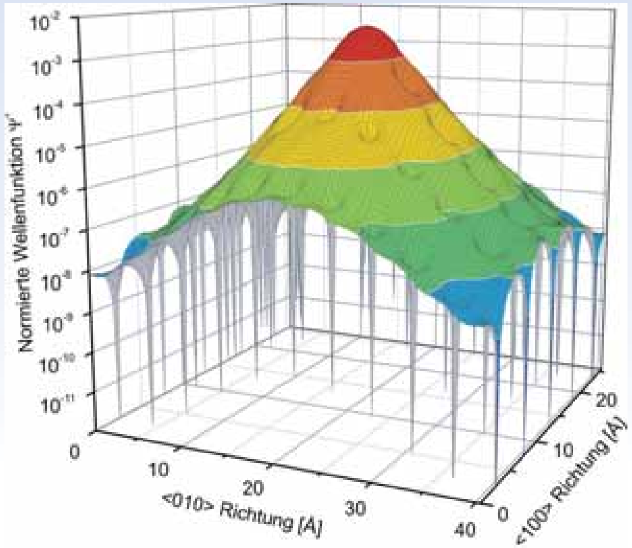
Positron Probe Microanalysis (PPMA) – Prospects and Challenges –

Torsten Staab, Matz Haaks, Karl Maier

University Bonn



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Introduction and Outline

- Electron Probe Micro Analysis (EPMA):
  - Micro beam instrument: “spot” size 1-2µm (= information volume)
    Imaging modes: SE-electrons, BS-electrons, CL
  - In-situ non-destructive chemical analysis - locally detecting elements (characteristic X-rays → EDS- / WDS-analysis)
  - Main feature: acquiring 2D-

2D-elemental maps

- Positron Probe Micro Analysis (PPMA): developed in Bonn 1994 - 1997
  - Other instruments: Munich (next talk) & Japan (developments)
  - Similar “spot” as EPMA possible → similar information volume
  - Similar sample preparation – BUT: No coating necessary (tiny current)
  - Mapping of lattice defects: vacancies, dislocations, grain boundaries, ..

2D-lattice defect maps

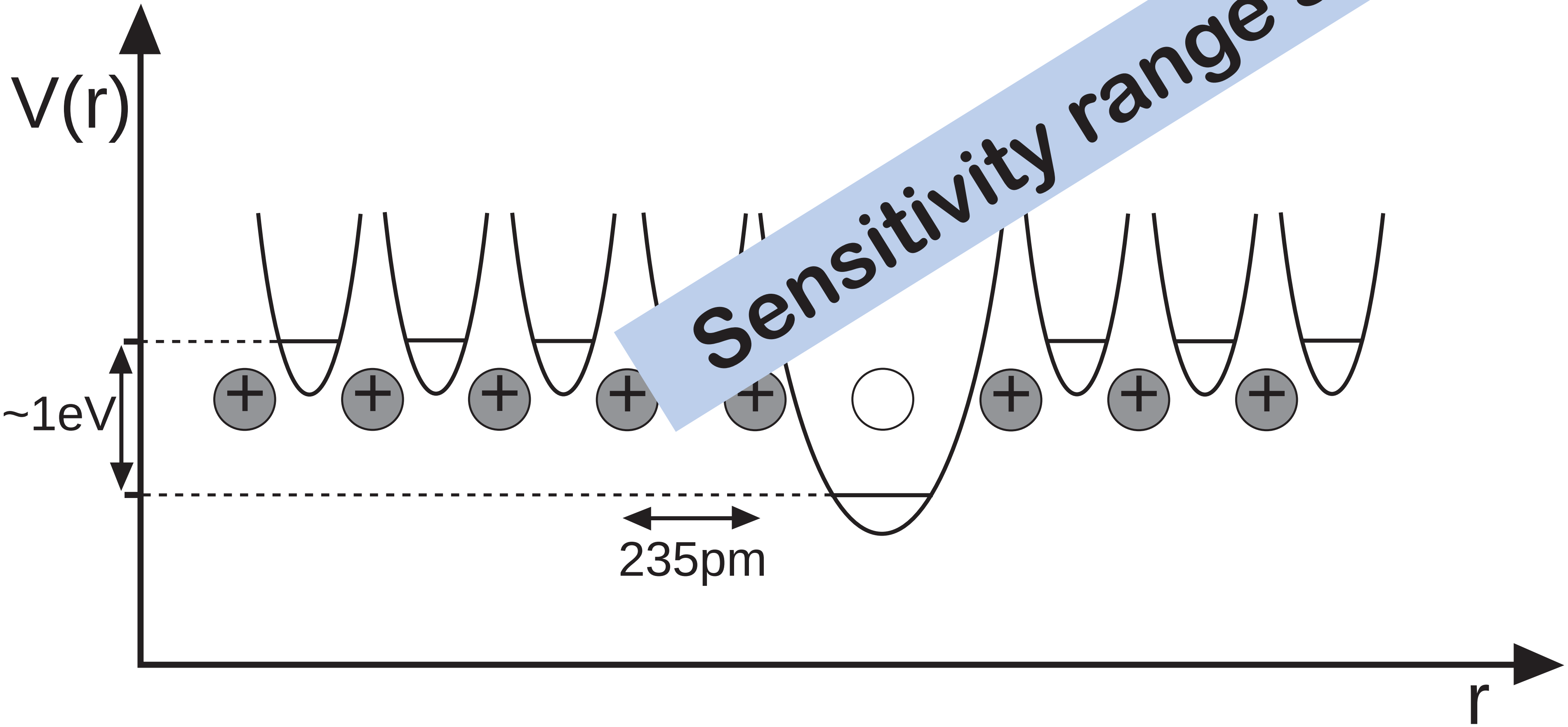
- Applications of PPMA:
  - Examples from metals and alloys, semiconductors
  - Complementary methods: EXAFS or SAXS

Supplementary information

Potential sensed by a Positron in a Vacancy

- Positron's sensitivity to vacancies and vacancy-like defects in metals and semiconductors: 1×10^{15} ... 1×10^{19} cm^{-3} and to dislocations in metals: 5×10^{8} ... 1×10^{11} cm^{-2}

Sensitivity range should fit



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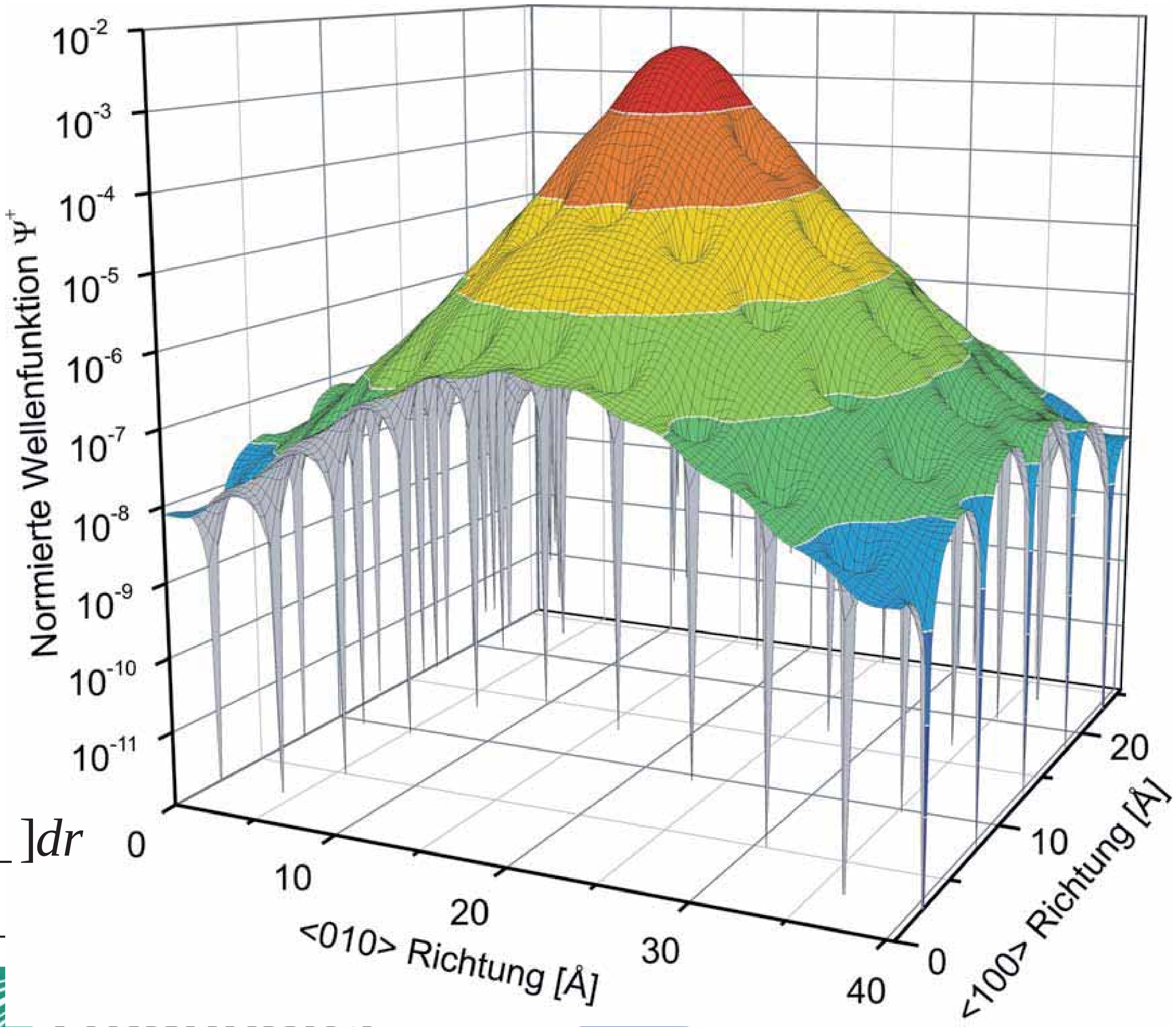
„Picture“ of a Positron in a Vacancy

- **Positron density at a vacancy in aluminum obtained by ab-initio calulations**



- **Annihilation rate**

$$1/\tau = \lambda = \pi r_0^2 c \int n_+(r)\, n_-(r)\, g[n_-]\, dr$$

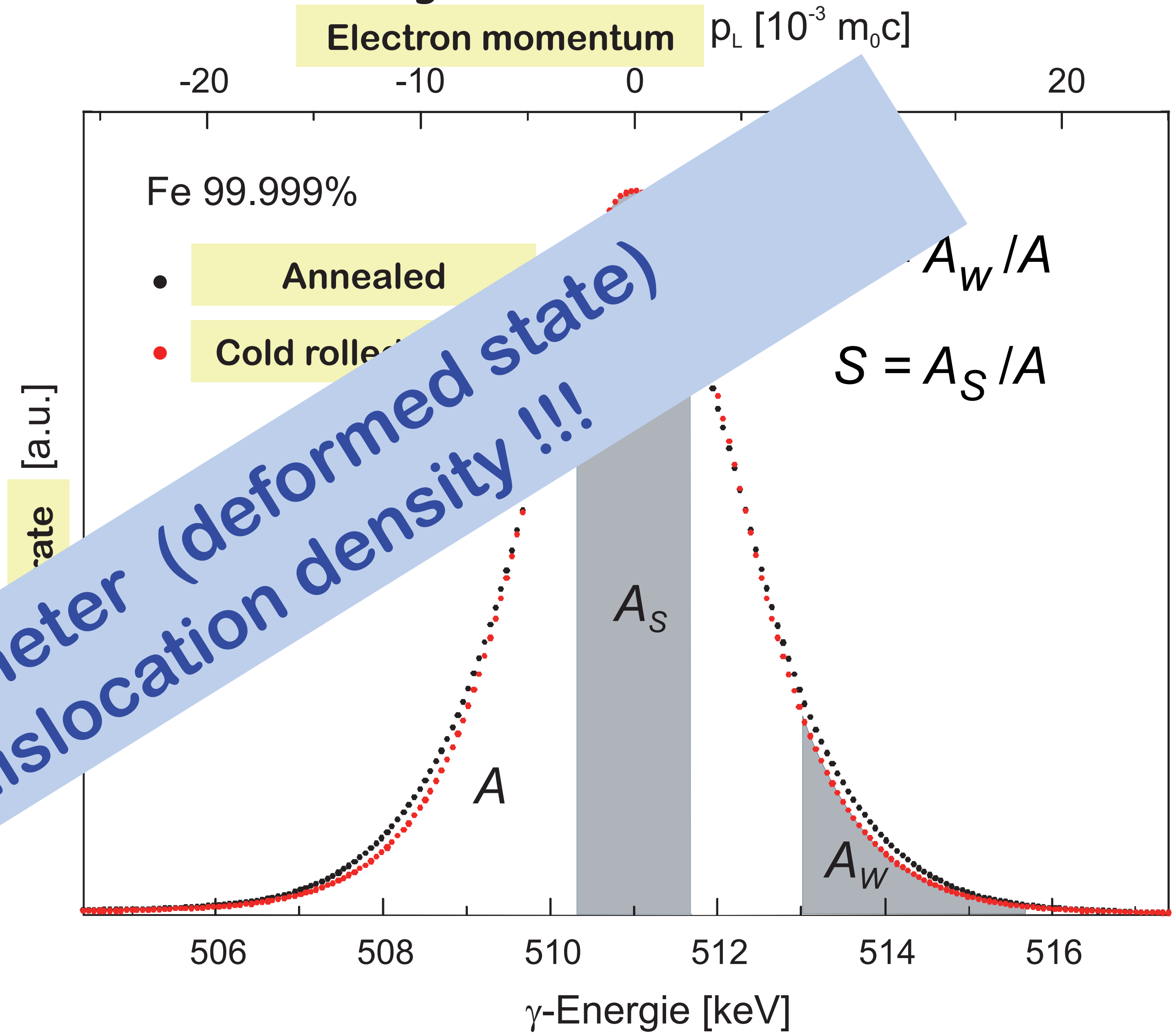
Source: Björn Korff– Dissertation Bonn 2010

Identification of Lattice Defects by e^--Momentum

- Momentum distribution of electrons in a solid:
  S- and W-parameter
- Annihilation of electron and positron:
  2 x 511keV quanta
- Measurement via a High-Purity Germanium-Detektor

$$S = A_S / A$$

$$W = \ldots$$



High S-Parameter (deformed state)
= high dislocation density !!!

