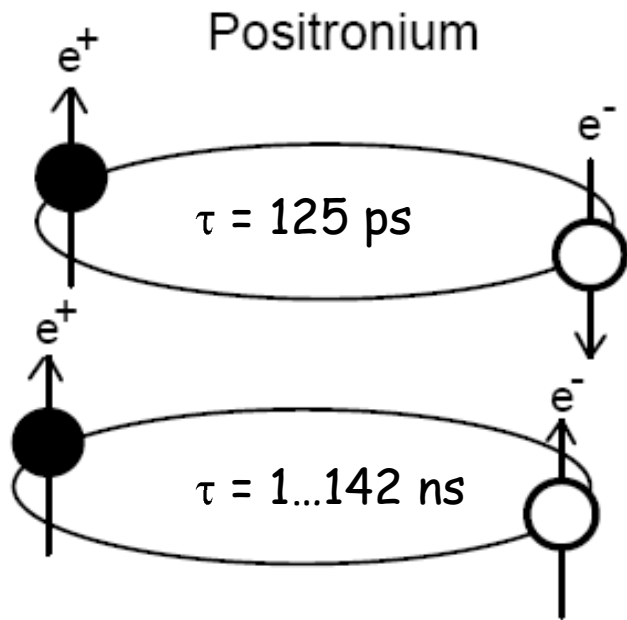
o-Positronium Lifetime allows Porosimetry

- In materials without free electrons Positronium may be formed (Polymers, glass, liquids, gases)
- p-Ps annihilates without interaction with host material
- o-Ps lifetime in vacuum 142 ns
- in matter: positron may pick off another electron with opposite spin \rightarrow fast annihilation with two gammas



Pick-off Annihilation

positrons form Ps

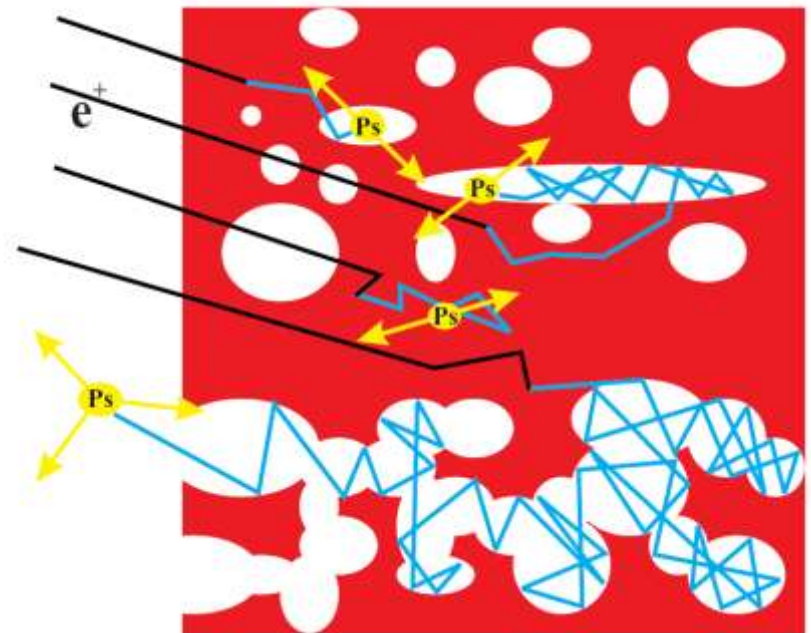


para-Ps
 1S_0

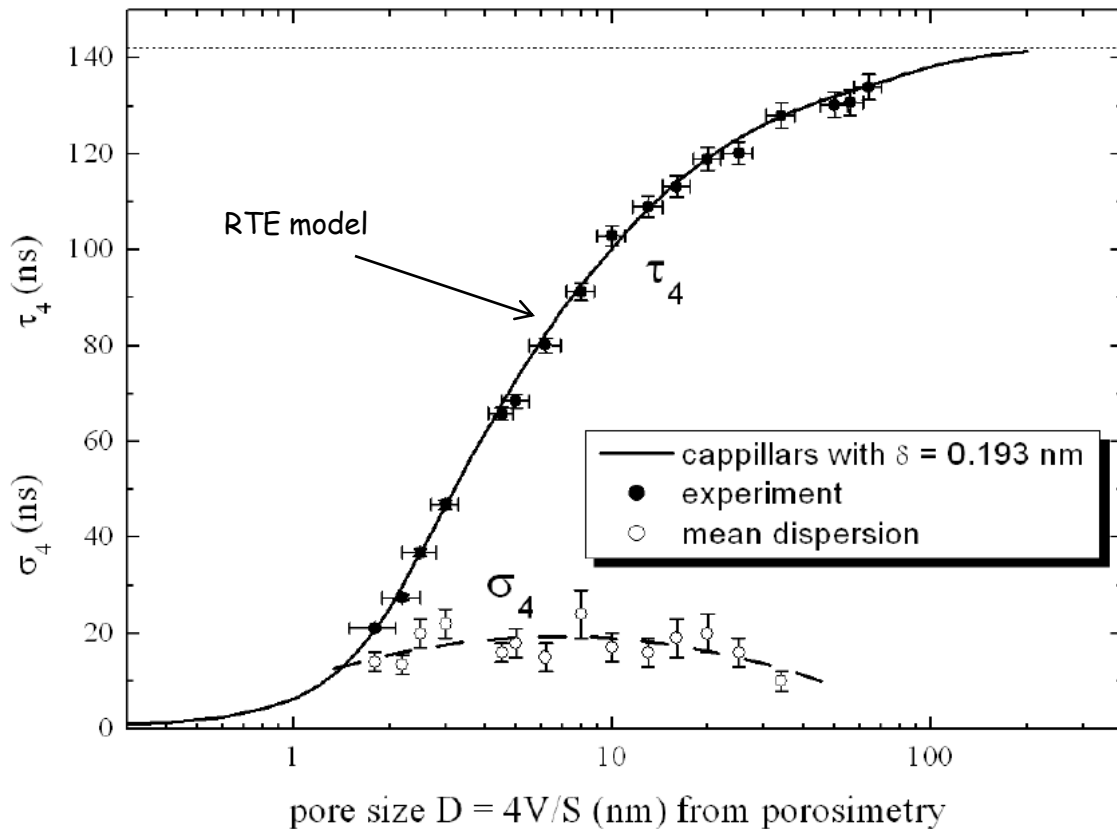
ortho-Ps
 3S_1

pick-off annihilation:

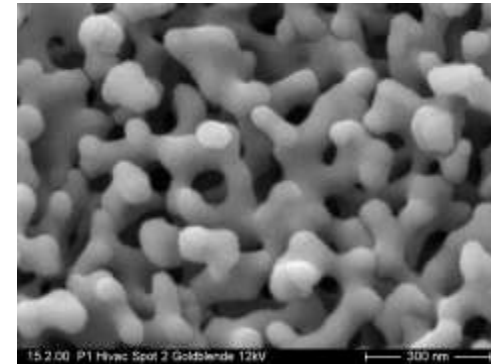
- o-Ps is converted to p-Ps by capturing an electron with anti-parallel spin
- happens during collisions at walls of pore
- lifetime decreases rapidly
- lifetime is function of pore size 1.5 ns to 142 ns



o-Ps lifetime τ_4 versus pore size in CPG Glass



- we measured porous CPG glass in a broad pore size range

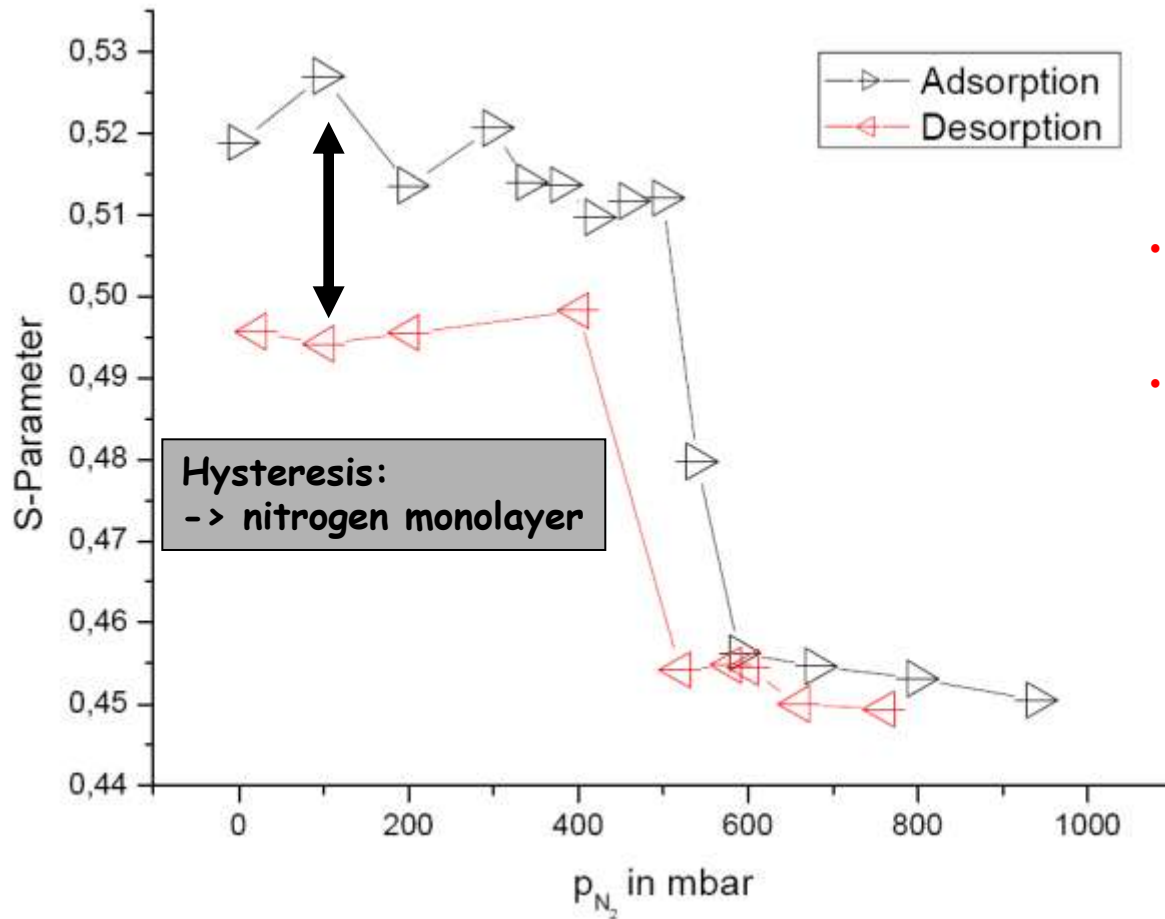


- given pore size obtained by N_2 -adsorption and/or mercury intrusion technique
- for $T=300$ K fair agreement to the RTE model

S.Thränert, Dissertation, MLU Halle 2008



Cryo-condensation in nano-pores

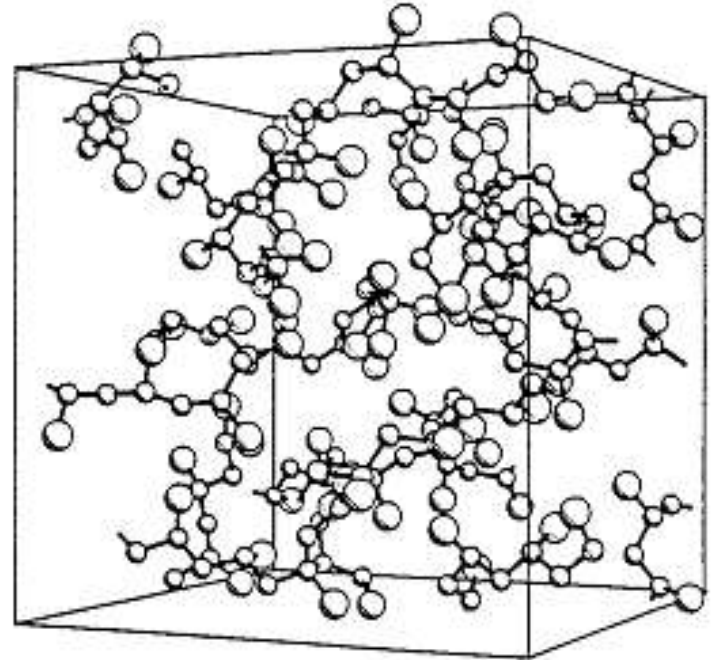


- N_2 -Adsorption at $T = 80$ K and $d = 16$ nm
- study of phase transitions under confinement conditions possible



Structure of the free Volume in Soft Matter

- free volume due to structural, static or dynamic, disorder
- important for several macroscopic properties of these materials,
- viscosity, molecular transport, structural relaxation, and physical aging
- Positron Annihilation: Detection of subnanometric local free volumes (holes):
- mean size and size distribution

