In-situ Determination using Materials Behaviour using HESR

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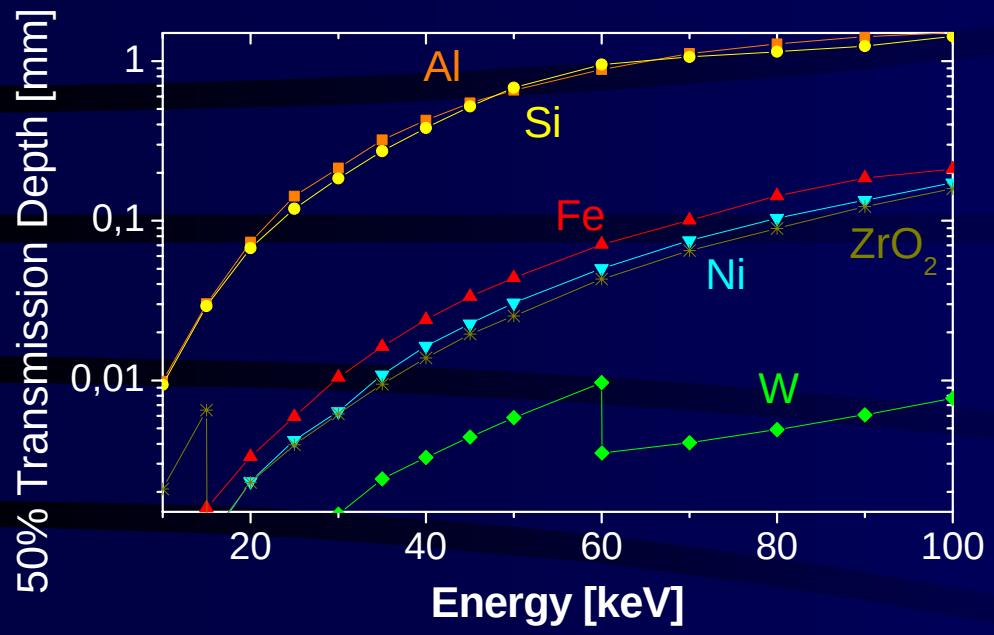
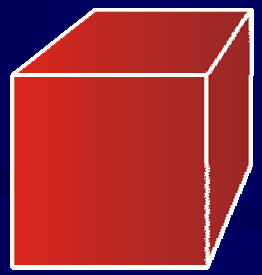
- **Introduction:**
  Why are high energy X-rays interesting for engineering applications ?
- **Strain and Texture Analyses with High Local Resolution**
  Examples, trends, requirements
- **In-situ and Time-resolved Strain Analyses**
  Examples, trends, requirements
- **Future Perspectives:**
  Combined Experiments, Complementary methods, ..

Introduction: Reasons for using HESR in Engineering



HESR: E > ~ 40keV
V: μm^3..some 100 μm^3



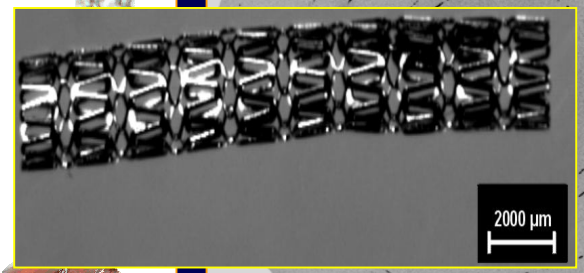
neutrons:
V > 1 mm^3

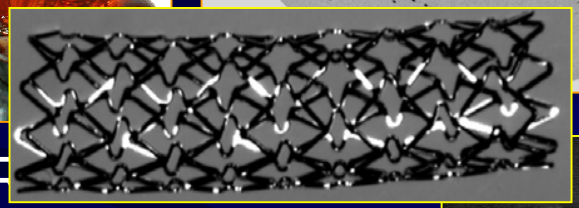
(HE)SR:
- high photon flux ⇒ short data acquisition time, small gauge volume
- high parallelism ⇒ small and well defined gauge volume
- large available energy range ⇒ penetration depth defined by photon energy, measurements at the surface and in the bulk feasible