Source. … Bonn 2009

1st Application of PPMA

Plastic Zones in front of Crack Tips

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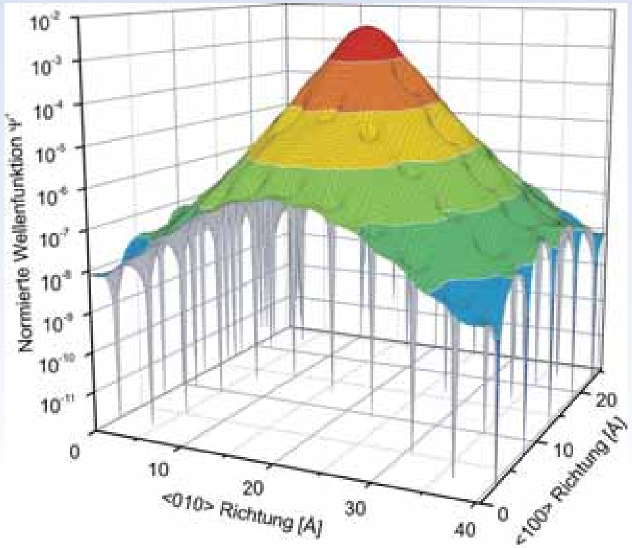

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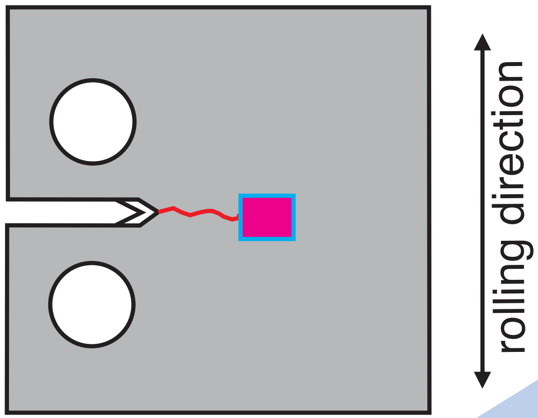
Typical Applications of a Positron-Microprobe

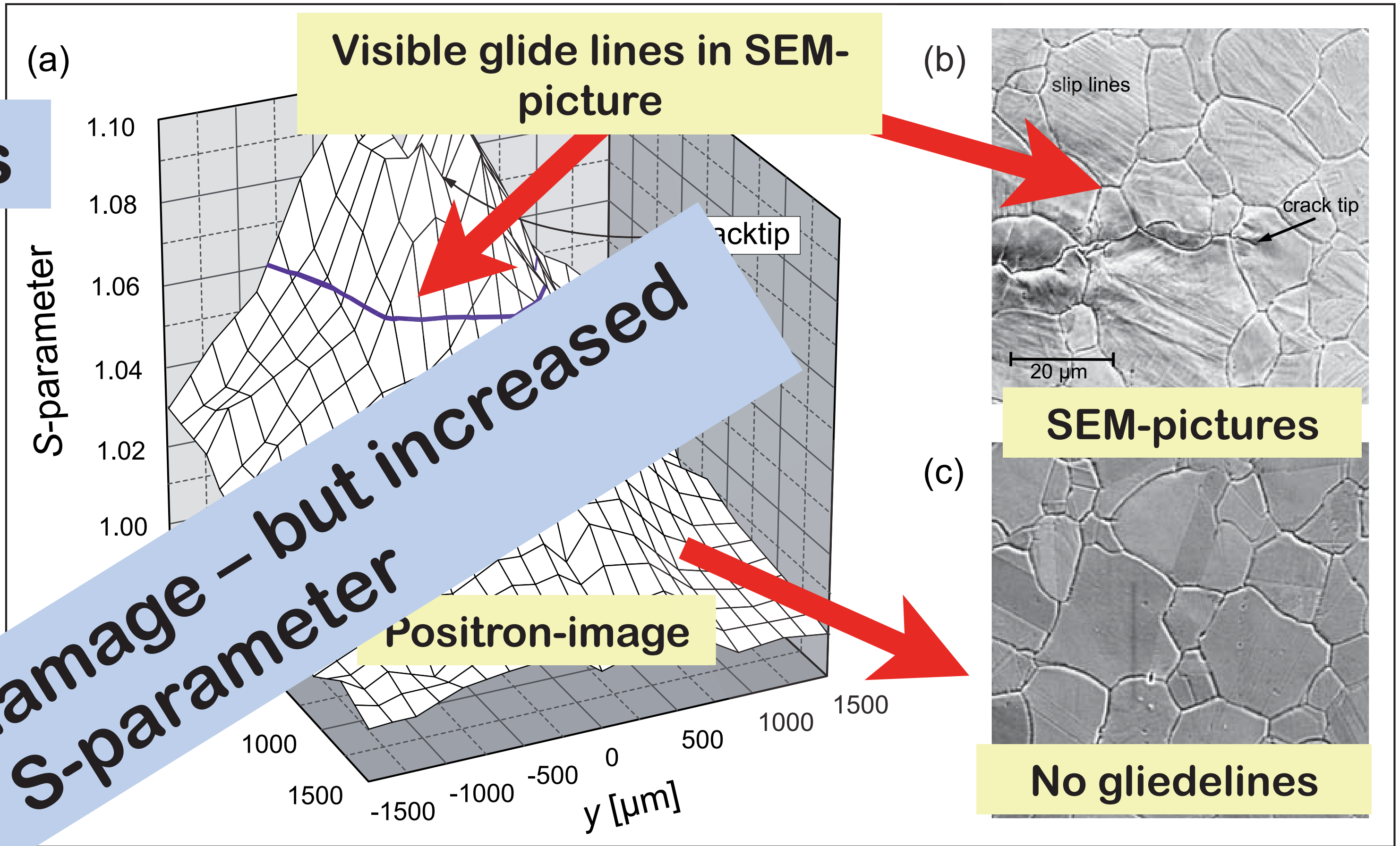
- Deformation / fatigue in metallic materials
- First image of a plastic zone in front of a cracktip: stainless steel AISI 321
  - 1997/1998

S=1 – NO defects

Cracking: experiment



scaled CT-geometr



Visible glide lines in SEM-picture

Positron-image

SEM-pictures

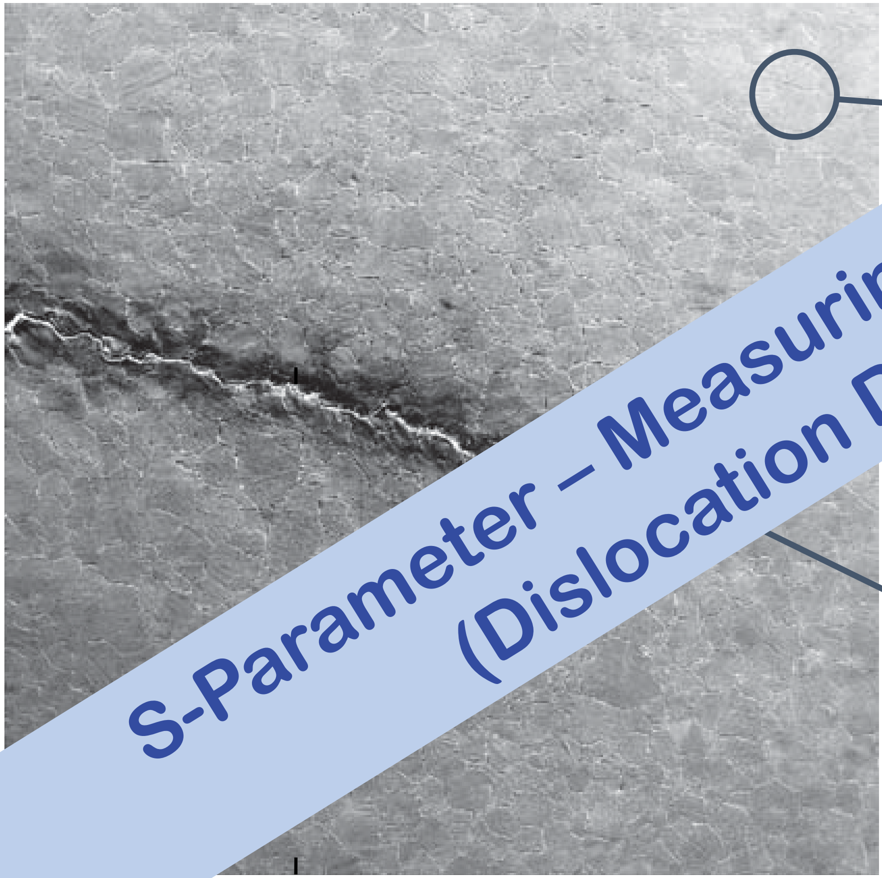
No gliedelines

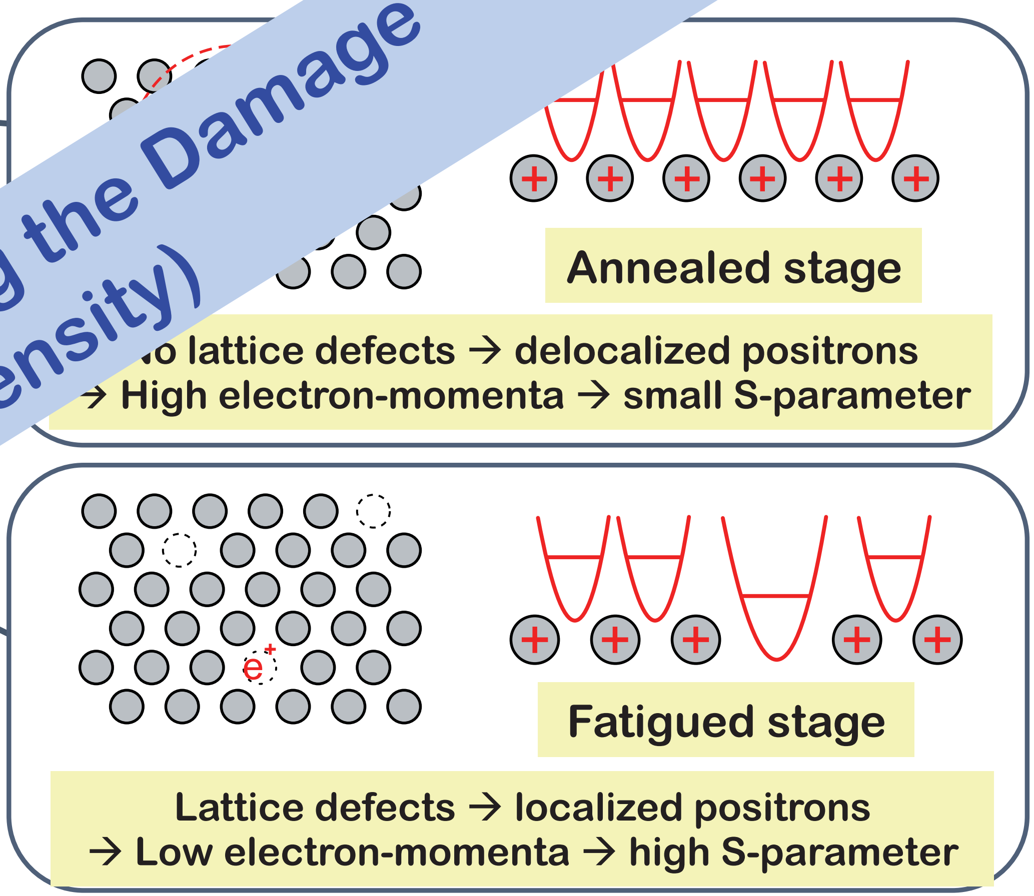
No visible damage – but increased S-parameter

<u>Bildquellen:</u> Matz Haaks – Dissertation Bonn 2009

Detection of lattice defects after/during fatigue

- Positron Annihilation: signals from vacancy-like latti



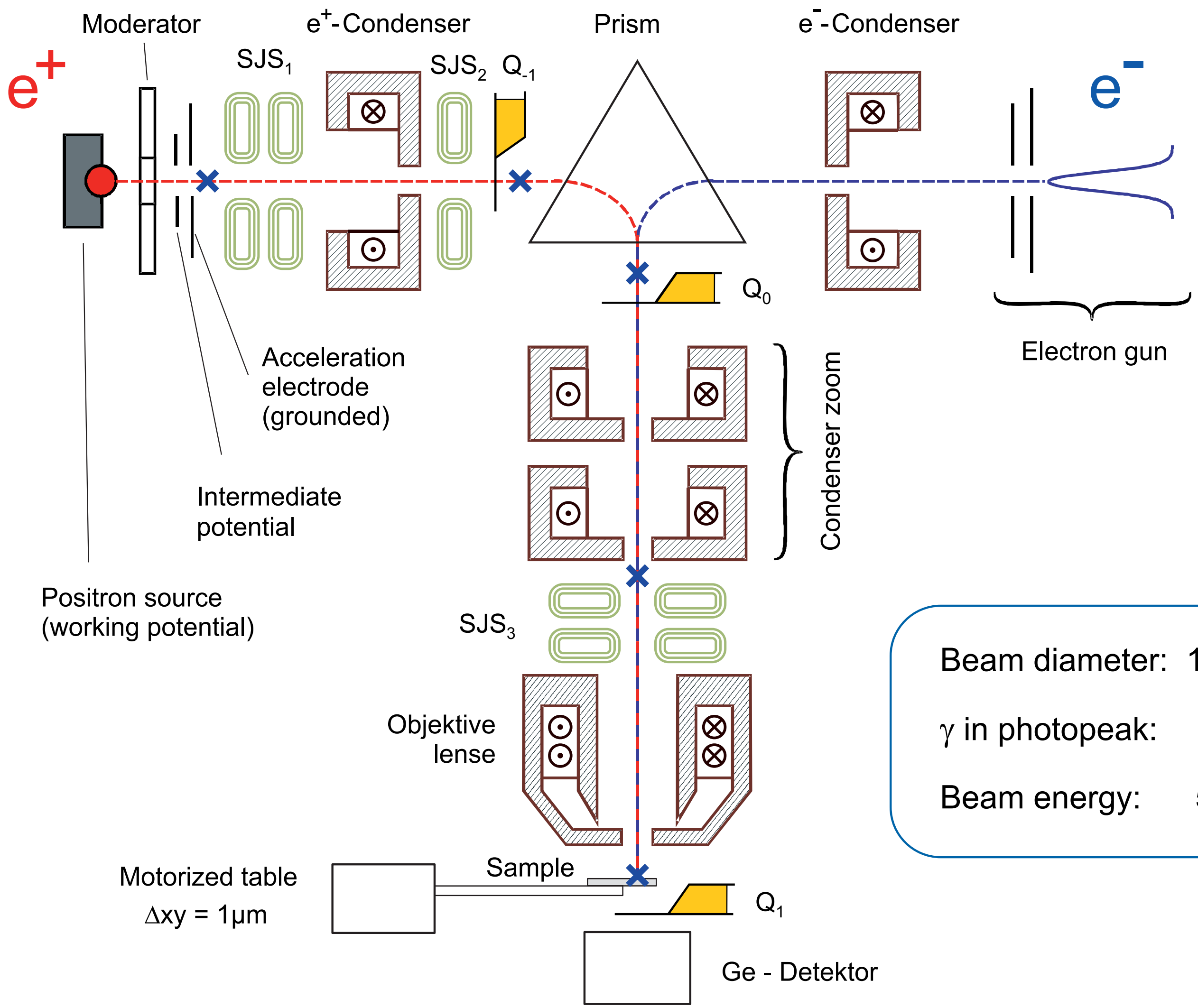


S-Parameter – Measuring the Damage (Dislocation Density)

tz Haaks – Habilitation Bonn 2009

Design of a Positron Microprobe: Modified SEM

- **Set-up:**
  - 1994 – 1997
  - Zeiss-Leo – Oberkochem (J. Bihr, B. Huber) &
  - Group of Prof. Karl Maier (University Bonn)