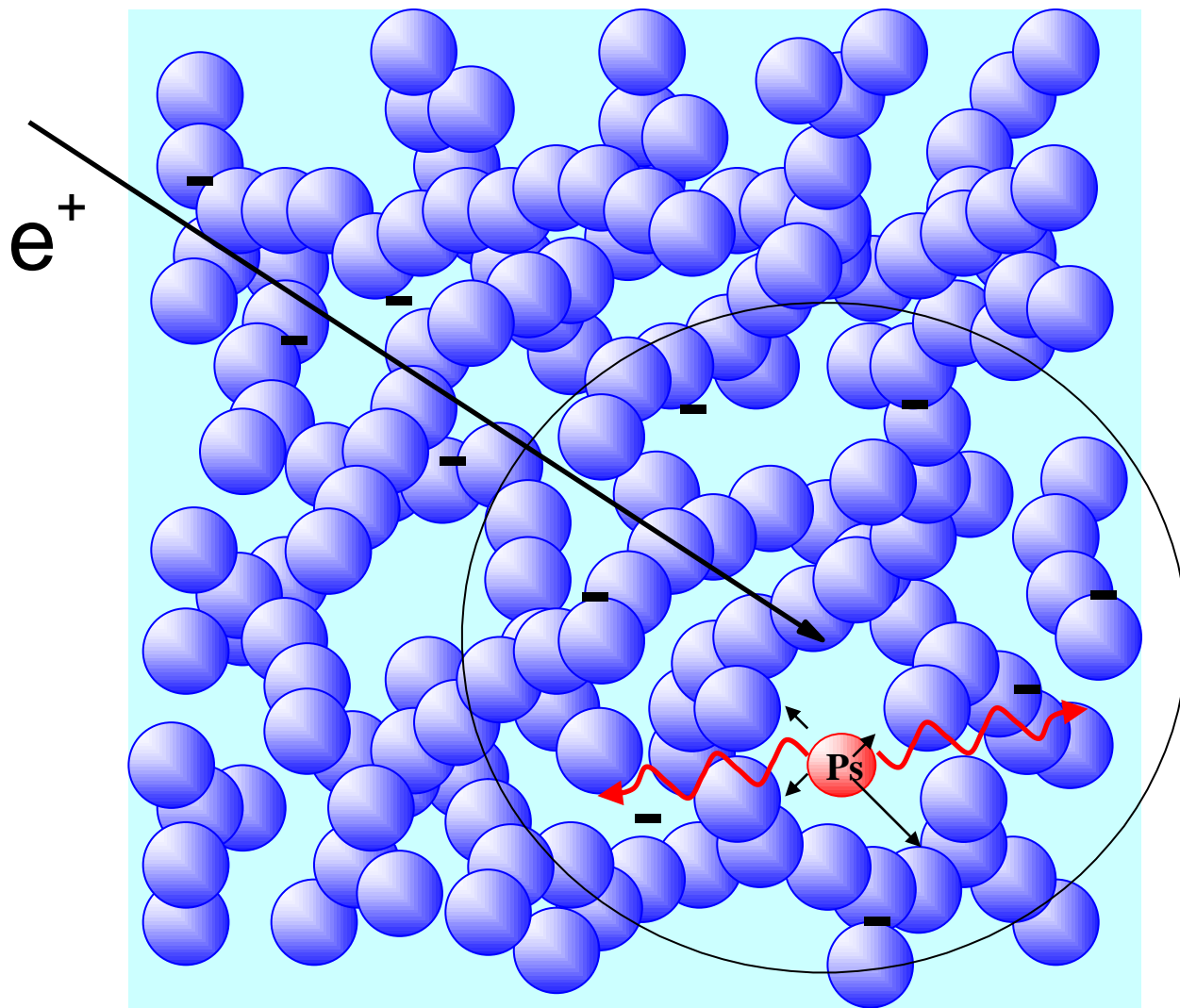


Schematic representation of the Polypropylene microstructure
(Simulation, Theodoru et al. 1985)

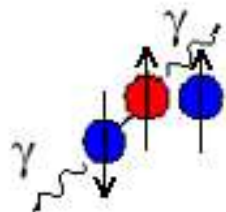


Ps Formation in Polymers

β^+ source $^{22}\text{NaCl}$



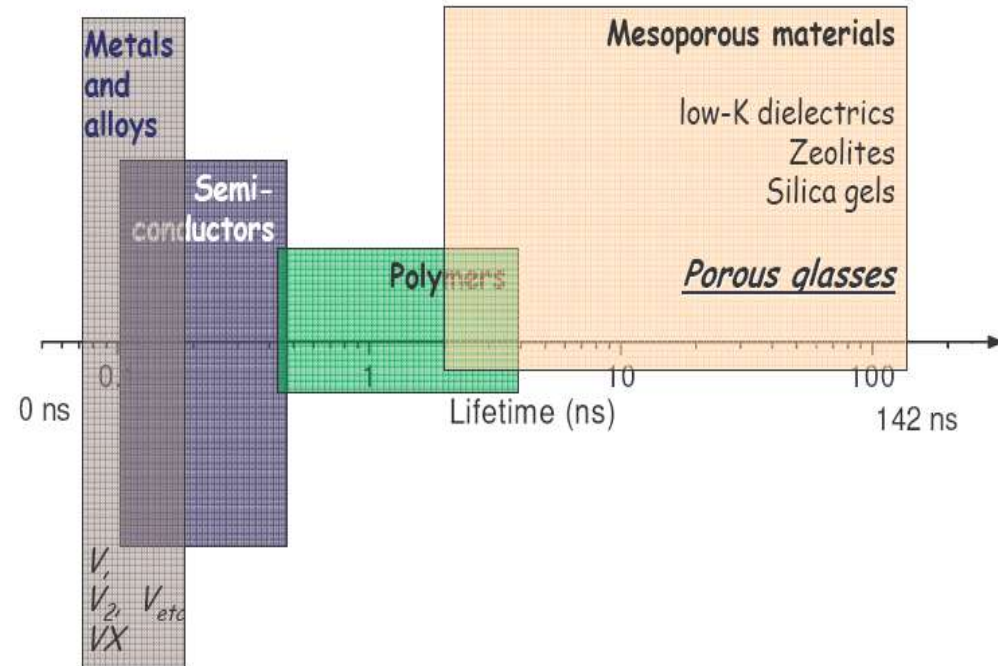
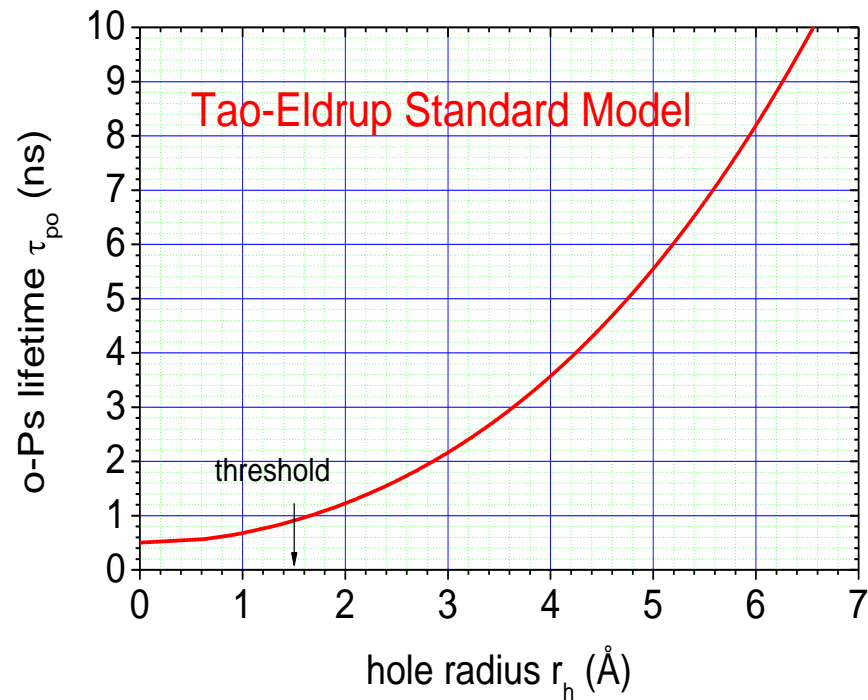
Pick-off Annihilation



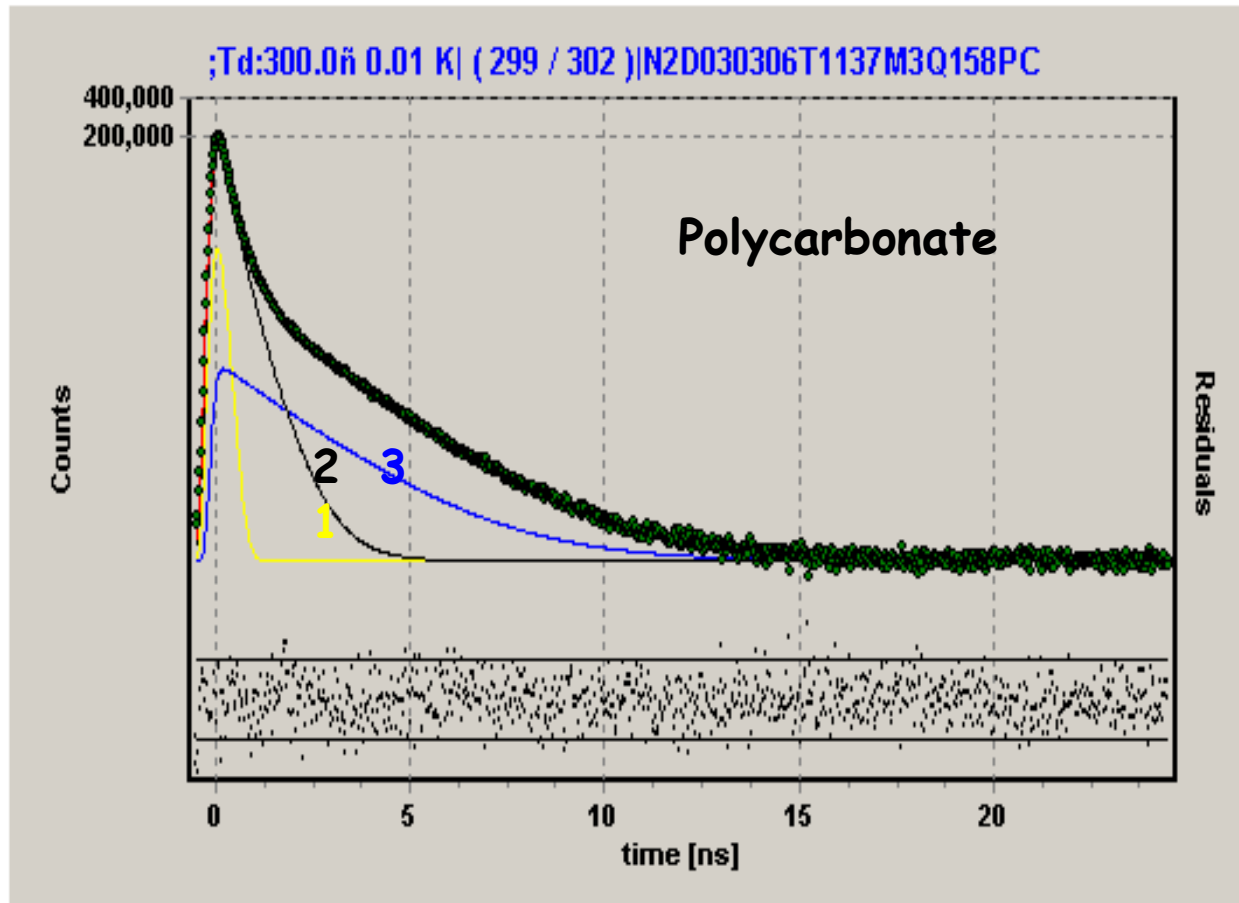
Lifetime $\tau_3 > 0.5$ ns

The o-Ps Lifetime as a Function of the Hole Size

$$\tau_{o-Ps \text{ pickoff}} = \frac{0.5 \text{ ns}}{\left[1 - \frac{r_h}{r_h + \delta r} + \frac{1}{2\pi} \text{Sin} \left(\frac{2\pi r_h}{r_h + \delta r} \right) \right]}$$