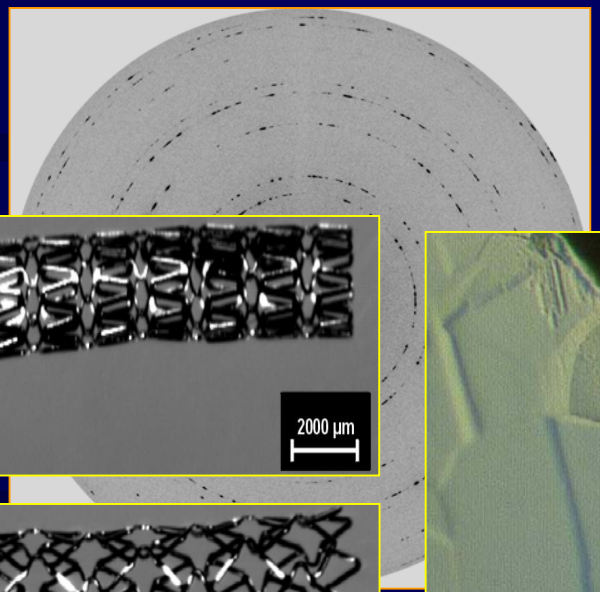
Experiment principles

- monochromatic radiation: $\varepsilon = 1 - \sin\theta/\sin\theta_0$
- white radiation: $\varepsilon \sim (E_0 - E^*) / E_0$

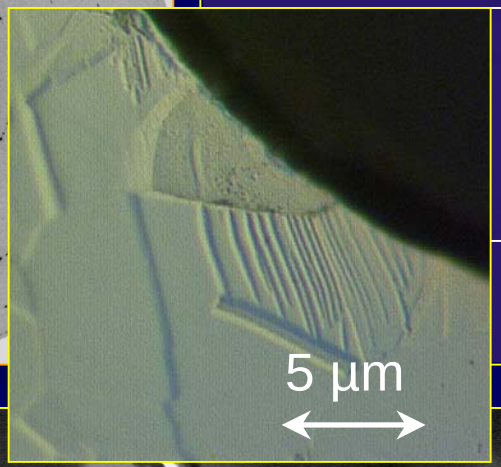
Example 1: Orientation and Strain Analyses in Individual Grains in Metallic Stents