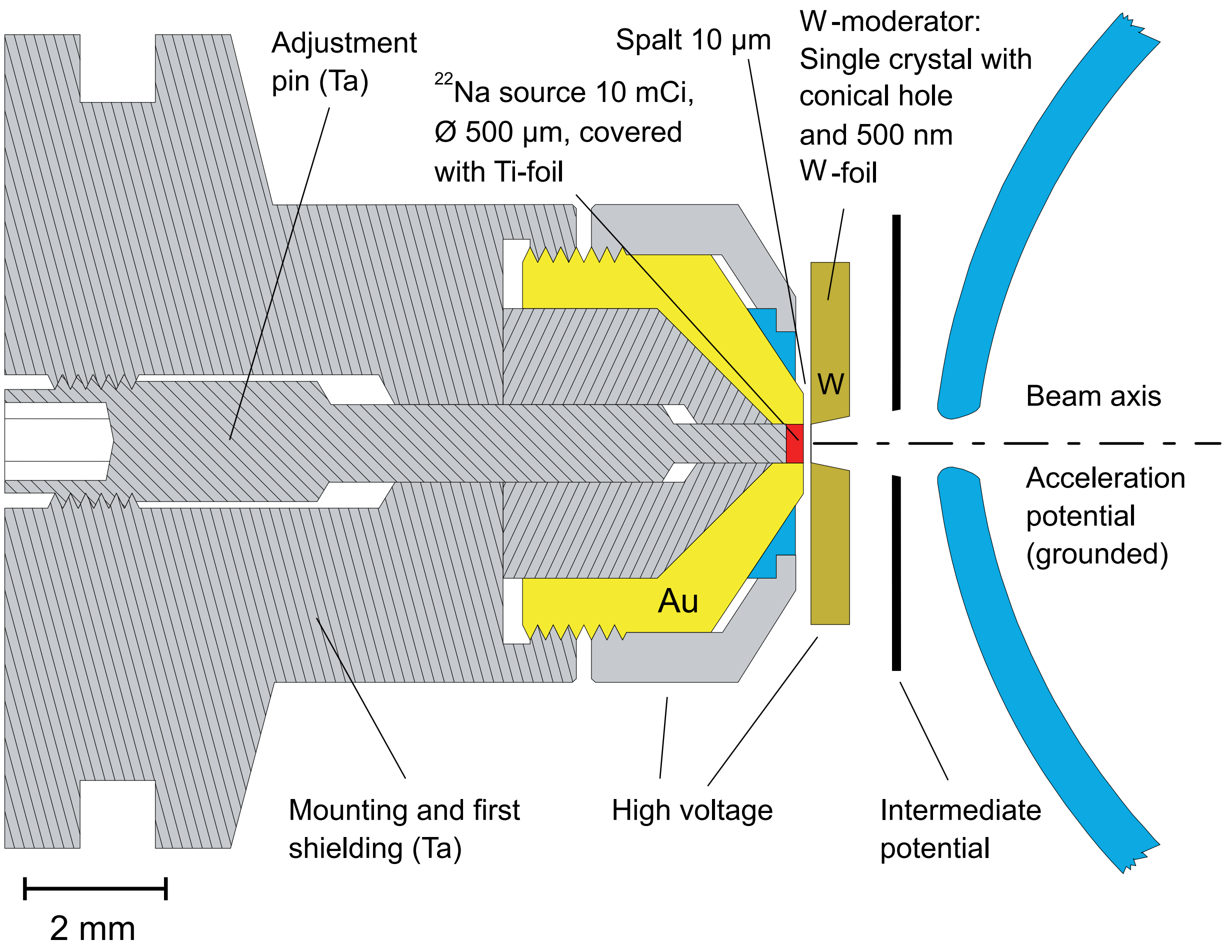


Beam diameter:	1 - 200 µm
γ in photopeak:	1000 cps
Beam energy:	5 - 30 keV

Source: Matz Haaks – Habilitation Bonn 2009

Positron-Microprobe: Finely Focussed Positron Beam

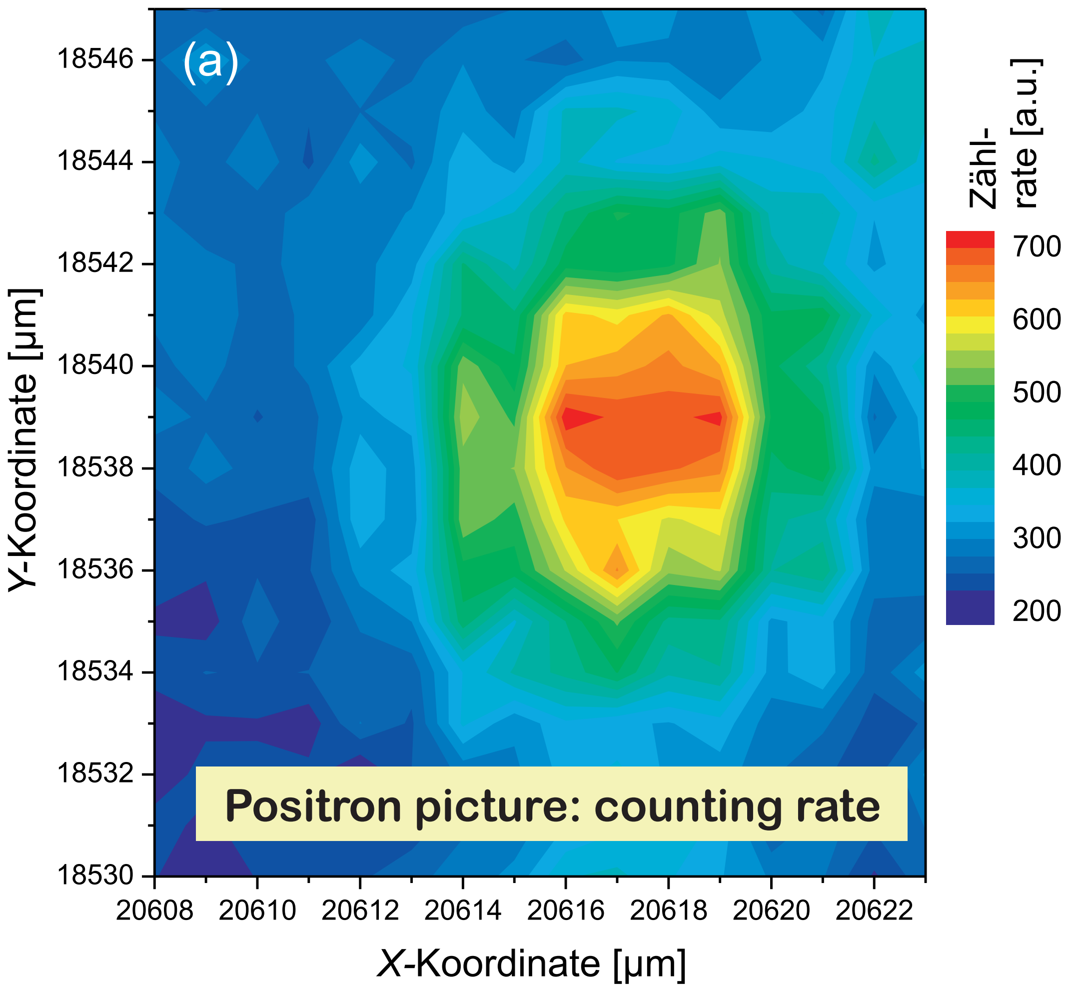
- **Positron source with new constructed moderator: since 2003**

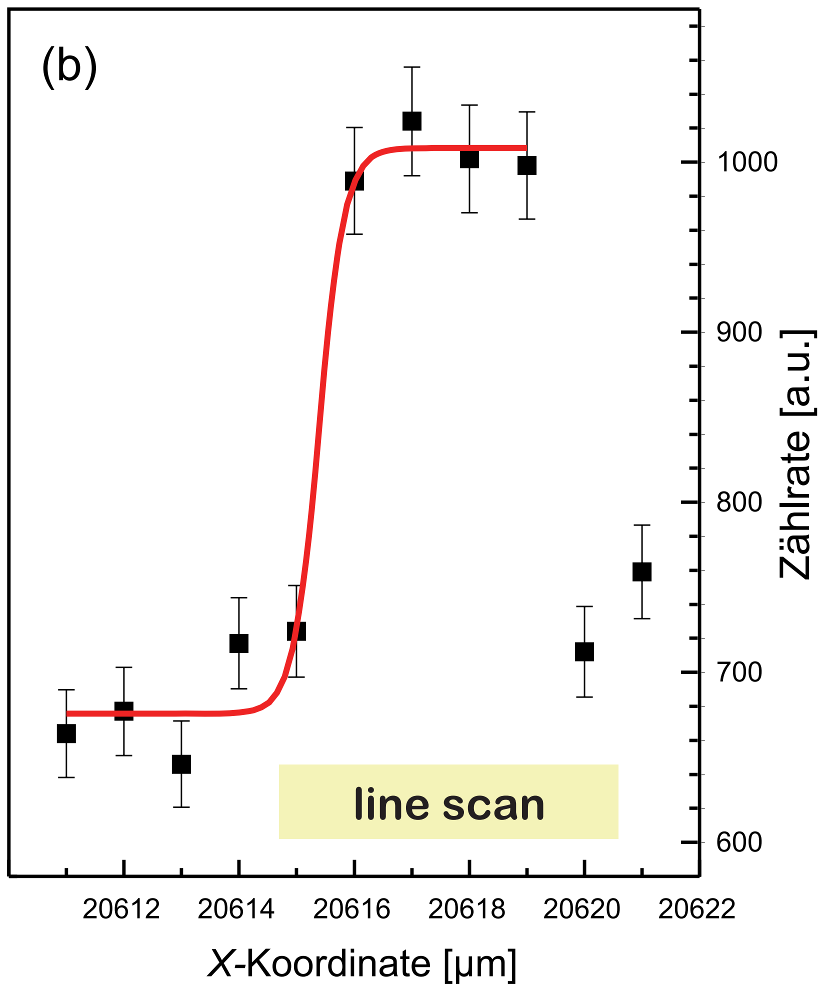


<u>Source:</u> Matz Haaks – Habilitation Bonn 2009

Positron Microprobe: Lateral Resolution around 1µm

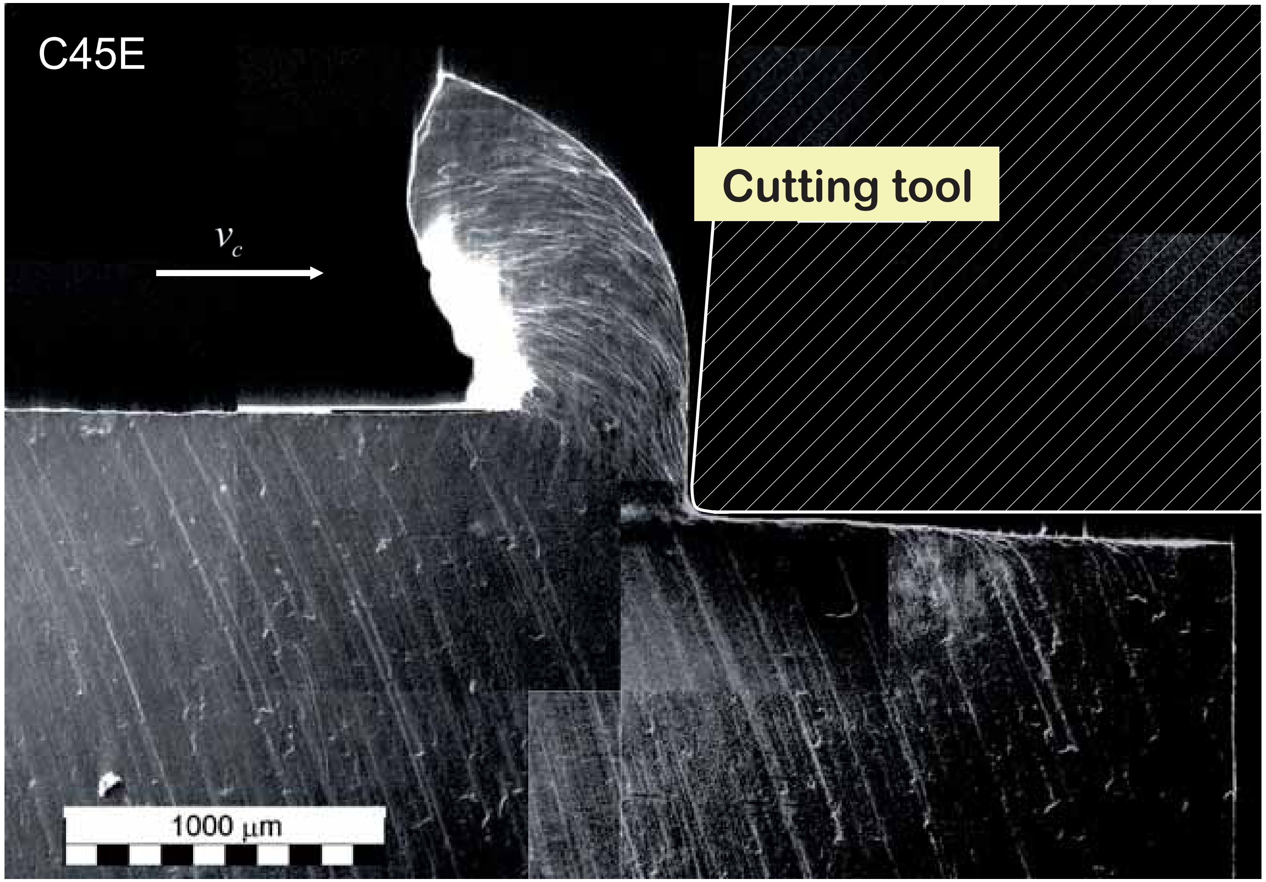
- **Picture of a 5µm hole in a gold foil**