Typical PALS Spectrum



p-Ps

$$\tau_1 \approx 0.125 \text{ ns}$$

free positrons

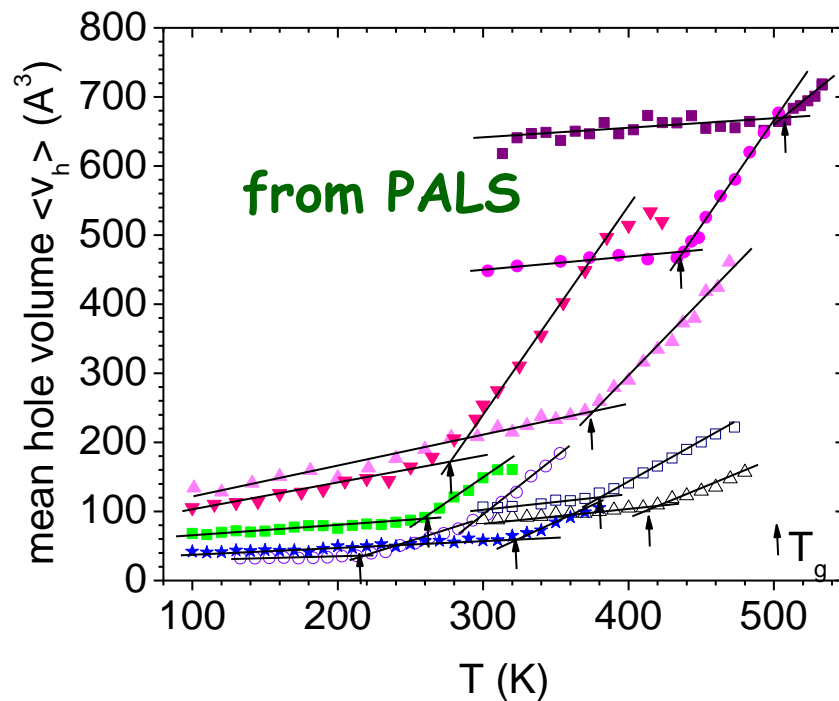
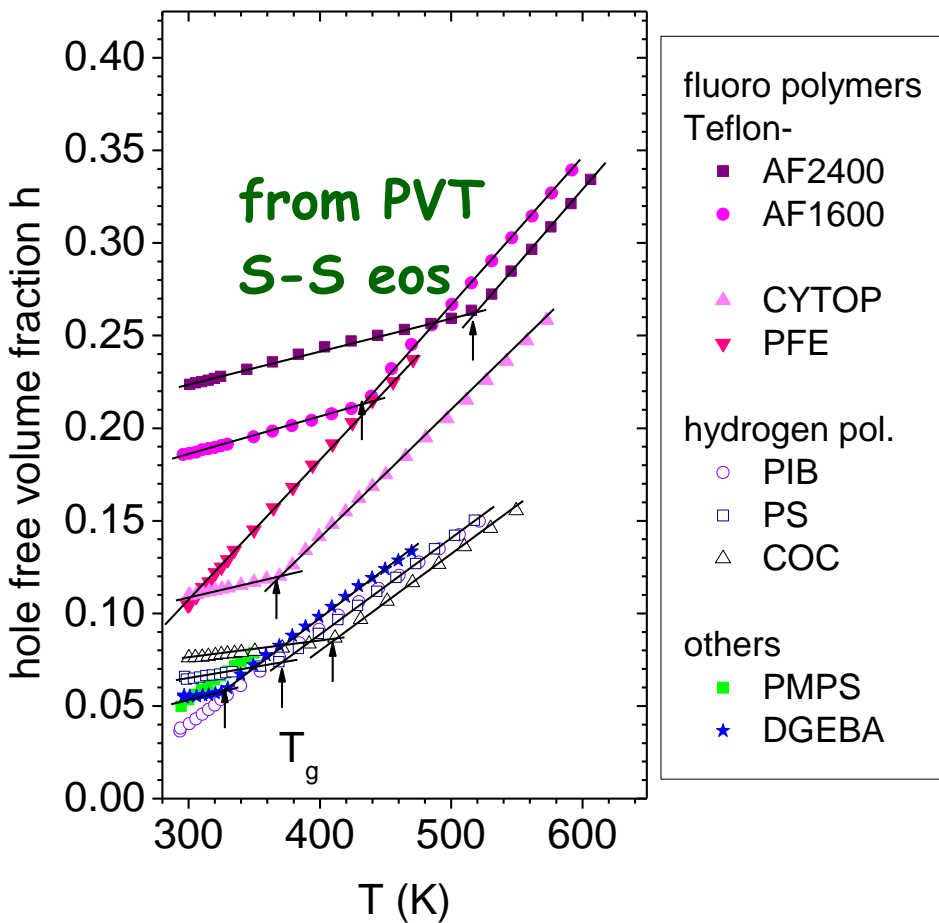
$$\tau_2 \approx 0.35 \text{ ns}$$

o-Ps (pick-off
annihilation)

$$\tau_3 = 1 - 8 \text{ ns}$$

$$n(t) = I_1 \exp(-t/\tau_1) + I_2 \exp(-t/\tau_2) + I_3 \exp(-t/\tau_3)$$

Temperature Dependence of the Hole Free Volume

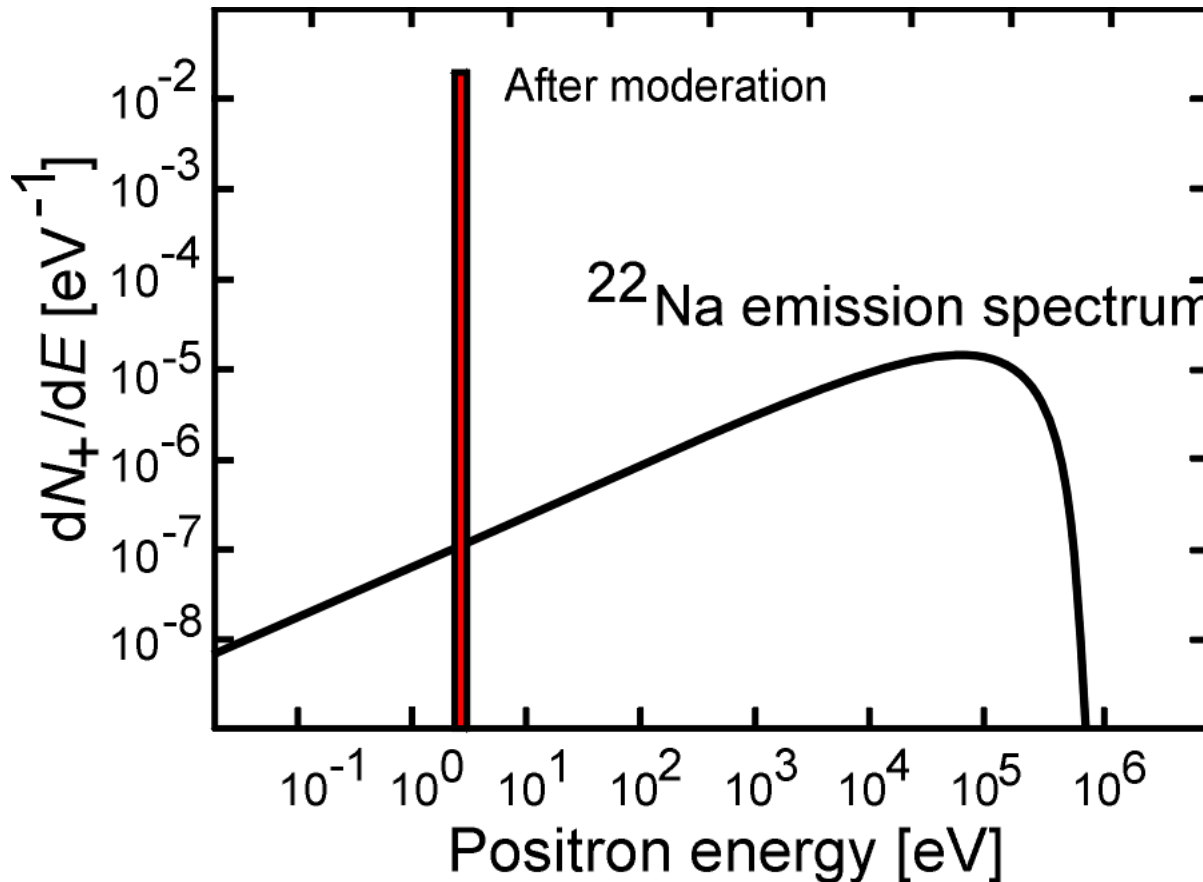


Dlubek et al., *Macromolecules*, 41 (2008) 6125



Moderation of Positrons

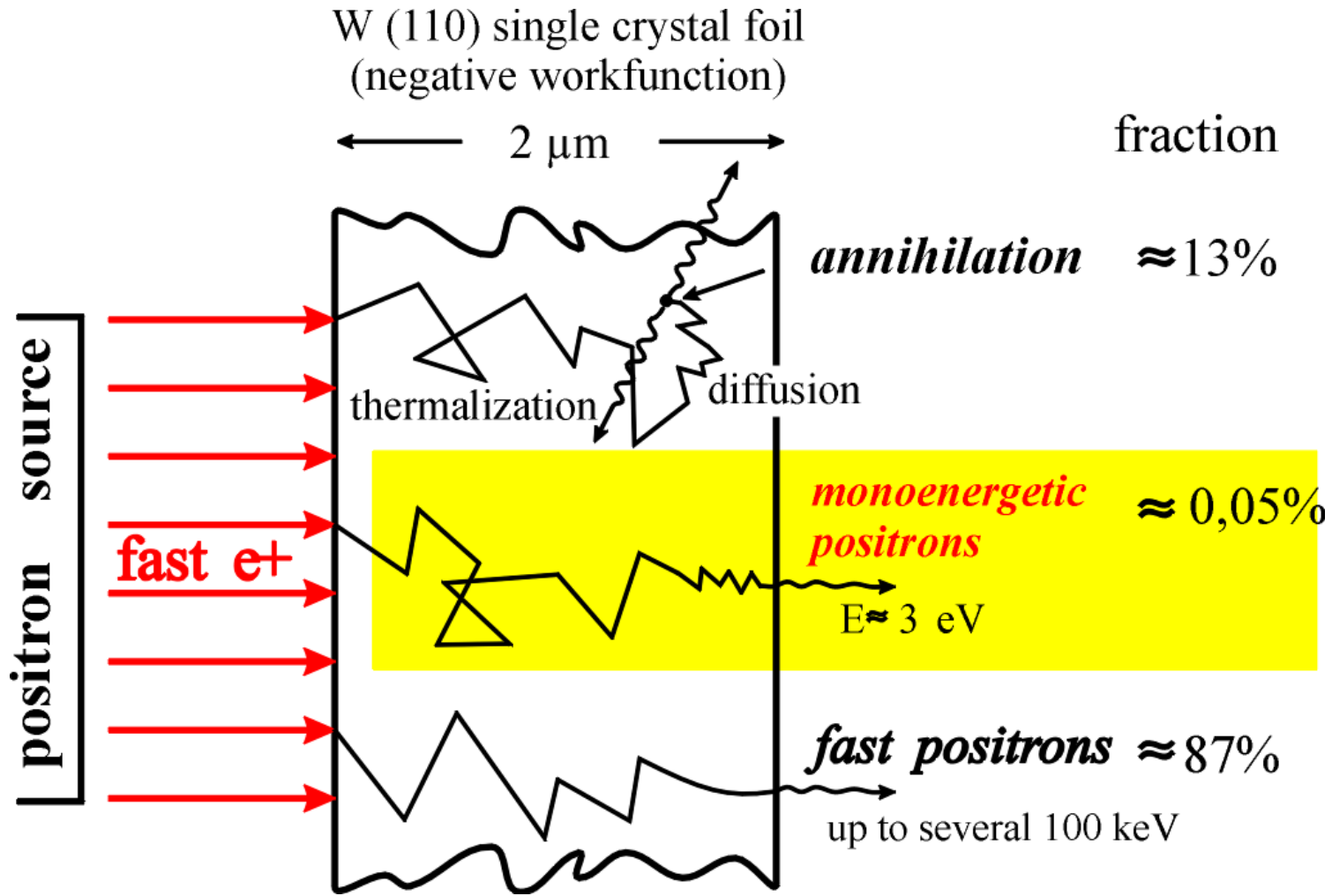
Mean (maximum) implantation depth of un-moderated positrons from a isotope ^{22}Na source ($1/e$ 0.999): Si: $50\mu\text{m}$ ($770\mu\text{m}$)



- 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



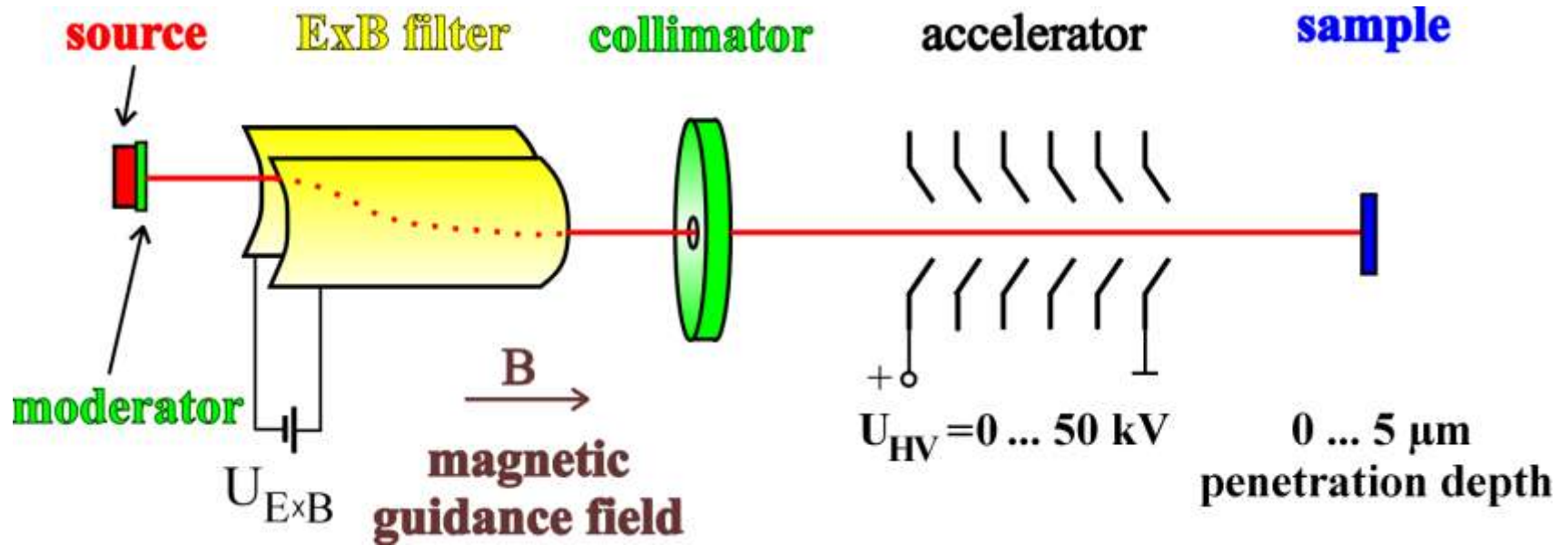
Moderation of Positrons



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

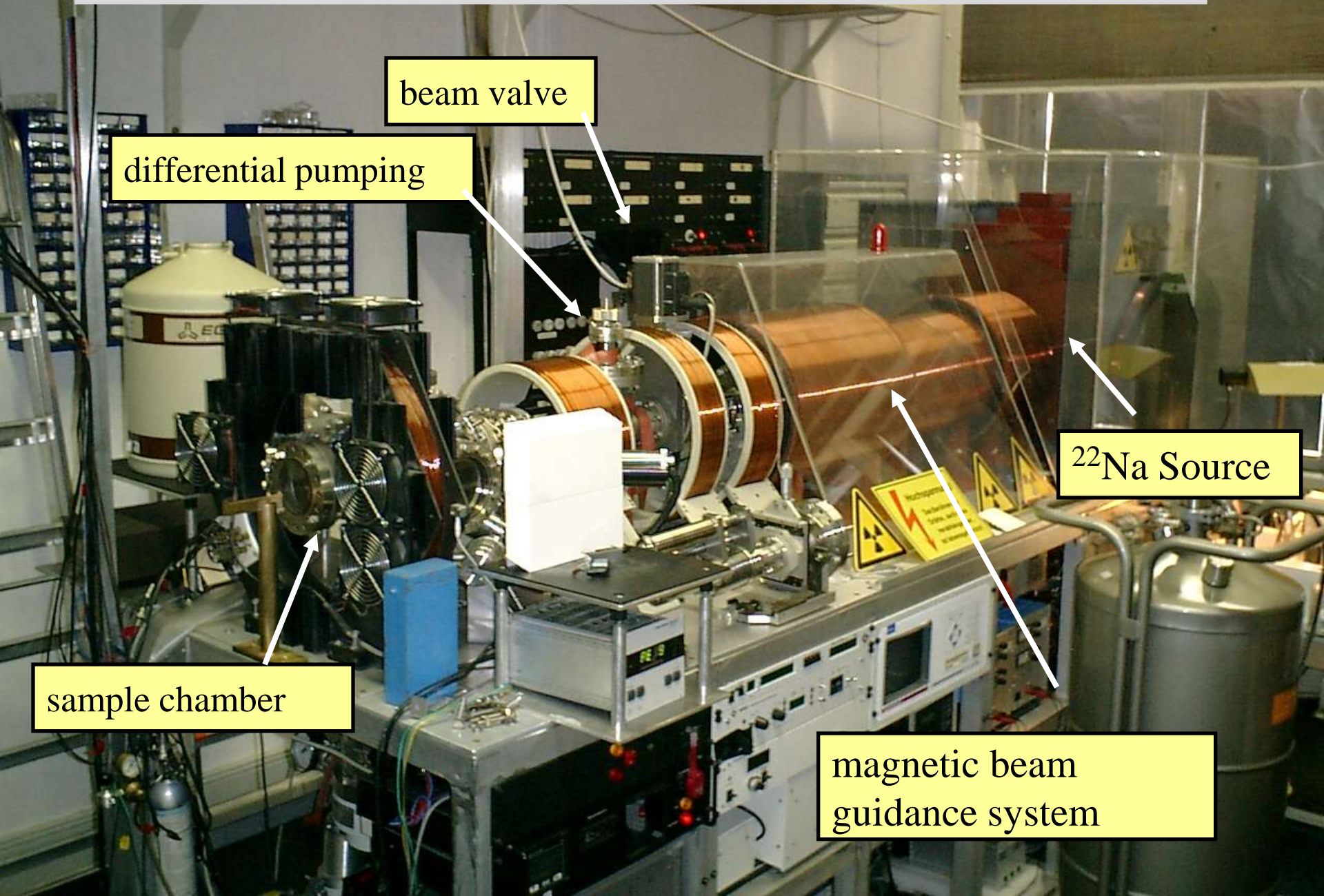


The Positron Beam System at Halle University



- spot diameter: 5mm
- time per single Doppler measurement: 20 min
- time per depth scan: 8 hours

The positron beam system at Halle University



beam valve

differential pumping

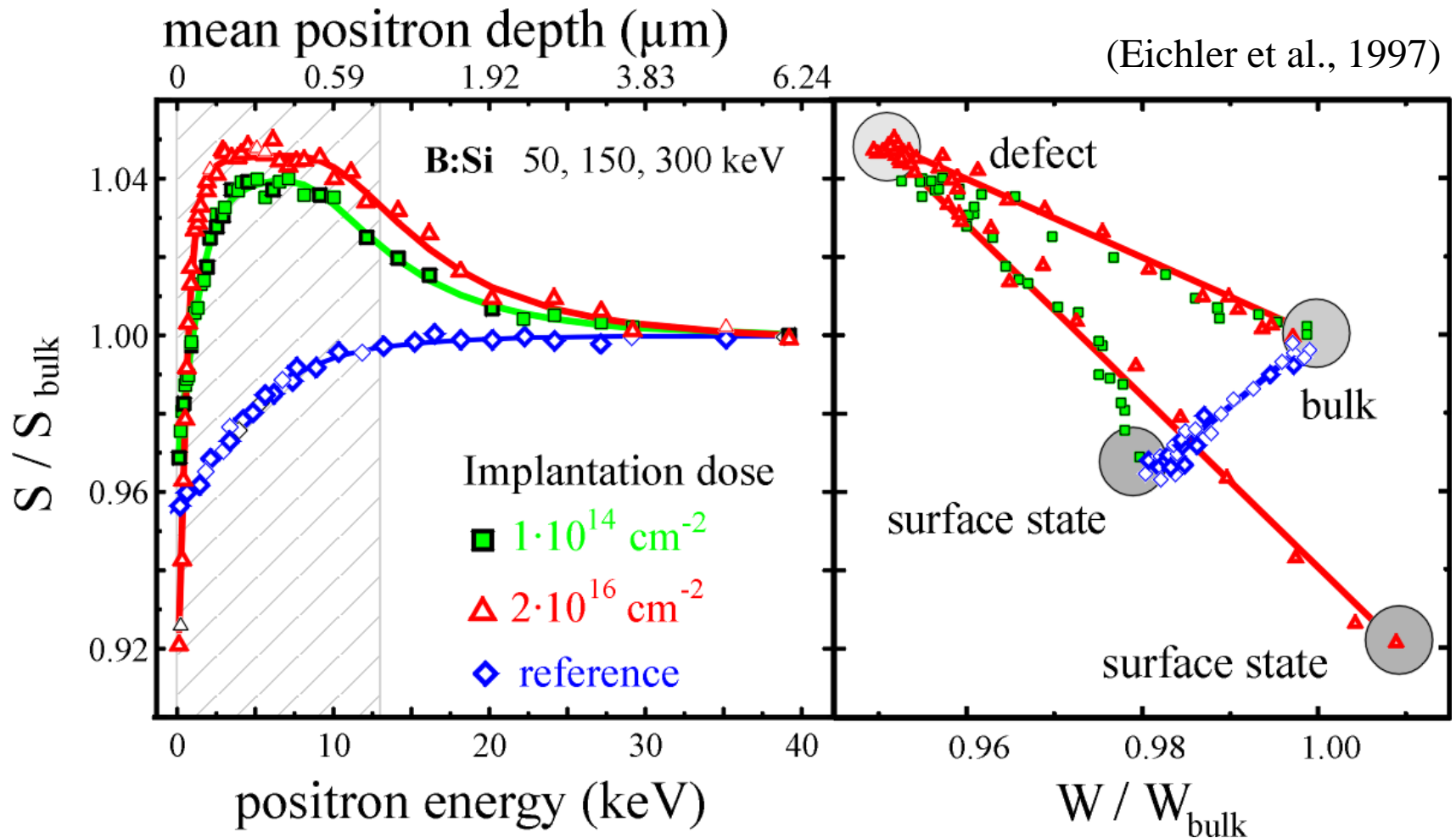
sample chamber

magnetic beam
guidance system

²²Na Source

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



User-dedicated intense Positron Sources in Germany

- Two intense positron sources available (positrons by pair production)
- **NEPOMUC** (NEutron induced POsitrone Source MUniCh) at FRM-II
 - PLEPS (monoenergetic positron lifetime system)
 - PAES (Positron-induced Auger Electron Spectroscopy)
 - CDBS (Coincidence Doppler Broadening Spectroscopy)
 - SCM (Scanning Positron Microscope)
 - user beam line
- **EPOS** (ELBE Positron Source) at Research Center Dresden-Rossendorf
 - MePS (Mono-energetic Positron Spectroscopy)
 - GiPS (Gamma-induced Positron Spectroscopy)
 - CoPS (conventional setup using ^{22}Na sources)
- at both sites: web-based application system for beam time

NEPOMUC at FRM II

SR 11

Remoderator

PLEPS

Switch

SPM interface

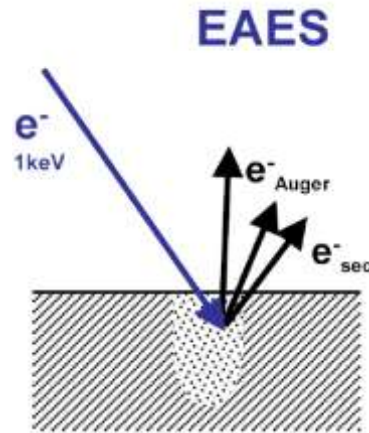
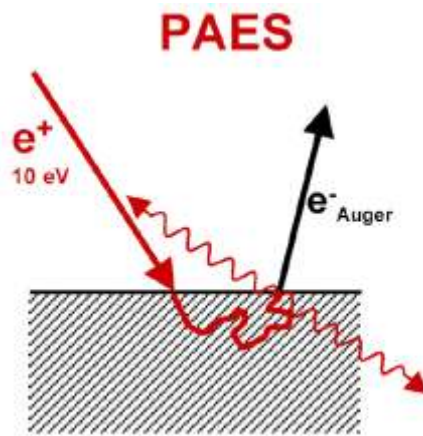
CDBS

PAES

Open
Beamport: Ps⁻



Positron Annihilation Induced AES

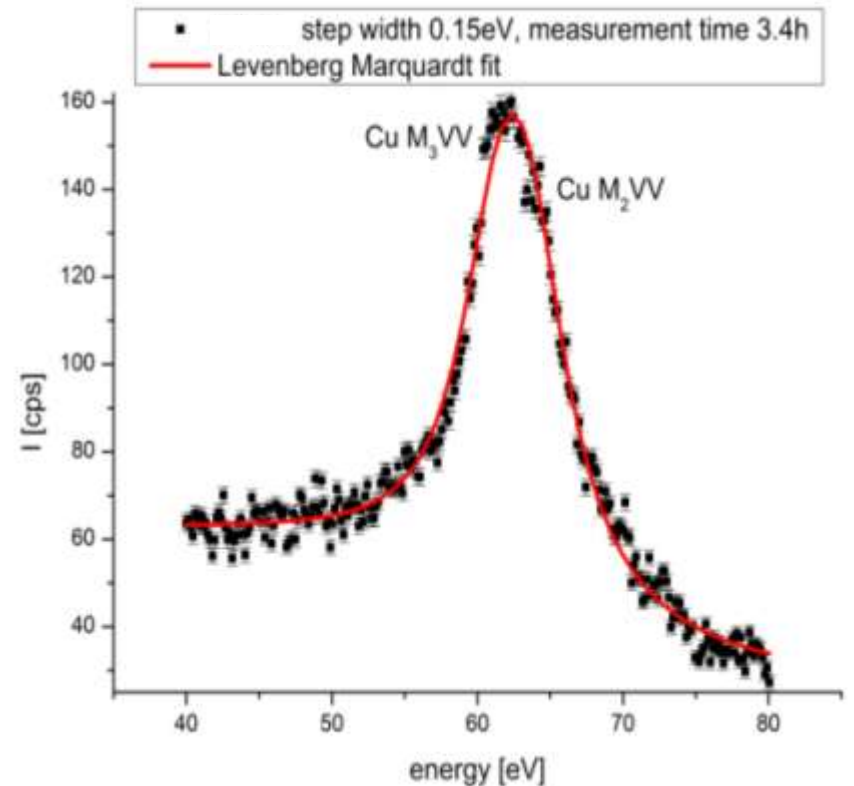


e^+ induced emission of Auger e^- :

- e^+ diffusion
- e^+ trapping in surface state
- annihilation with core e^-
- emission of Auger e^-