Application: High-Speed-Cutting

- **Depth of damage / recovery: cooperation with University Hannover 2003**



SEM picture

Electron micrograph

Inst. f. Fertigungstechnik u. Werkzeugmaschinen, Garbsen

Application: High-Speed-Cutting

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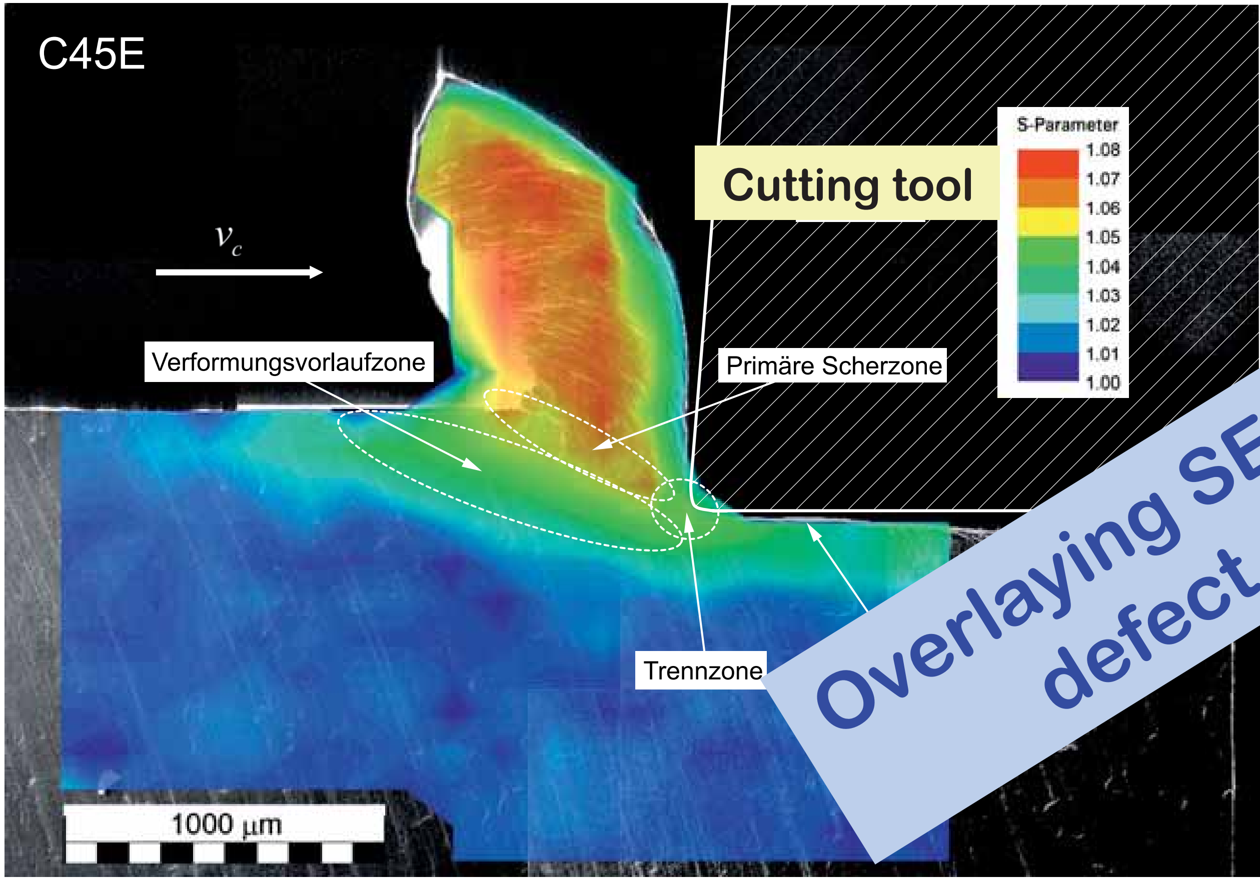

Overlaying SEM-picture & defect mapping

…e & positrons

Scanning positron image

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2^{nd} Application of PPMA
Scratches on a GaAs-Wafer
Making Damage Visible

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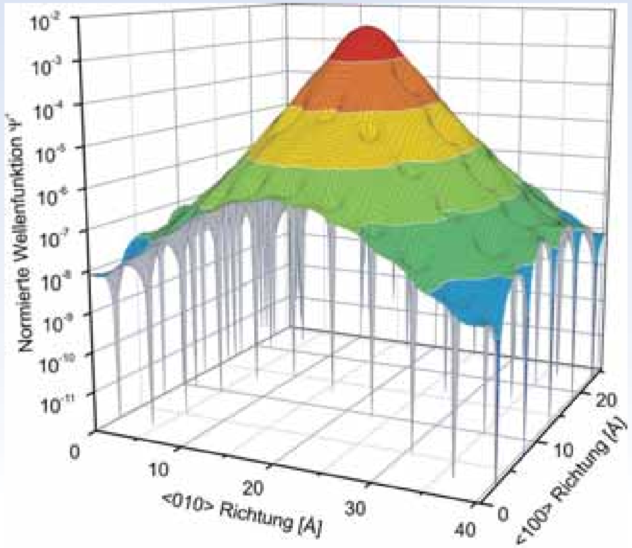

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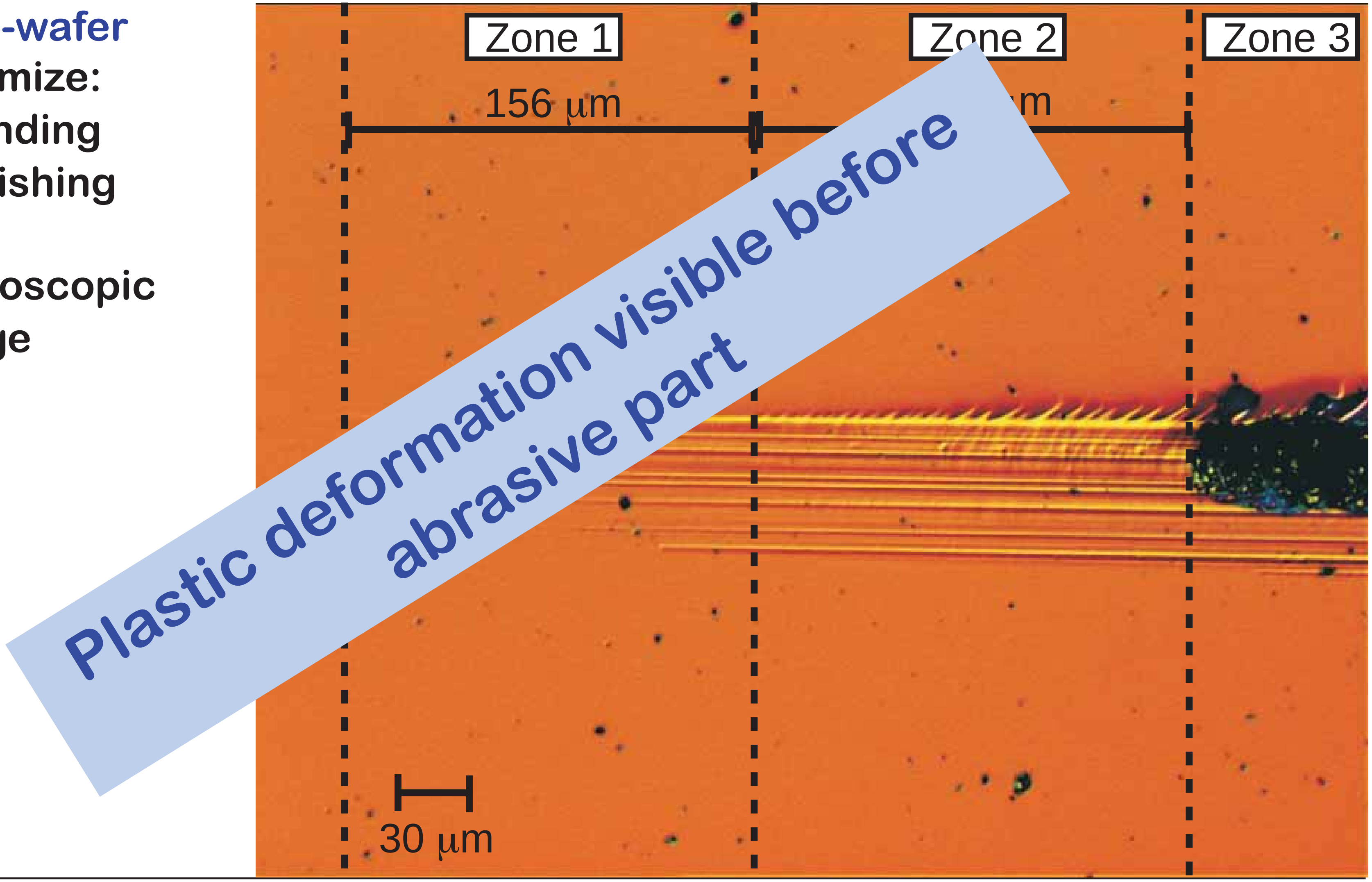
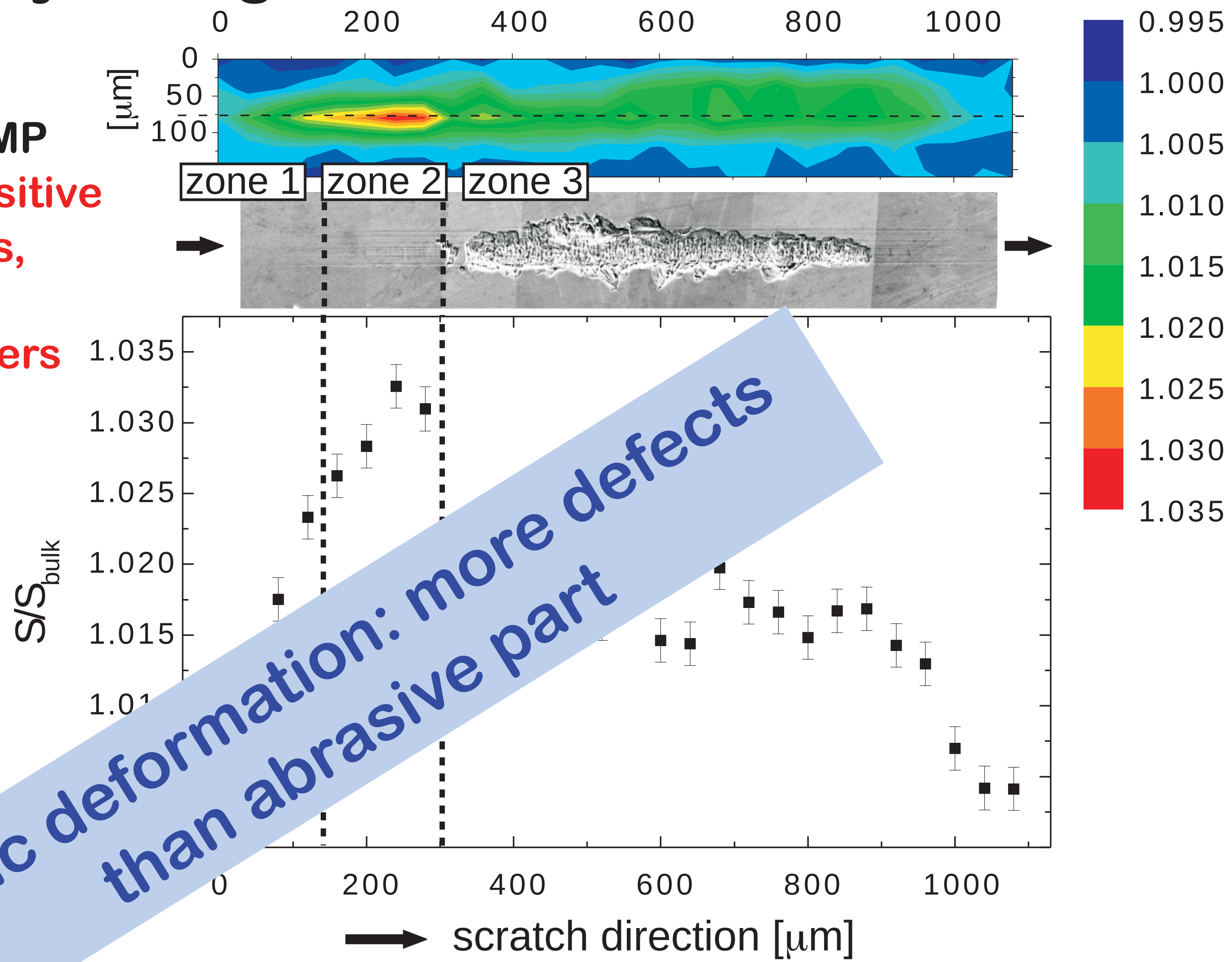
Scratching by a Single Diamond Grain

- **GaAs-wafer**
  - Optimize:
    - grinding
    - polishing
  - Microscopic image



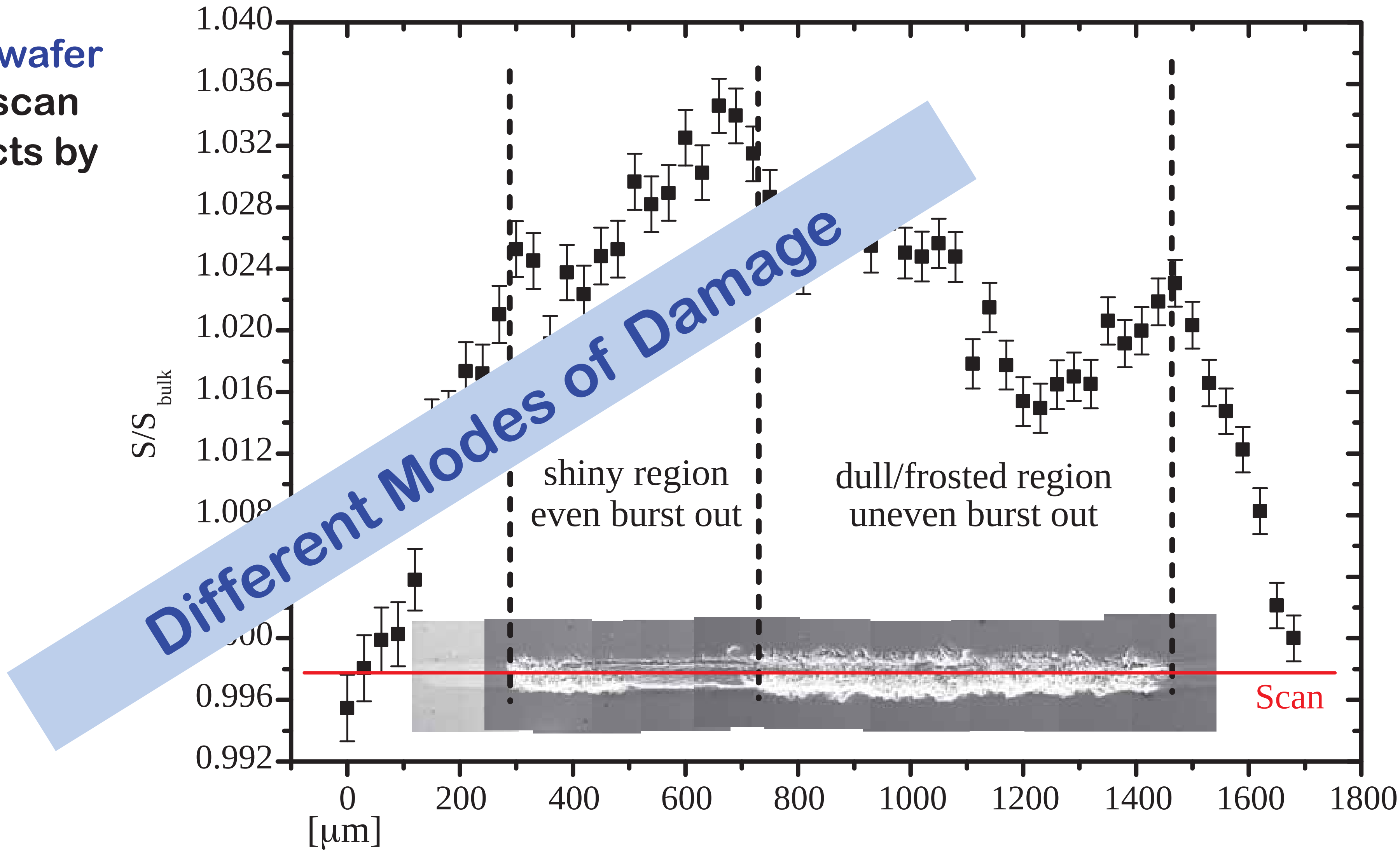
Scratching by a Single Diamond Grain

- GaAs-wafer
  - Damage by PMP
  - Positrons sensitive to dislocations, vacancies, vacancy clusters



Scratching by a Single Diamond Grain

- **GaAs-wafer**
  - Line scan
  - Defects by PMP



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Positron Annihilation
in
Complex Materials like Aluminum Alloys

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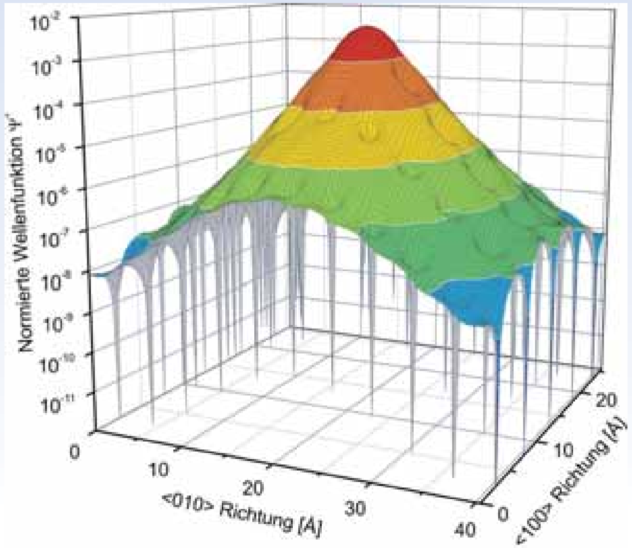

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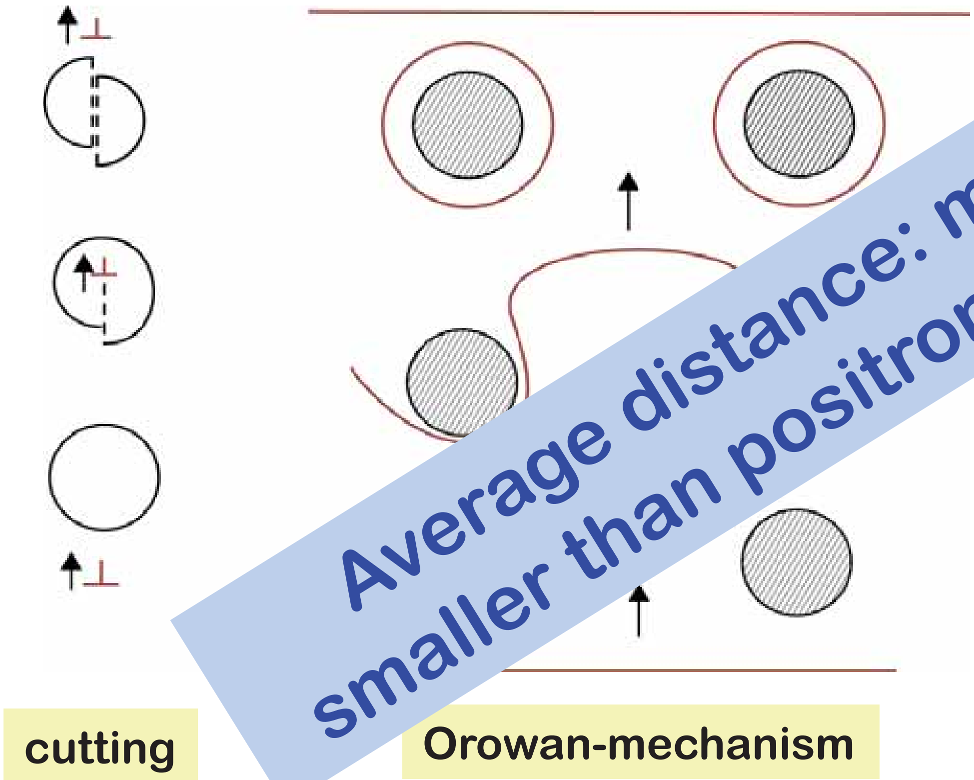
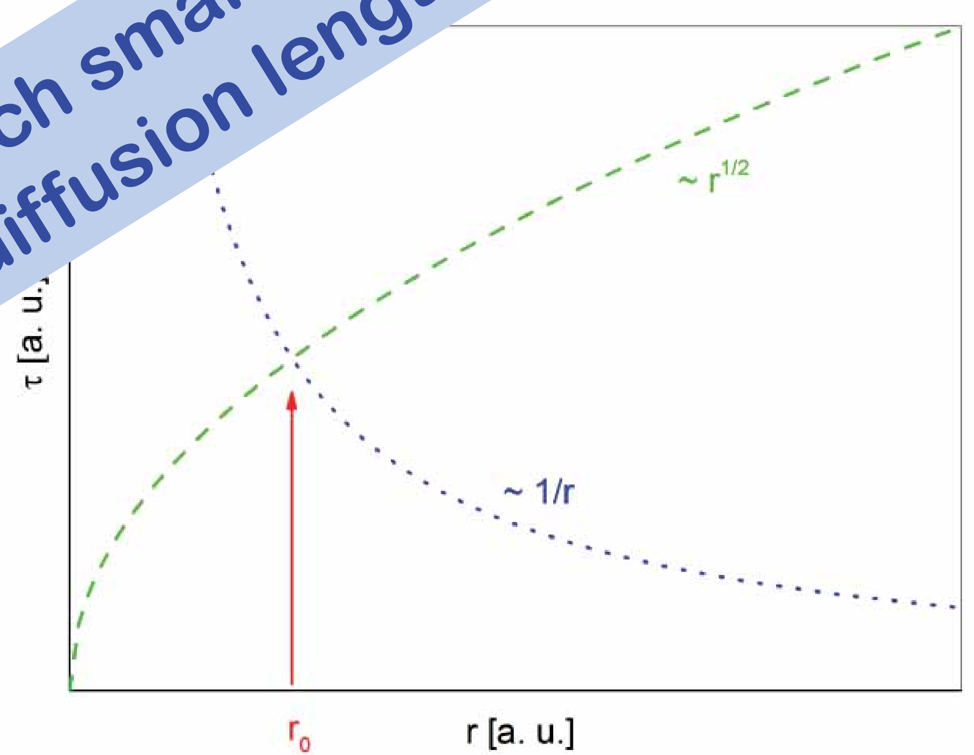
Understanding Materials: Preventing Damage?

Source: Airbus – Urheber: Roger Green

Hardening / Strengthening Mechanisms in Metals

- Alloy-hardening // work-hardening // Grain-size reduction
- Al-alloys: precipitation-hardening – particle-cutting < > Orowan-mechanism
  - finely dispersed particles in the nano-meter range



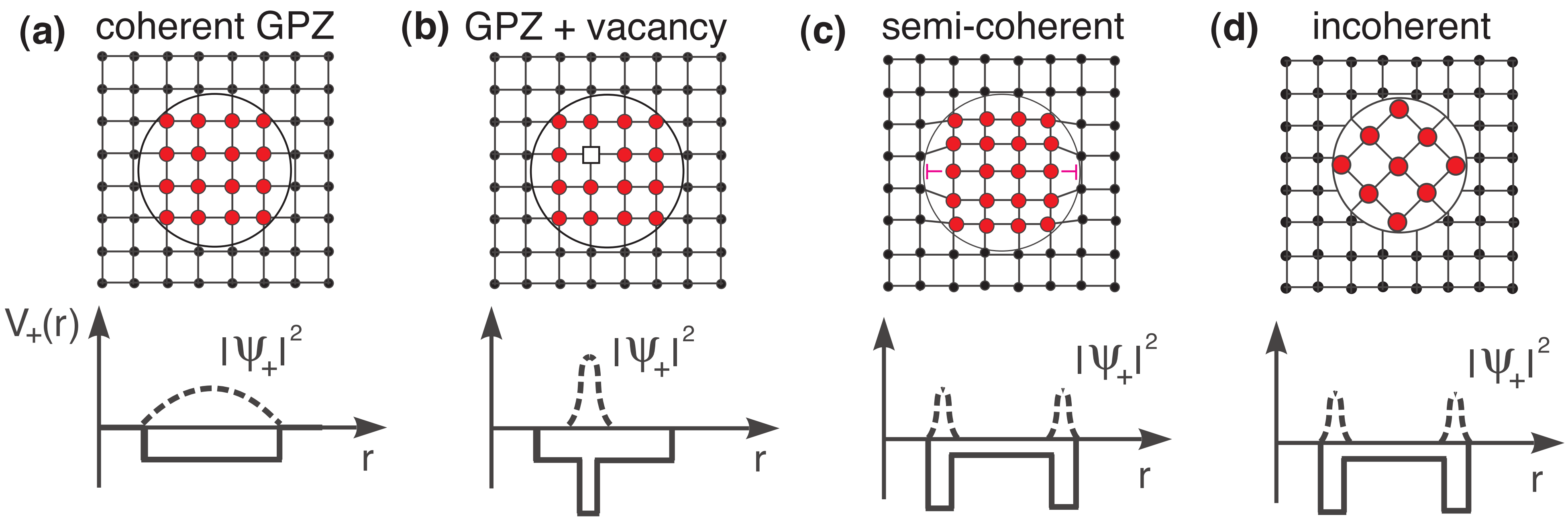


Average distance: much smaller
smaller than positron diffusion length

Positron Trapping to Precipitates

- **Different annihilation signals during aging samples:**
  - Coherent → semi-coherent → in-coherent

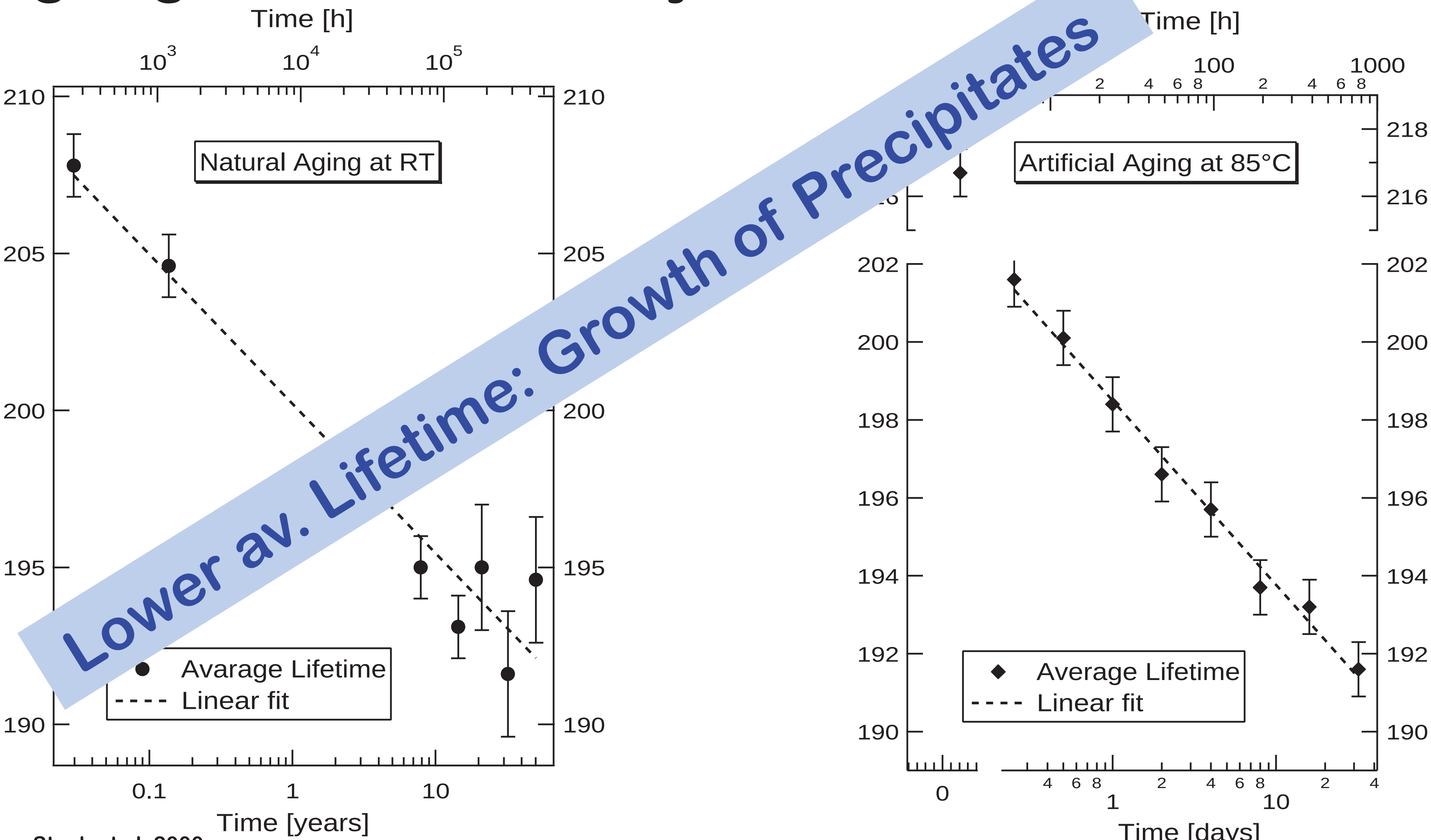
Different traps



- **Problem with deformed samples:**
  - Competing trapping of positrons to precipitates and dislocations