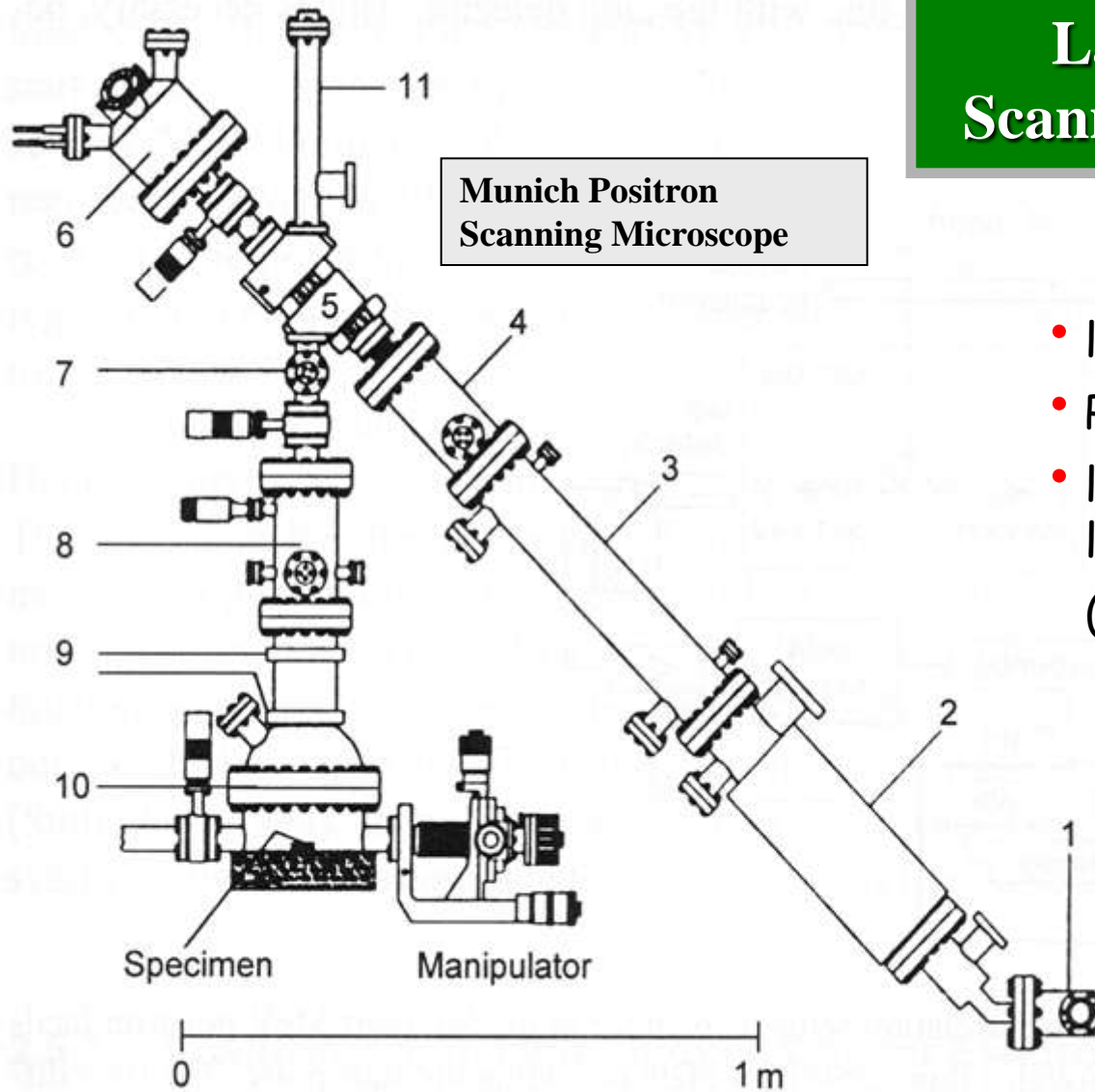
Features **PAES**:

- no e^-_{sec} background
- non-destructive
- topmost atomic layer



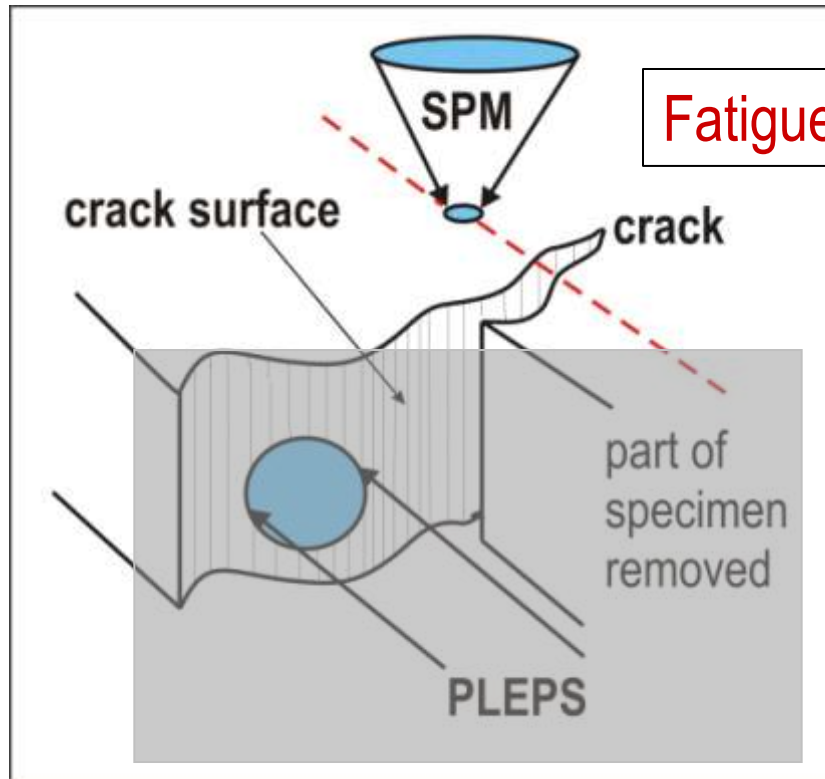
Lateral Resolution with Scanning Positron Microscope

Munich Positron Scanning Microscope



- lateral resolution 1...2 μm
- Positron lifetime spectroscopy
- lateral resolution principally limited by positron diffusion ($L_+ \approx 100\text{nm}$)

SPM on top of cracked sample

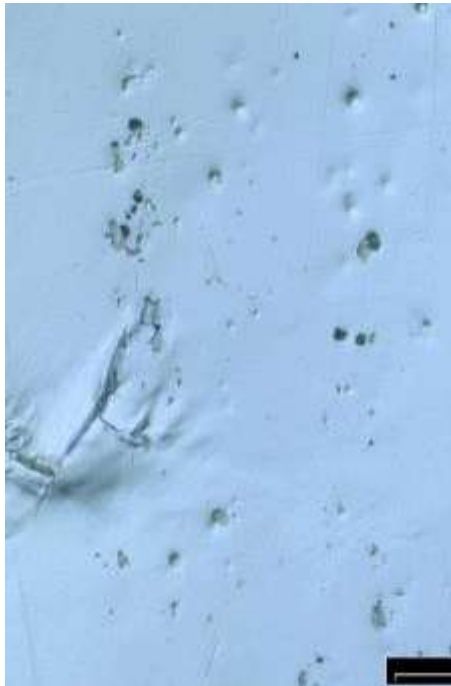


Fatigue-Crack in Al 6013

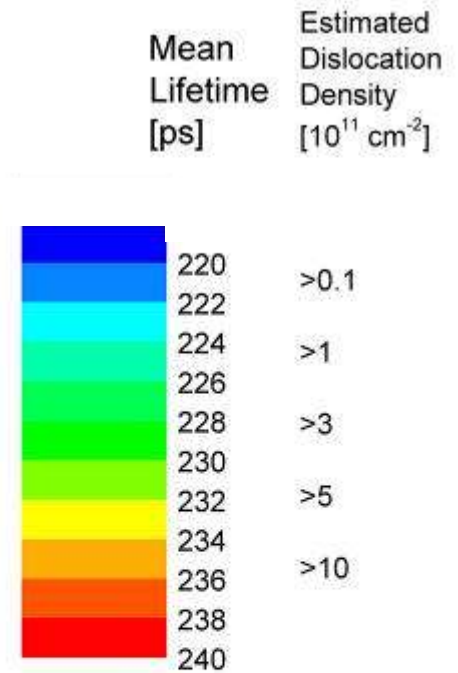
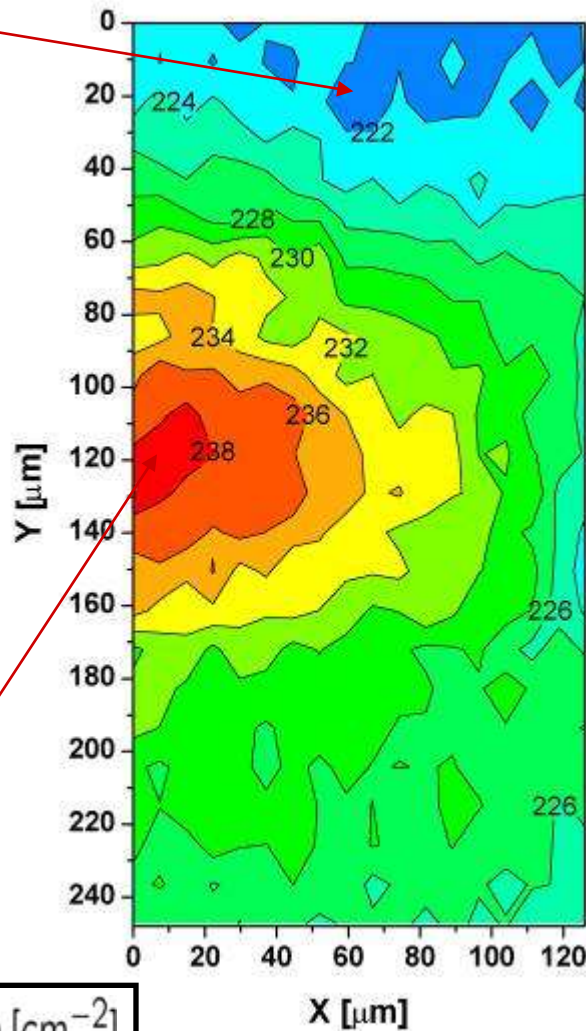
SPM: Lifetime-Image of Fatigue-Crack in Al 6013

resolution 5 μm ; step-size 10 μm

Trapping at Mg / Si-clusters!



Only **dislocations** close to crack-tip !



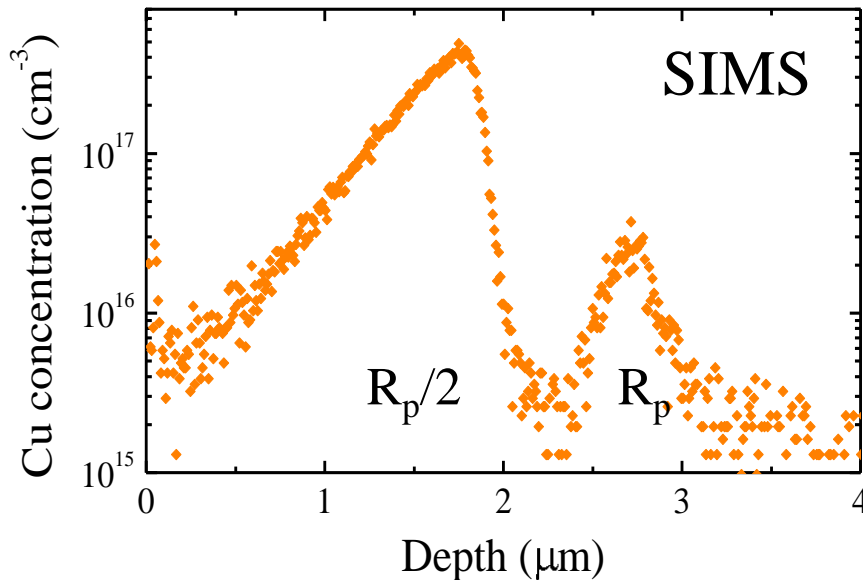
$$c_{disl} = 4 \cdot 10^{11} (\tau - 220 \text{ ps}) / (240 \text{ ps} - \tau) [\text{cm}^{-2}]$$

W. Egger, G. Kögel,
P. Sperr, W. Triftshäuser,
J. Bär, S. Rödling, H.-J.
Gudladt
Mater. Sci. Eng. (A)
387- 398 (2004) 317

Defects in high-energy self-implanted Si: The $R_p/2$ effect

- after high-energy (3.5 MeV) self-implantation of Si ($5 \times 10^{15} \text{ cm}^{-2}$) and RTA annealing (900°C , 30s): two new gettering zones appear at R_p and $R_p/2$ (R_p = projected range of Si^+)
- visible by SIMS profiling after intentional Cu contamination

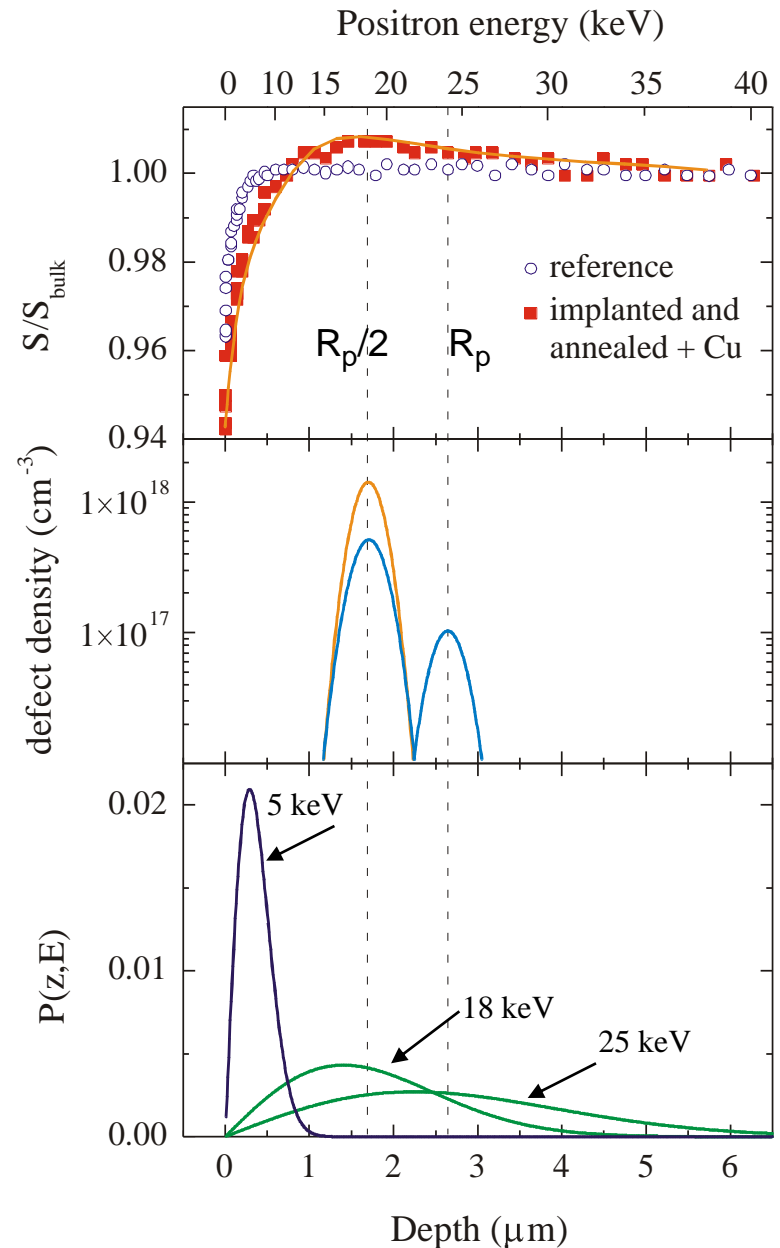
TEM image by P. Werner, MPI Halle



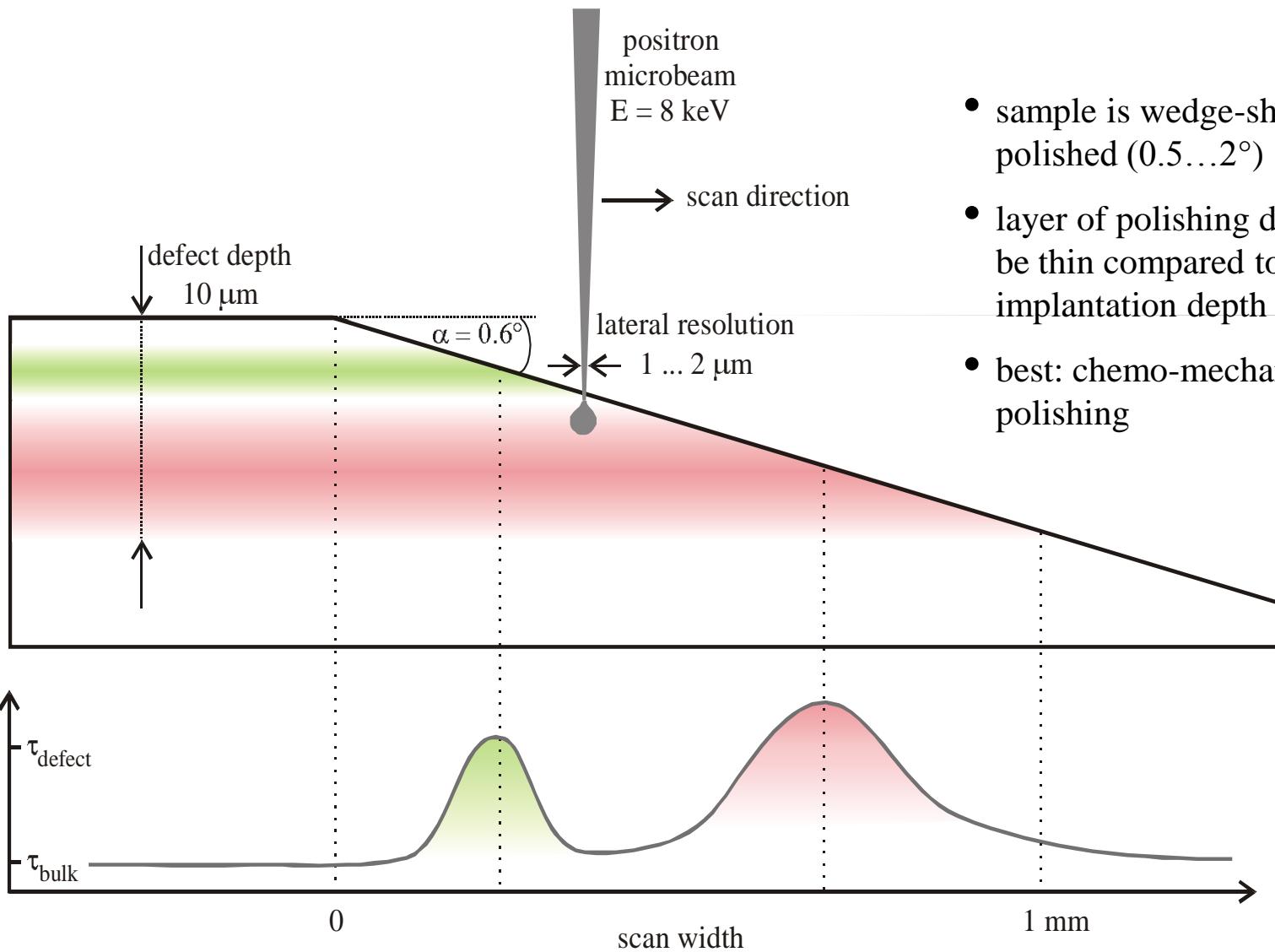
- at R_p : gettering by interstitial-type dislocation loops (formed by excess interstitials during RTA)
- no defects visible by TEM at $R_p/2$
- What type are these defects?

Investigation of the $R_p/2$ effect by conventional VEPAS

- the defect layers are expected in a depth of 1.7 μm and 2.8 μm corresponding to $E^+ = 18$ and 25 keV
- implantation profile too broad to discriminate between the two zones
- simulation of $S(E)$ curve gives the same result for assumed blue and yellow defect profile (solid line in upper panel)
- furthermore: small effect only
- no conclusions about origin of $R_p/2$ effect possible



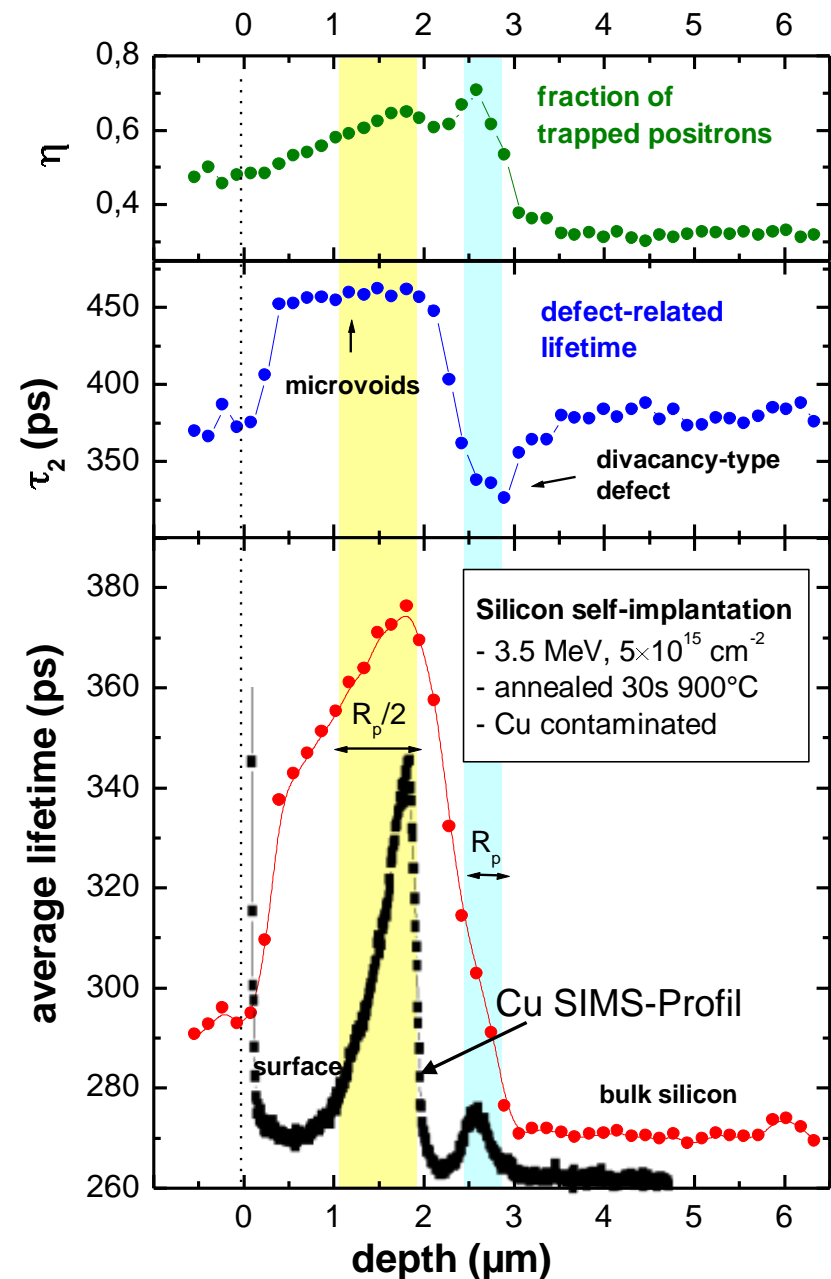
Enhanced depth resolution by using the Positron Microscope



- sample is wedge-shaped polished ($0.5 \dots 2^\circ$)
- layer of polishing defects must be thin compared to e^+ implantation depth
- best: chemo-mechanical polishing