Bildquellen: Habilitation T.E.M. Staab

Ageing of Aluminum Alloys – no dislocations



Source: Staab et al. 2000

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3rd Application of PPMA

Fatigue in Aluminum-Alloys

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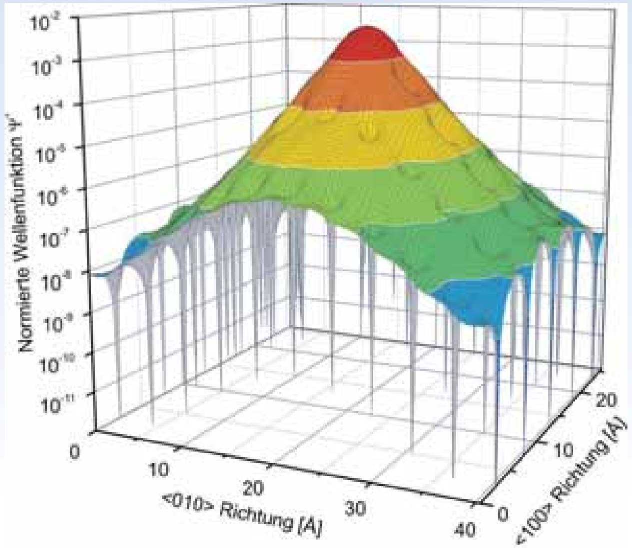

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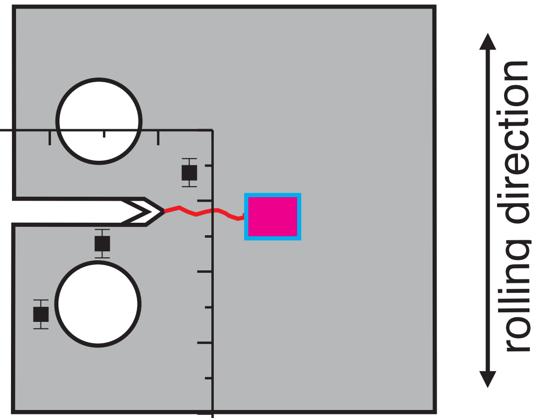
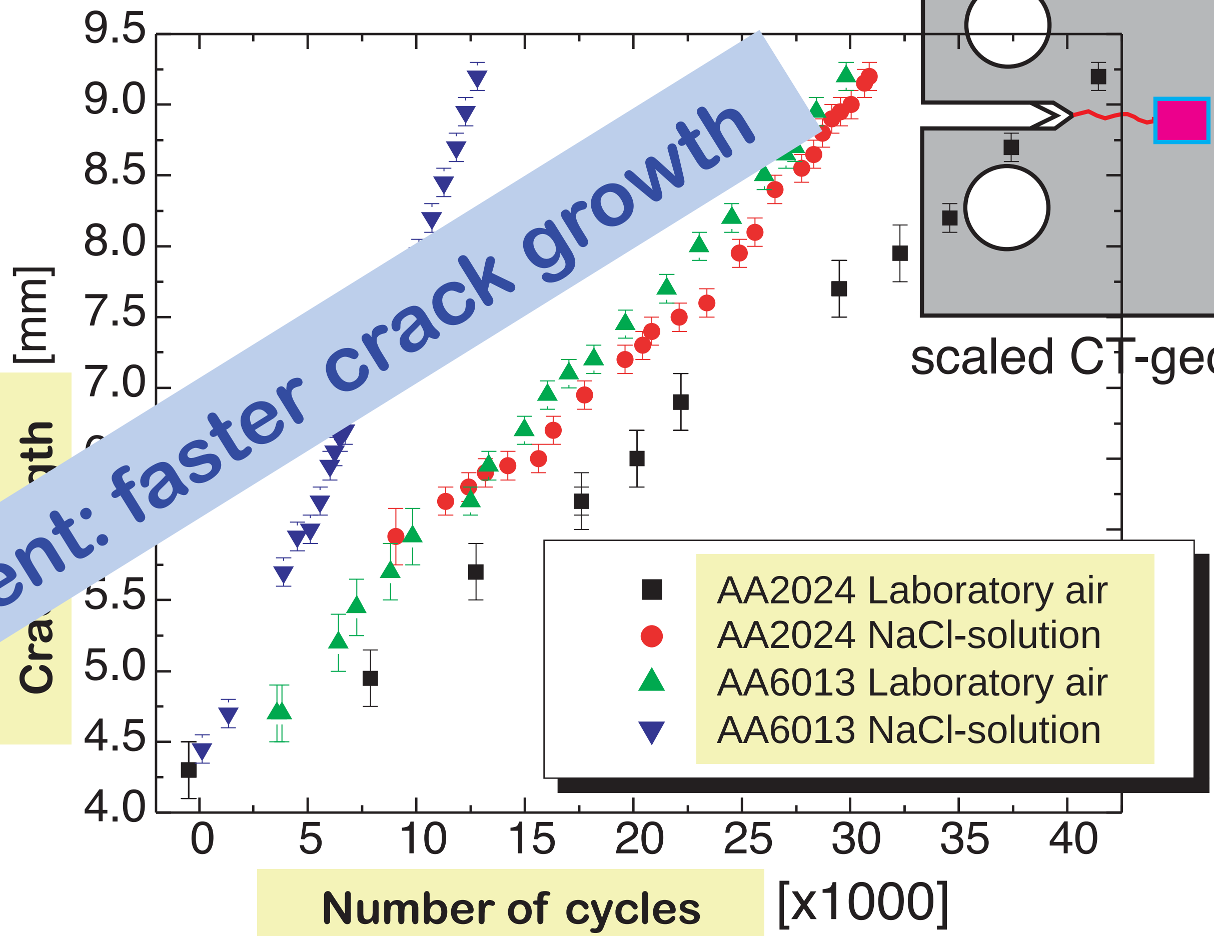
Crack Formation and Crack Growth

Geometry of samples



scaled CT-geometry

Fatigue: Piezo actors



Number of cycles [x1000]

Crack growth in aluminum-alloys

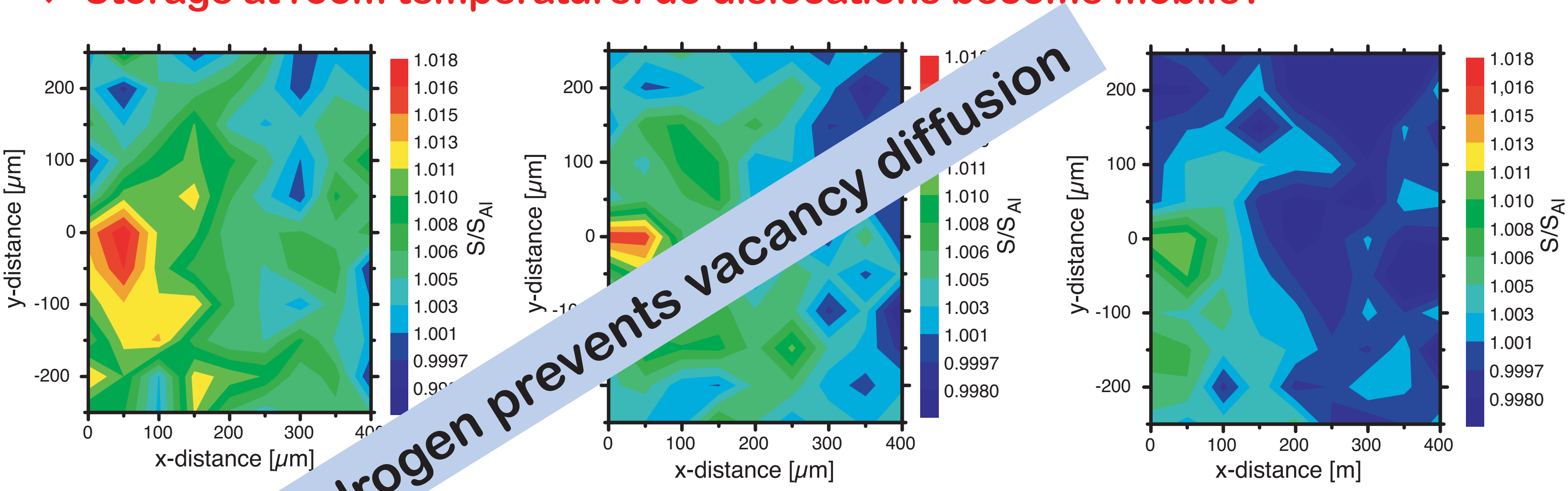
Corrosive Environment: faster crack growth

Sources: Chrstiane Zamponi – Dissertation Bonn 2003

Plastic Zone in Al-Alloys

- **Quite small plastic zone detected**
  - **Stable after fatigue in laboratory air**

- **Fatigue in corrosive solution: plastic zone changing with time – reason???**
  - **Storage at room temperature: do dislocations become mobile?**



Measurement directly after crack initiation

Measurement after 2 weeks

Measurement after 6 weeks

Sources: Chrstiane Zamponi – Dissertation Bonn 2003

4^{th} Application of PPMA

Characterizing Local Phases

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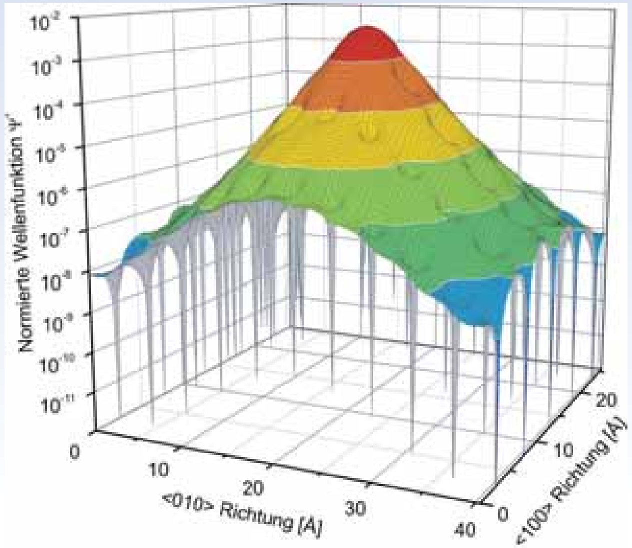

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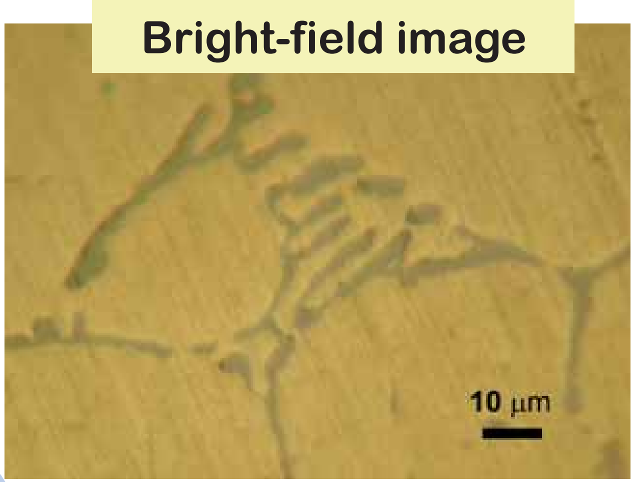
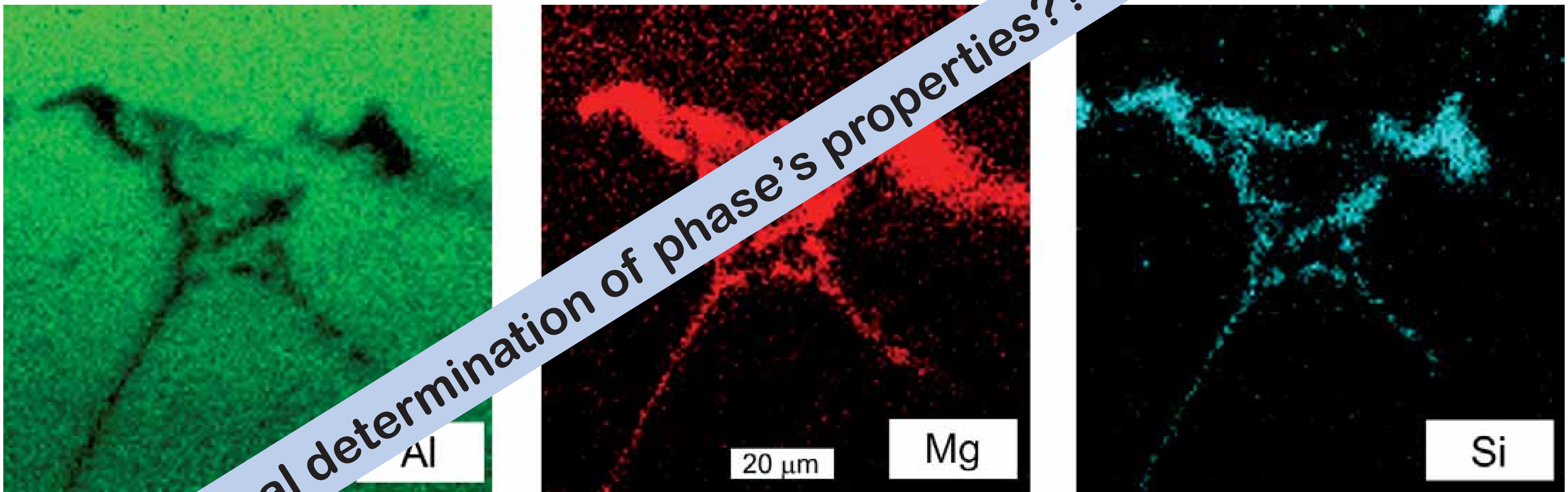
EDS-Mapping by SEM: Al-Mg-Si



- Over-aged Al-alloy: large precipitates – Mg_2Si
  - "Chinese script"
- Element mapping by EDS
  - Distribution of Mg and Si