First defect depth profile using Positron Microscopy

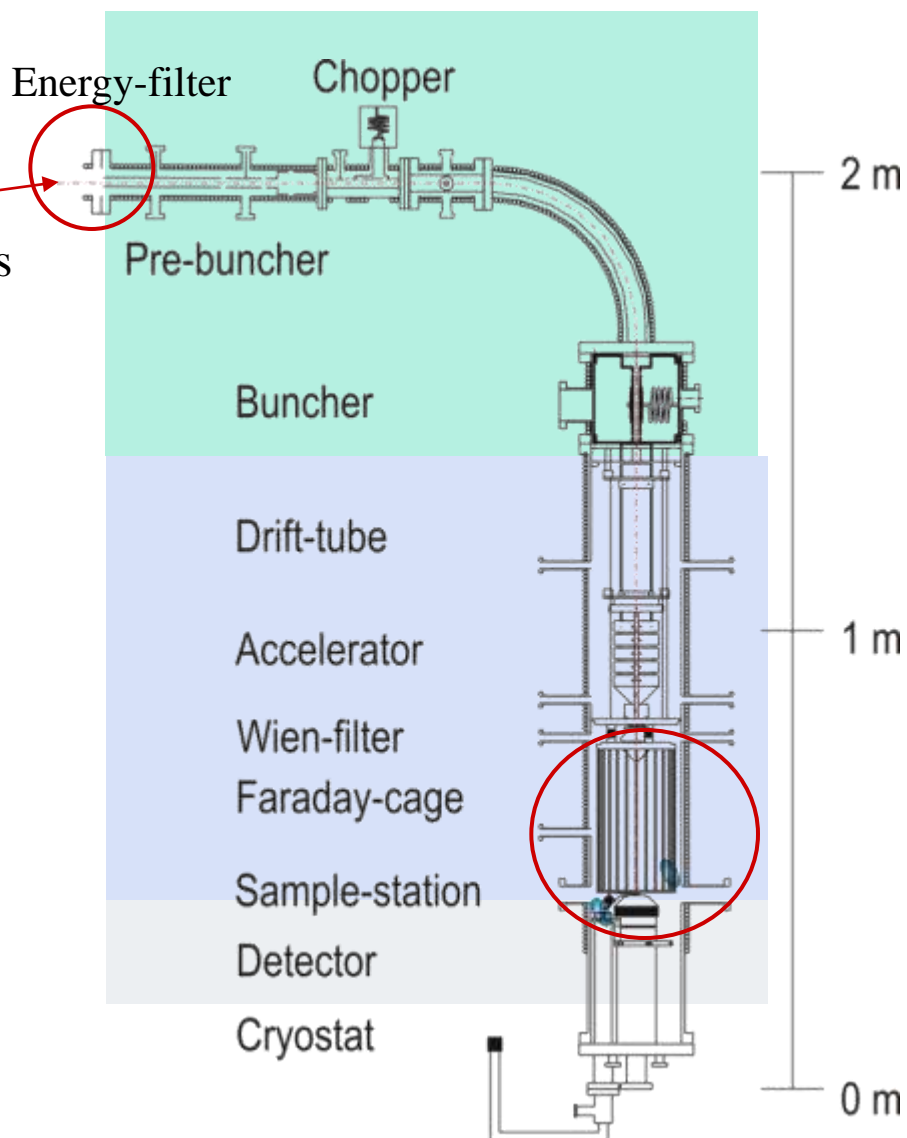
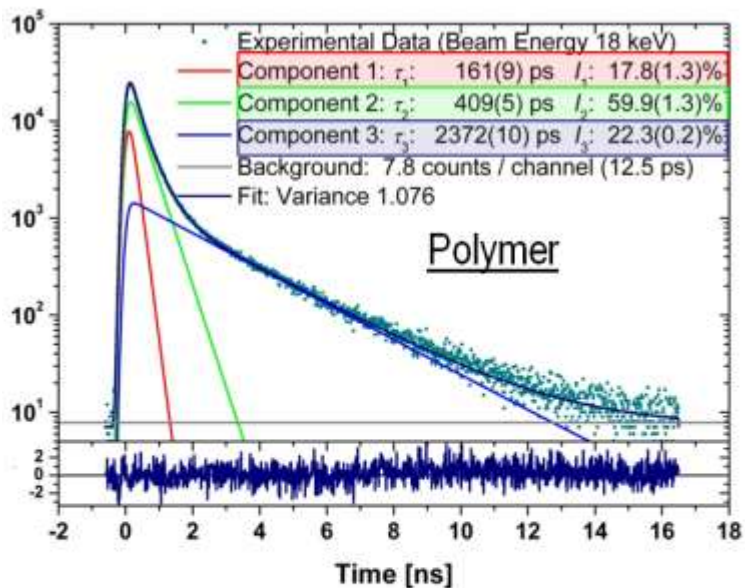
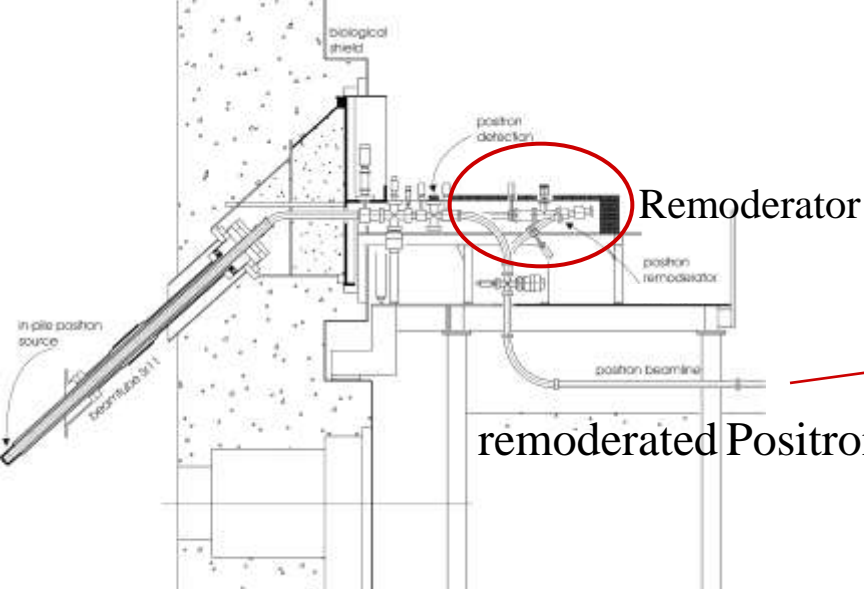
- 45 lifetime spectra: scan along wedge
- separation of 11 μm between two measurements corresponds to depth difference of 155 nm ($\alpha = 0.81^\circ$)
- beam energy of 8 keV: mean penetration depth is about 400 nm; represents optimum depth resolution
- no further improvement possible due to positron diffusion: $L_+(\text{Si @ 300K}) \gg 230 \text{ nm}$
- both regions well visible:
 - vacancy clusters with increasing density down to 2 μm ($R_p/2$ region)
 - in R_p region: lifetime $\tau_2 = 330 \text{ ps}$; corresponds to open volume of a divacancy; must be stabilized or being part of interstitial-type dislocation loops
- excellent agreement with gettered Cu profile



R. Krause-Rehberg et al., Appl. Phys. Lett. 77 (2000) 3932



PLEPS at NEPOMUC



- Measurement-time at 5000 cps:
600 s / spectrum ($> 3 \times 10^6$ counts)
- 3h-4h / depth -profile (15 spectra)
- up to 4 lifetimes resolvable

PLEPS at NEPOMUC



NEPOMUC user since June 2008

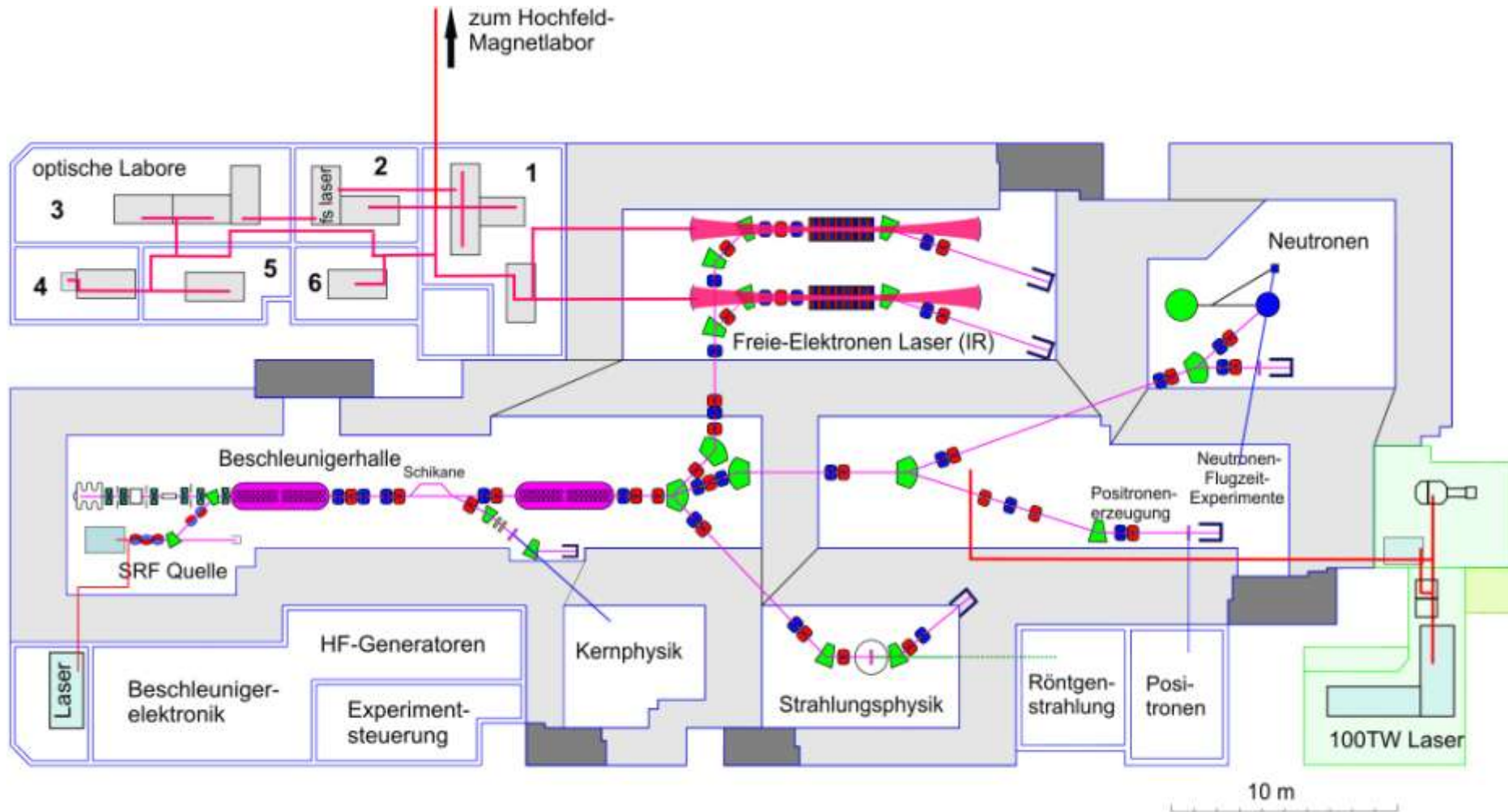
Z. Kajczos	KFK Budapest	Al ₂ O ₃
V. Slugen	TU Bratislava	Fe-Cr-Legierungen
R. Brusa	Universita` di Trento	SiO ₂ , Mg-Nb (H)
J. De Baerdemaeker	Universität Ghent	Ge, Polymere
A. Gentils	CSNSM CNRS	SiC
M.-F. Barthe	CERI Orleans	W
D. Keeble	University of Dundee	SrTiO ₃ , Polymere
J. Cizek	Universität Prag	ZnO
F. Tuomisto	HUT Helsinki	InGaN
S. Eijt	TU Delft	Mg-Ti (H)
A. Dupasquier	Politecnico Milano	STO/YSZ/STO
O. Moutanabirr	MPI Halle	AlN
R. Krause-Rehberg	Universität Halle	GaN
H.J.-Gudladt	UniBw München	Ferrit. / Austen. Stähle
P. Iskra	UniBw München	Si
G. Brauer	FZR Rossendorf	ZnO
A. Ulbricht	FZR Rossendorf	Fe-Cr-Legierungen
K. Schmidt	IPP München	W
K. Rätzke	TU Kiel	Polymere
M. Krause	GSI	Graphit
A. Nix	Universität Göttingen	ta-C

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

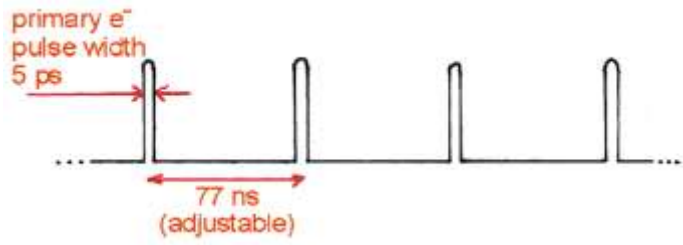
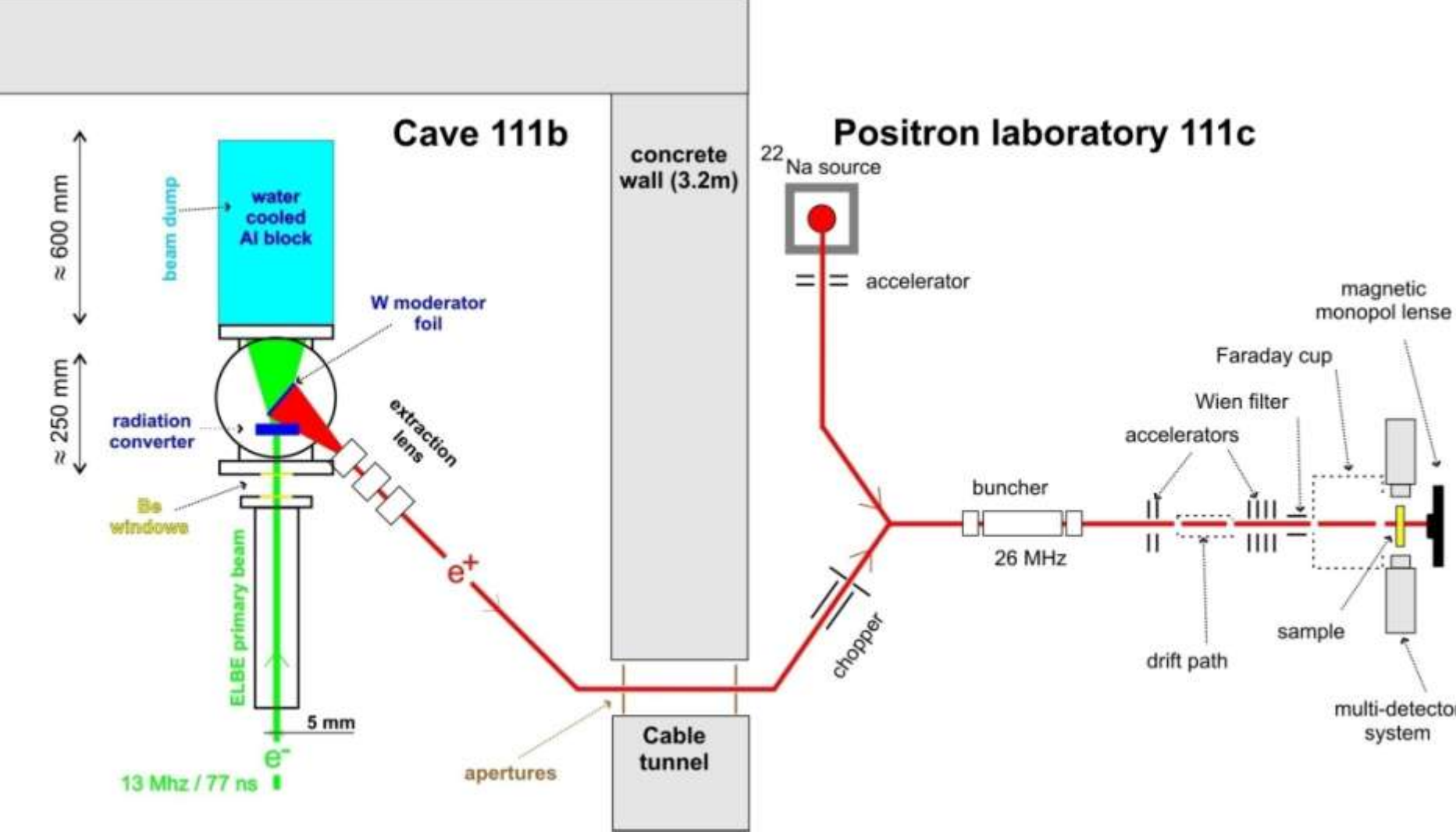


Ground plan of the ELBE hall



1: Diagnosestation, IR-Imaging und biologische IR Experimente
 2: Femtosekundenlaser, THz-Spektroskopie, IR Pump-Probe Experimente
 3: Zeitaufgelöste Halbleiter-Spektroskopie, THz-Spektroskopie

4: FTIR, biologische IR Experimente
 5: Nahfeld und Pump-Probe IR Experimente
 6: Radiochemie und Summenfrequenz-Erzeugung, photothermische Spektroskopie



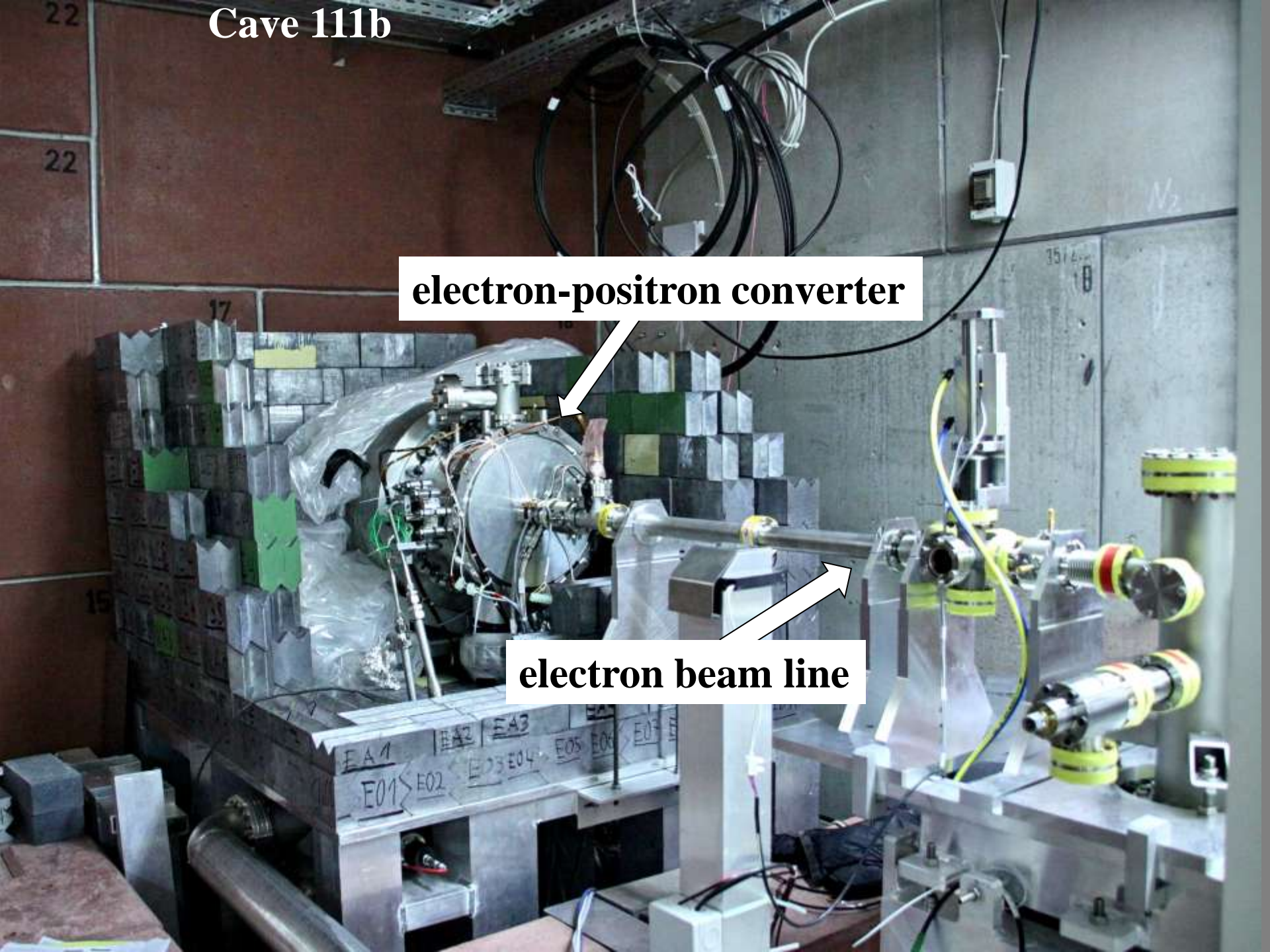
EPOS scheme



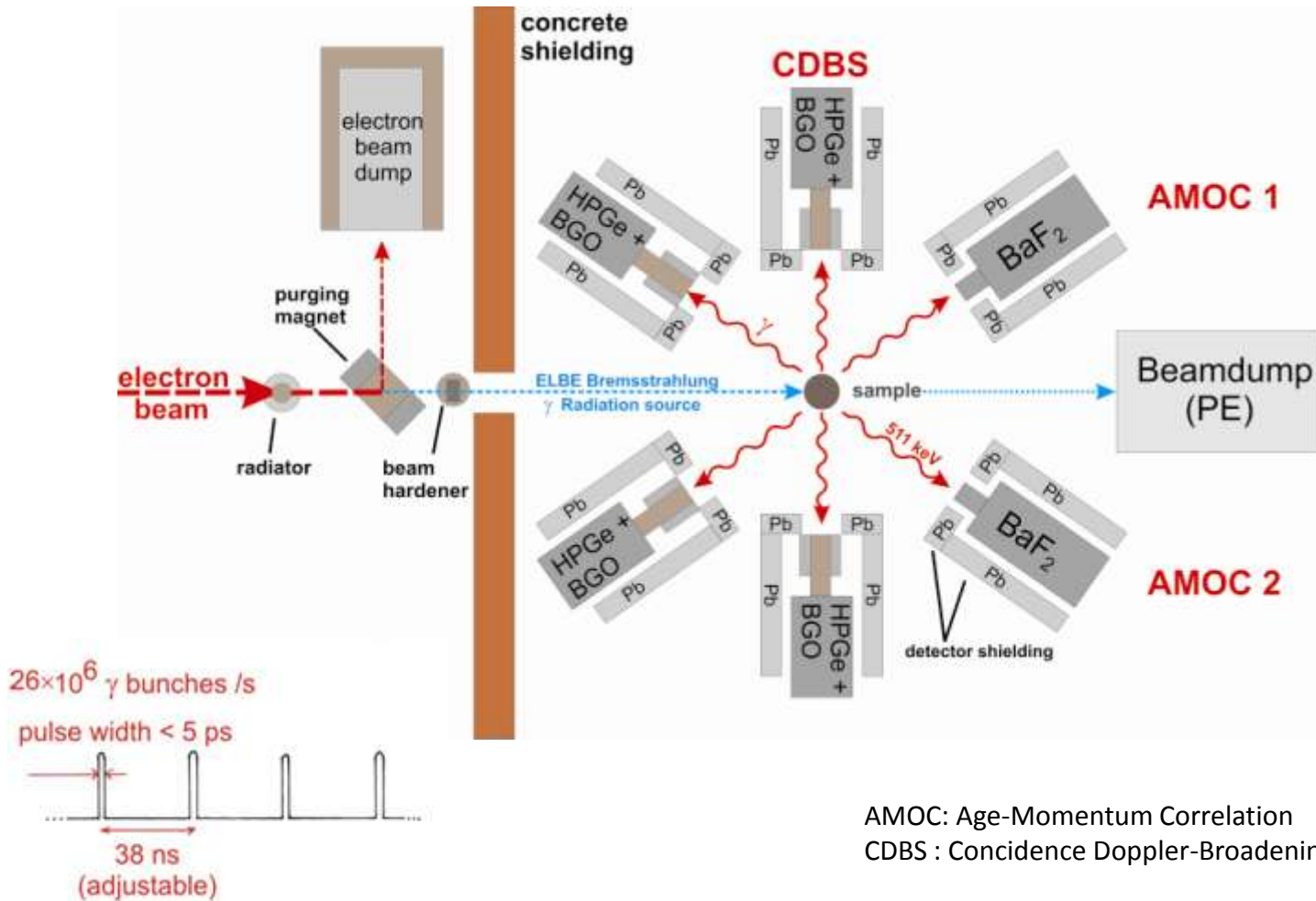
Cave 111b

electron-positron converter

electron beam line



GiPS: Gamma-induced Positron Spectroscopy

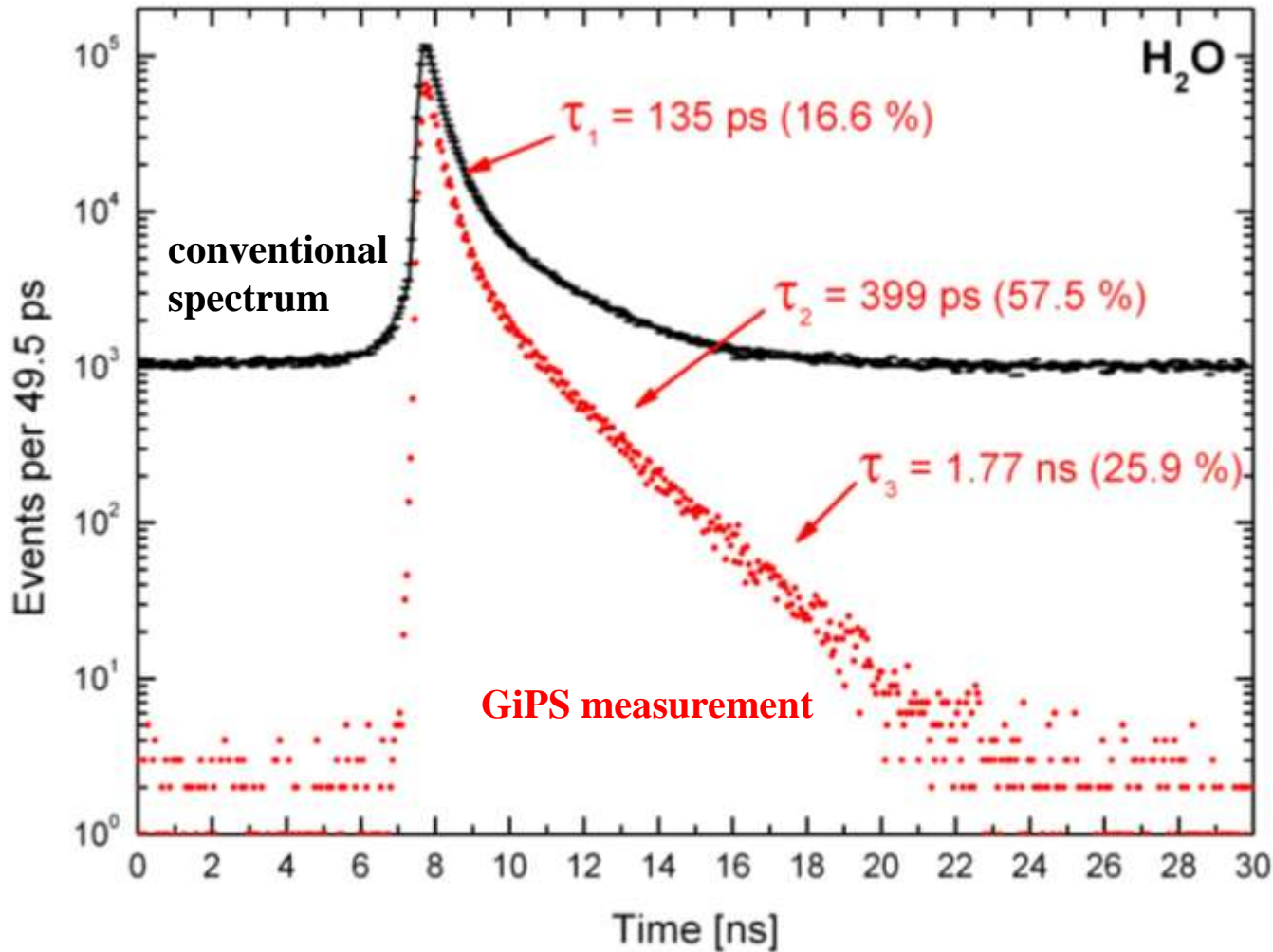


AMOC: Age-Momentum Correlation
CDBS : Coincidence Doppler-Broadening Spectroscopy

- 3 coincident setups were used: 2 AMOC and 1 CDBS spectrometer
- only coincident detection ensures high spectra quality

Example: Water at RT

- total count rate in spectrum: 12×10^6



- Black spectrum: conventional measurement by Kotera et al., Phys. Lett. A 345, (2005) 184

Applications of GiPS since begin of 2009

- neutron irradiated Fe-Cr alloys (highly activated up to 50 MBq ^{60}Co)
- Reactor pressure vessel steel samples from Greifswald nuclear power station
- Iron samples after mechanical damage (LCMTR-ISCSA-CNRS, Frankreich)
- set of Zircony alloys (Collaboration Mumbai/India)
- porous glass (Chem. Department/Univ. Leipzig)
- biological samples
- liquids

Positron Annihilation Spectroscopy: Applications

Variety of applications in all fields of materials science:

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

This presentation can be found as pdf-file on our Website:
<http://positron.physik.uni-halle.de>