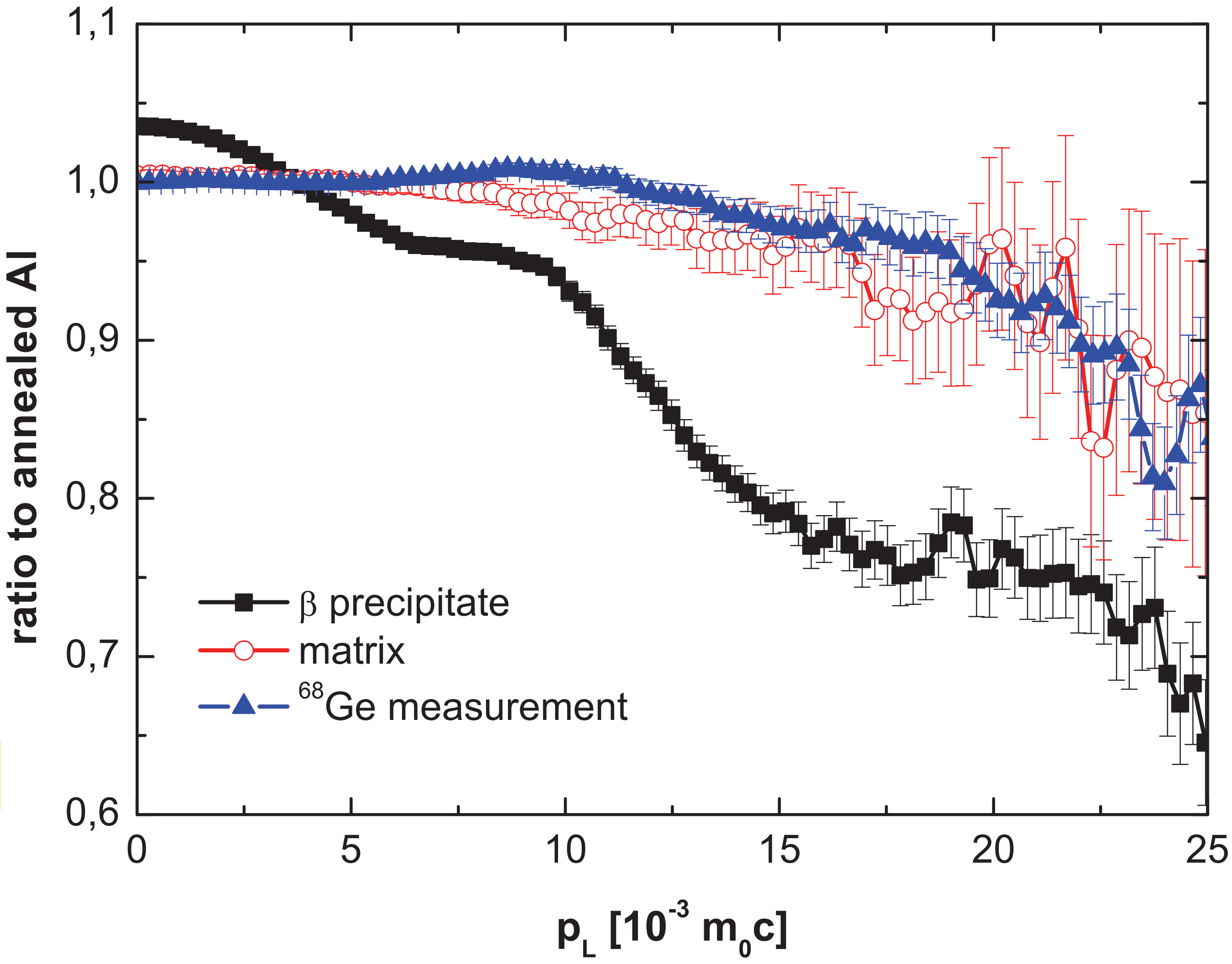
Local determination of phase's properties??

Source: B[illegible] Klobes– Dissertation Bonn 2009

Al-Mg-Si Alloys: Comparison to Bulk Measurement

- Al-Mg-Si alloy: Mg_2Si
  - Overaged stage
- ^{68}Ge: bulk measurement
  - All positrons annihilate in the bulk (large precip.)
- PPMA
  - Distinguish matrix and secondary phases

Ratio-to-bulk: pure Al = 1.00

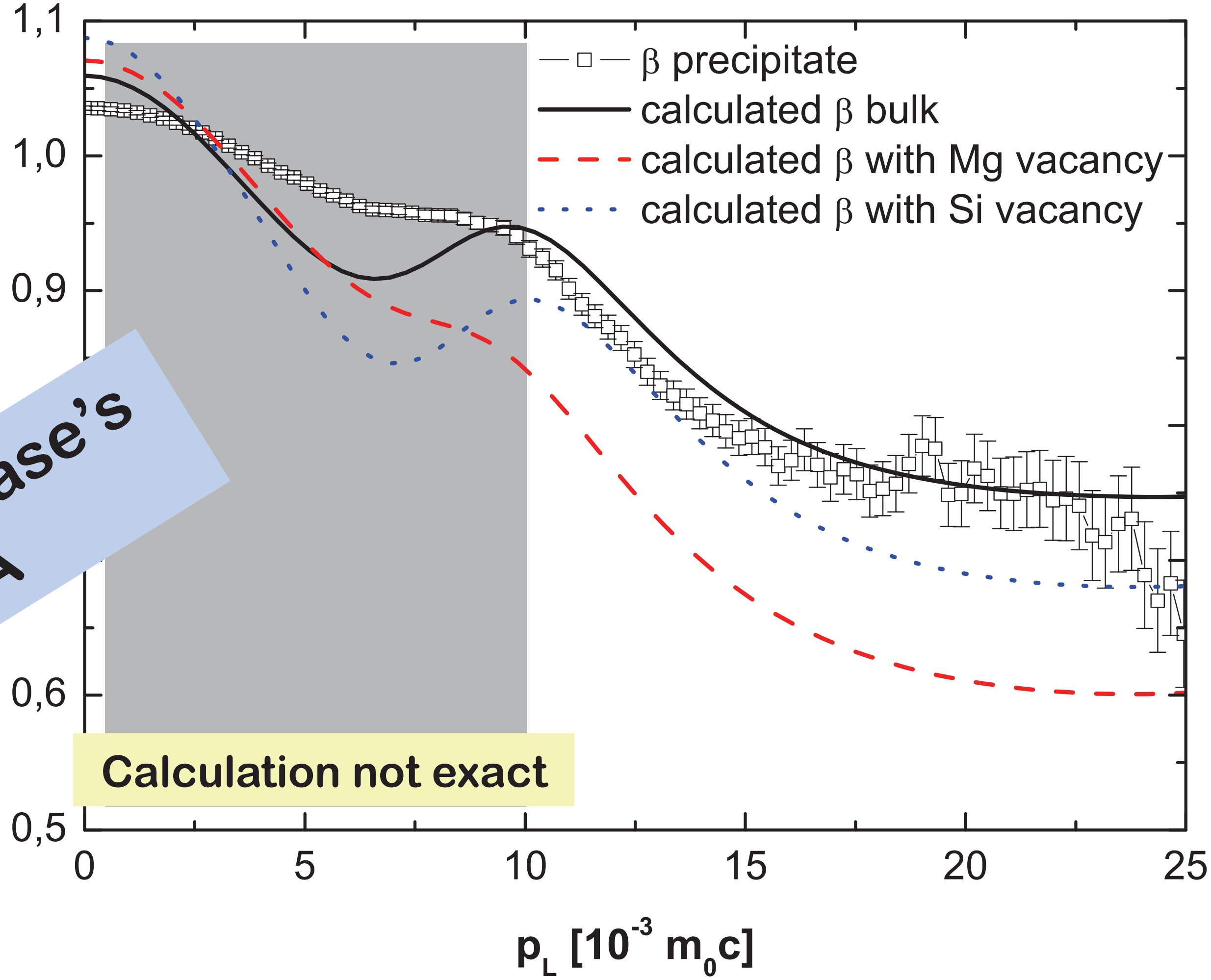


Source: Benedikt Klobes– Dissertation Bonn 2009

Al-Mg-Si Alloys: Ab-initio Calculations

- **Lattice structure of Mg_2Si**
  - **Momentum distribution**

- **Conclusion:**
  - **Mainly bulk Mg_2Si**
    BUT: contribution from vacancies on sub-lattice:
    - Si: 41%
    - Mg: 16%



Local determination of phase's properties by PPMA

The Blind Eye of Positrons

Employing Complementary Methods

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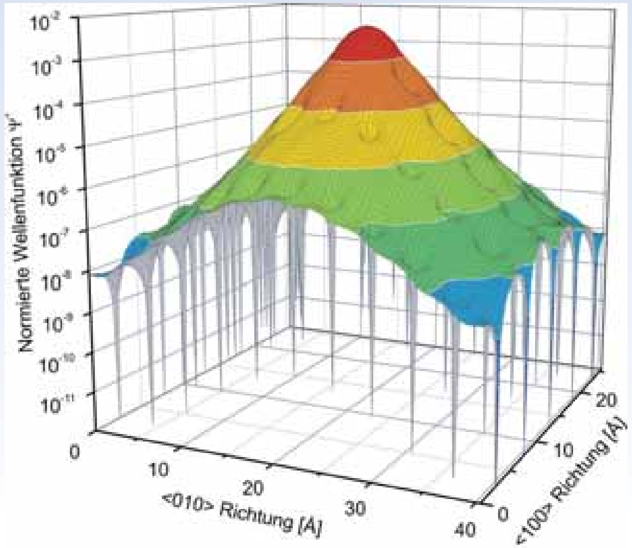

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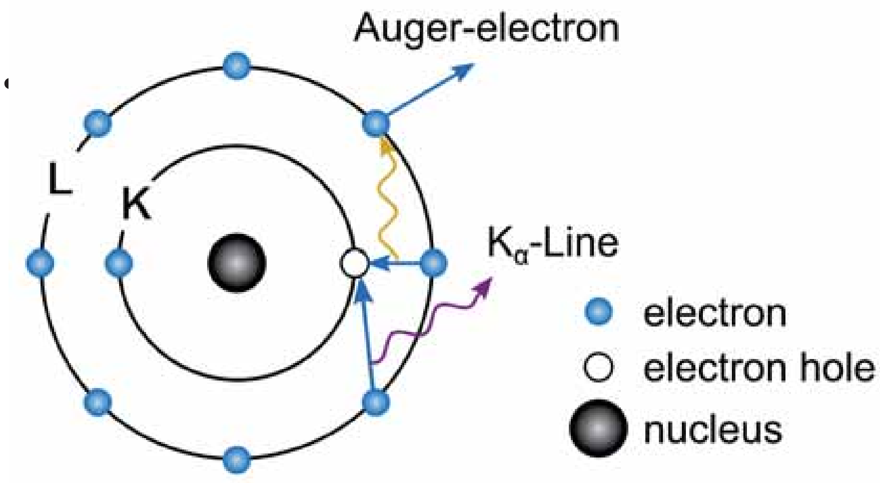
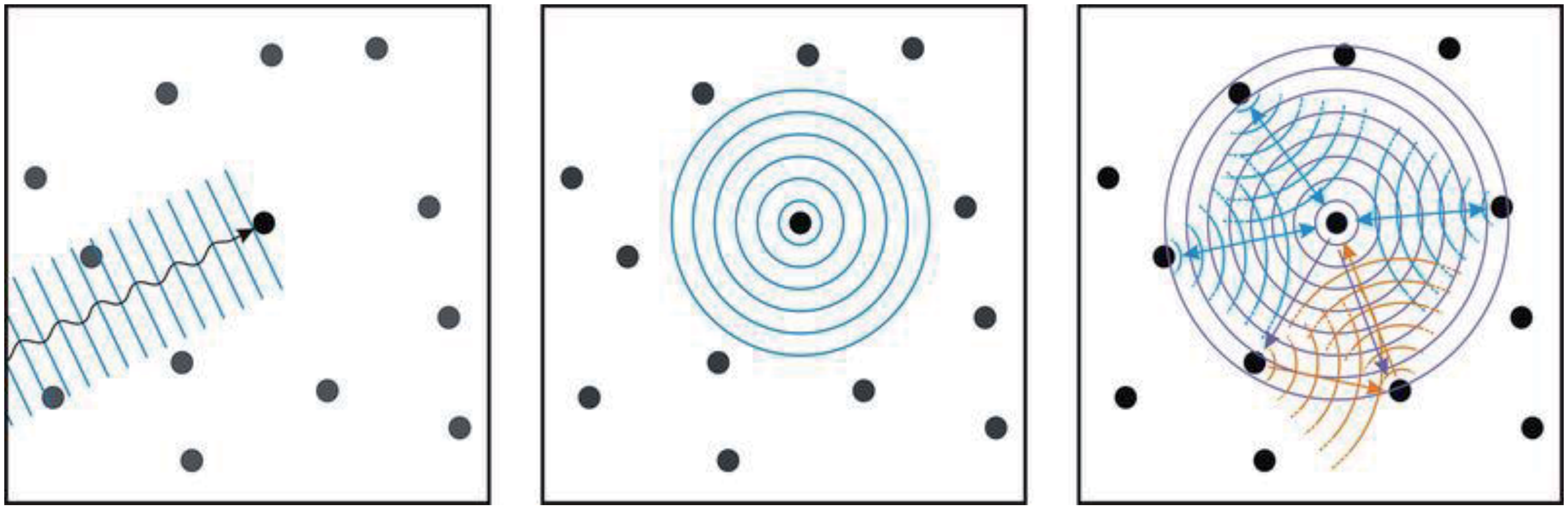
Just Look at Vacancies … but …



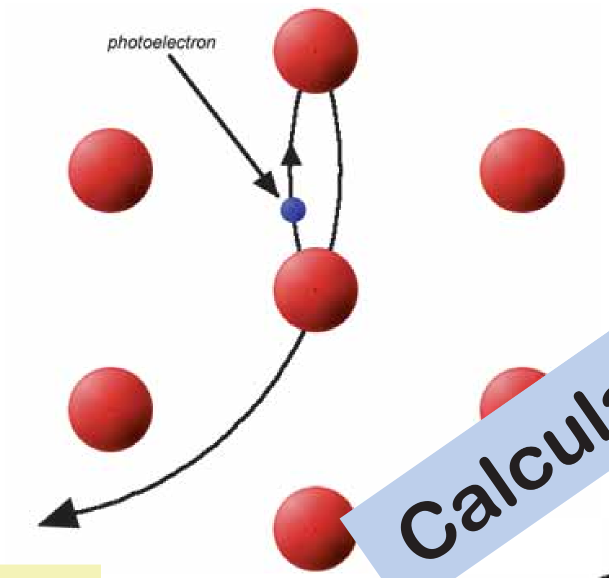
- Positrons in metals are sensitive to:
  - Vacancies
  - Dislocations
  - Grain boundaries

XAFS principle

- BUT not to alloying or trace elements
  - Solution: X-Ray Absorption Fine Structure (XAFS)
  - EXAFS / XANES sensitive to specific elements: k-edge absorption



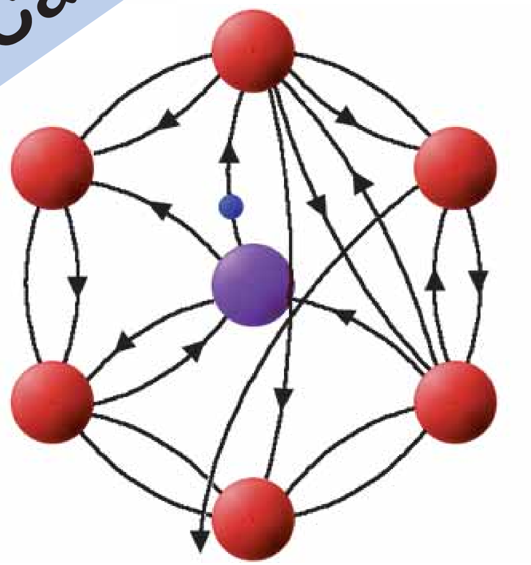
XAFS curve for Al-alloys

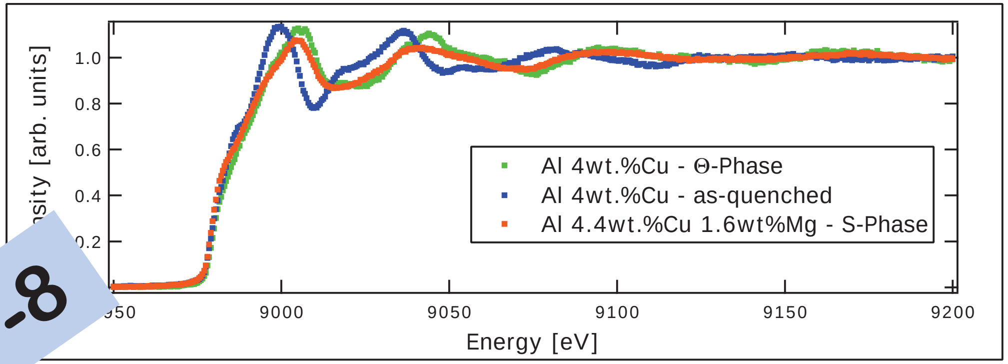


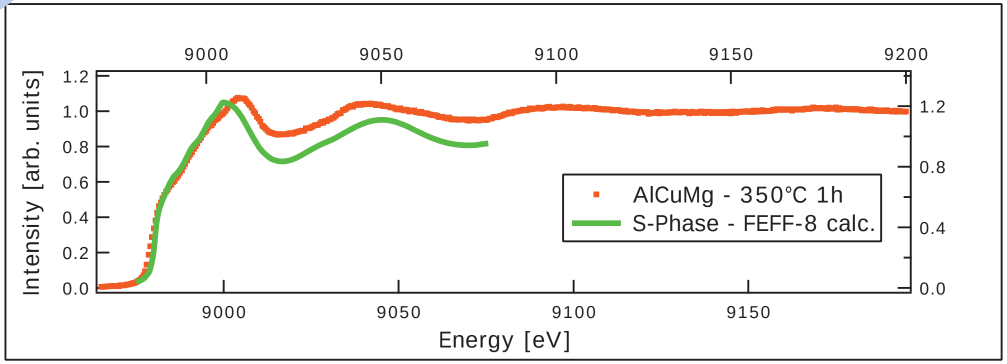
EXAFS

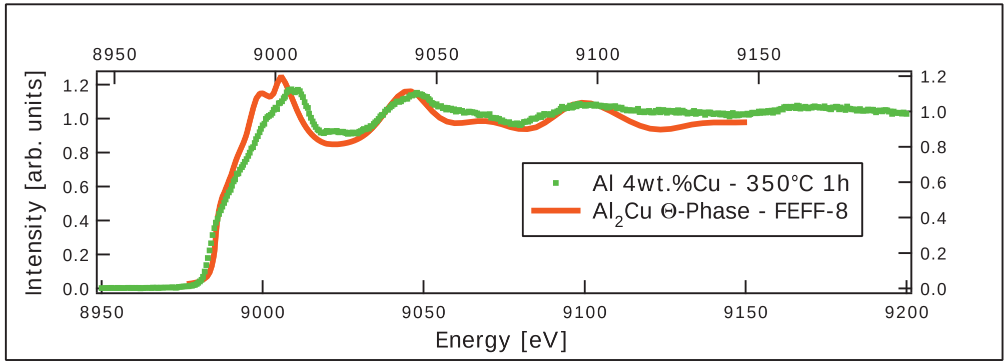
Calculations: FEFF-8

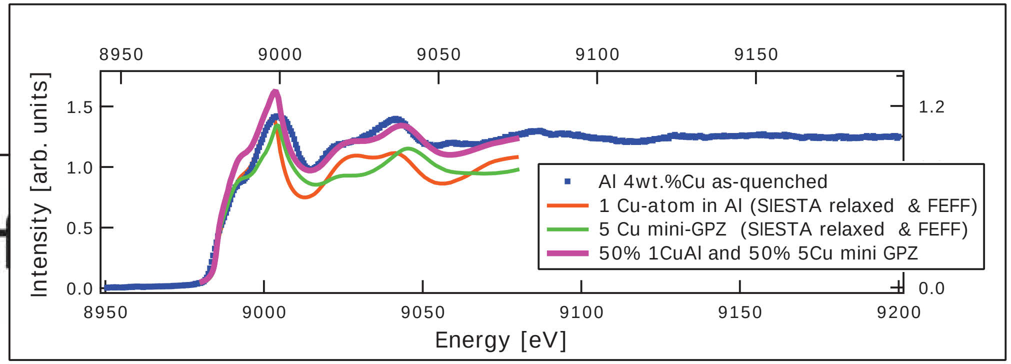
XANES











Source: Wikipedia

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Bildquellen: T.E.M Staab

Small Angle X-Ray Scattering
(SAXS)

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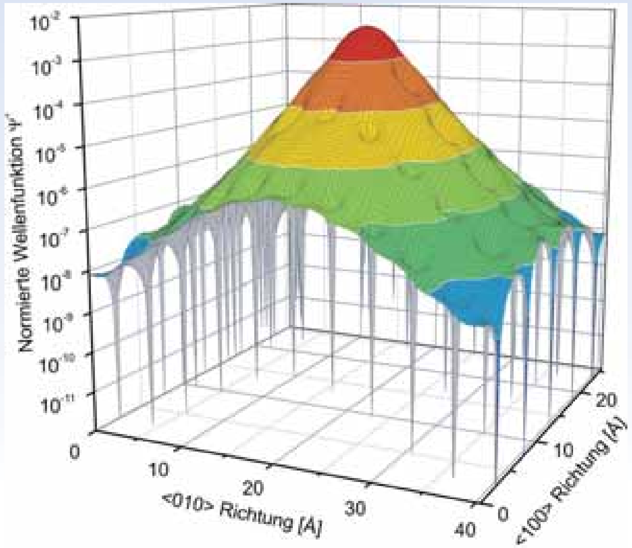

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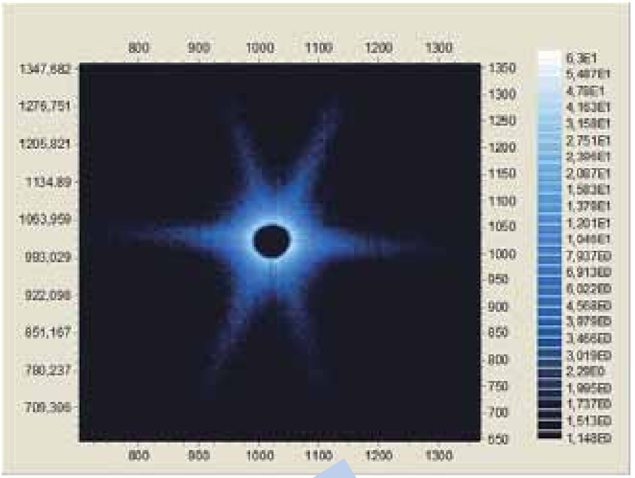
Changes in FSW-Welded AA2198

- Changes of scattering pattern across the weld line in FSW-welded AA2198
- Reflects changes in precipitate's morphology
- Lateral scan across the weld line possible (laboratory (beam spot: 150 – 400µm)

a



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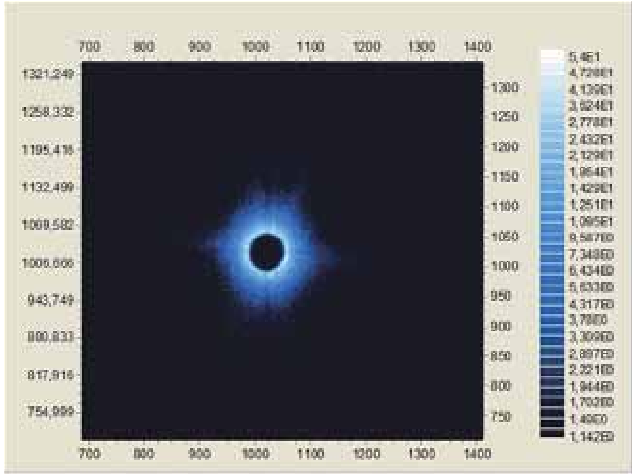

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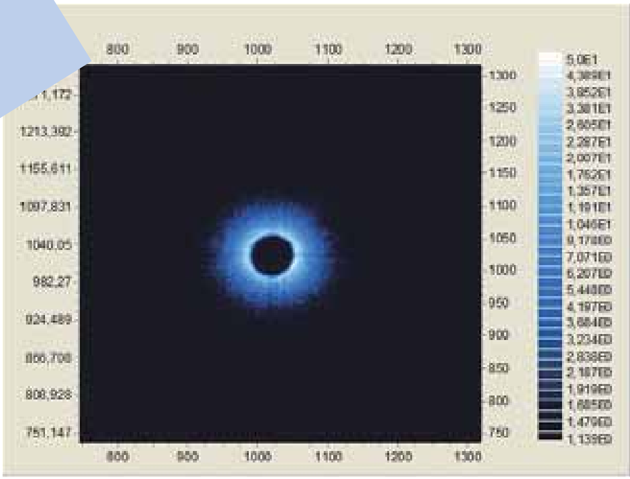

Determine particle size & particle size distributions

Source:

Future Applications:
Positron Annihilation &
Small Angle X-Ray Scattering

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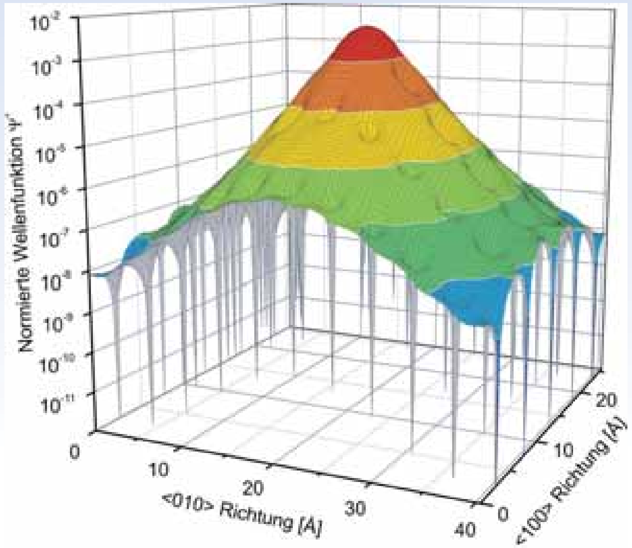

Source: Wikipedia

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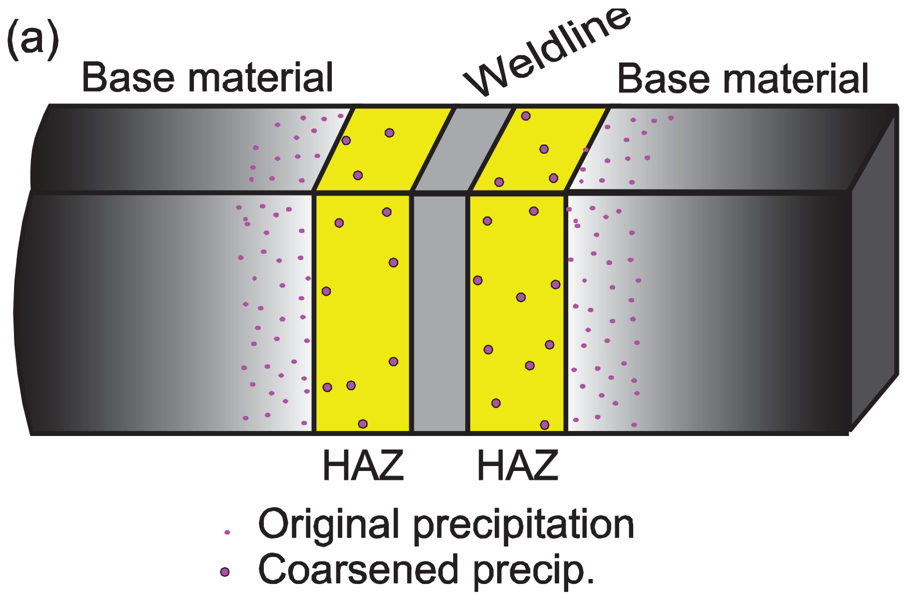
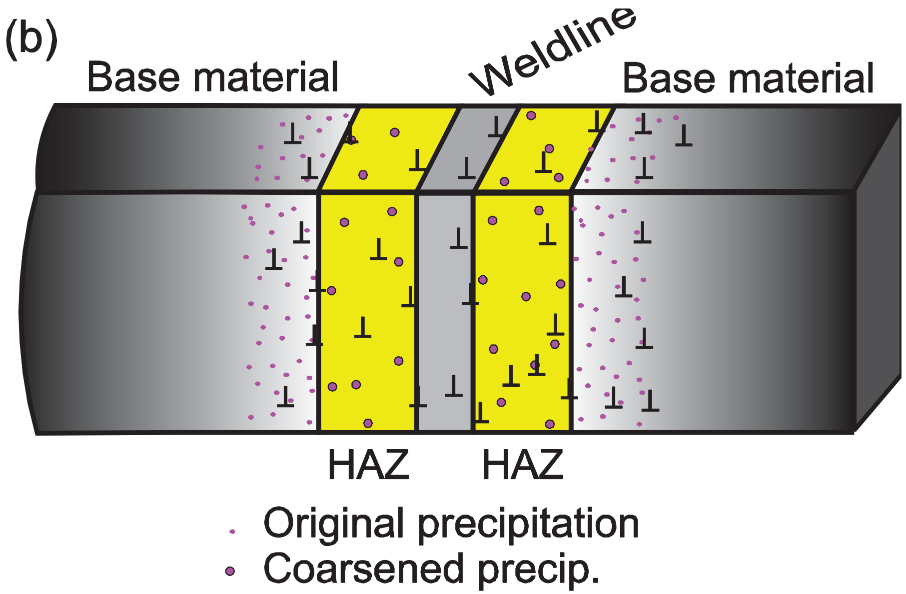
Investigation of Microstructural Changes: Weld-Line

- **Deformed samples:**
  - Competing trapping of positrons to precipitates and dislocations





- **Solution:**
  - Determine precipitate sizes and distributions by e.g. SAXS
  - Combine with Monte Carlo Simulation of the positron diffusion

Summary

- **Positron Micro Probe (PMP) – principles of PPMA (together with EPMA)**
- **Examples for applications: microscopic damage (Steel, GaAs, Al-alloys, ..)**
- **Combination with complementary methods: EXAFS & SAXS (focused beams)**
- **Future topics: fatigue in welded parts - ab-initio simulations**

LCTM – at the [illegible] Würzburg

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Thank you for your attention

PD Dr. T[illegible]

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Julius-Maximilians-Universität Würzburg
Chemische Technologie der Materialsynthese

Ende der Präsentation