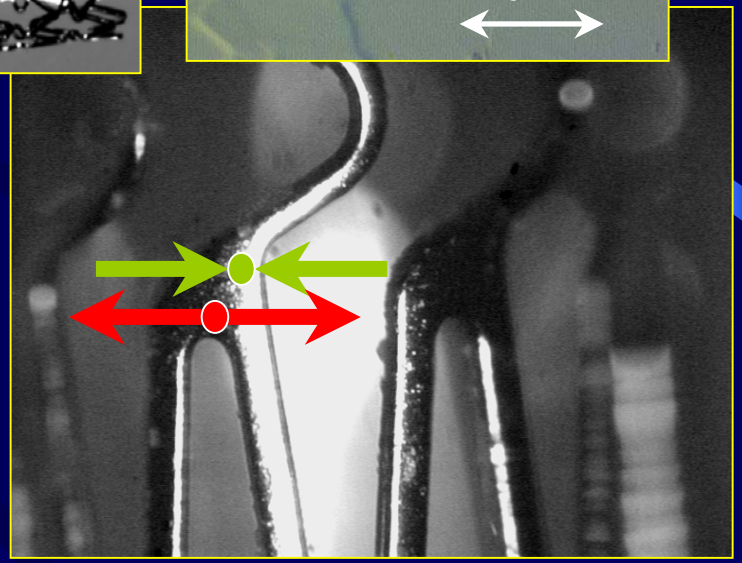






Gas detector /R. Kampmann et al. GKSS/





TF

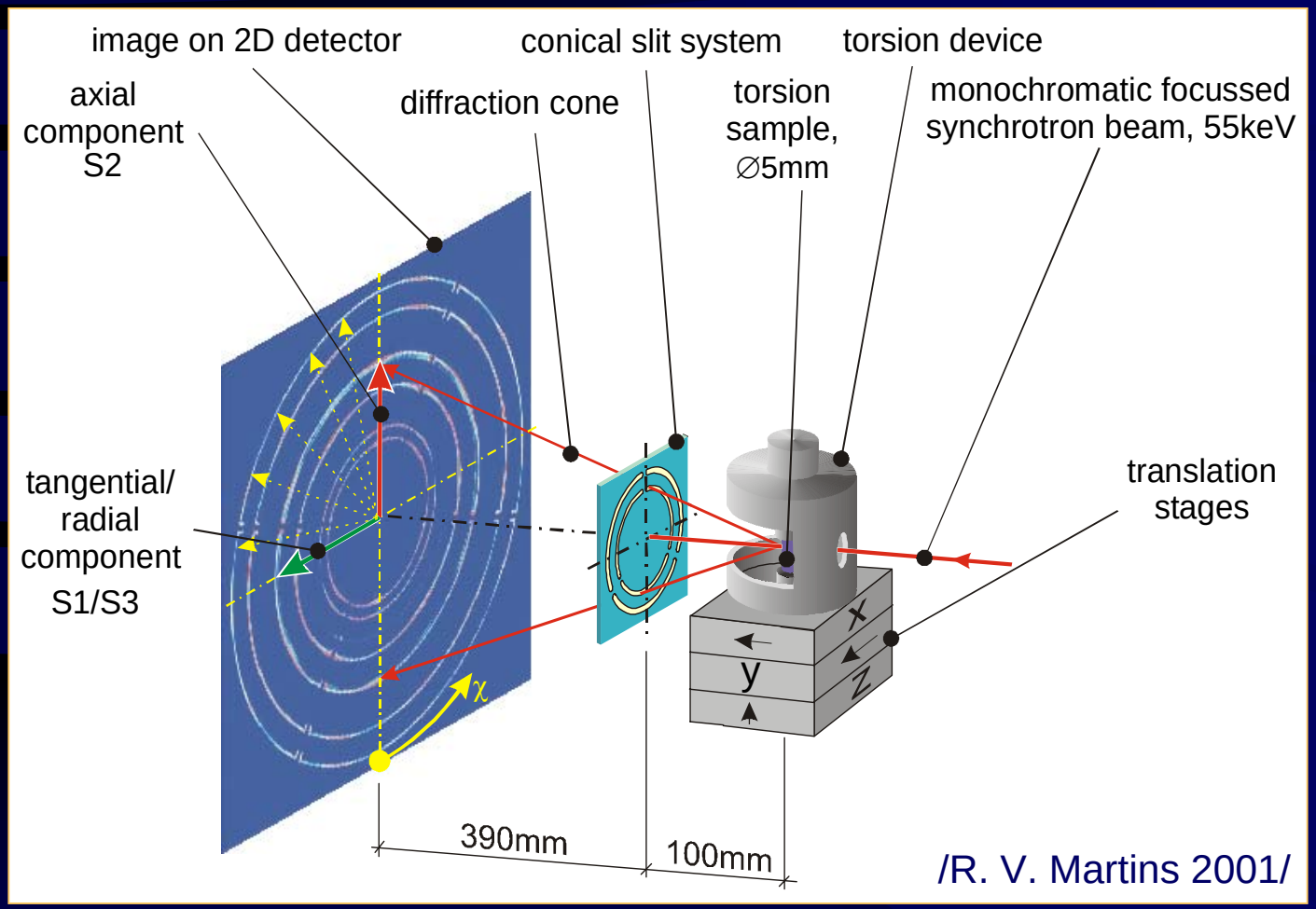
y

→ Strain tensors in individual grains

Stress tensor [MPa]: Error [MPa]:

$$\begin{pmatrix} 230 & -27 & 15 \\ & 210 & 44 \\ & & 120 \end{pmatrix} \pm \begin{pmatrix} 38 & 32 & 20 \\ & 42 & 31 \\ & & 45 \end{pmatrix}$$

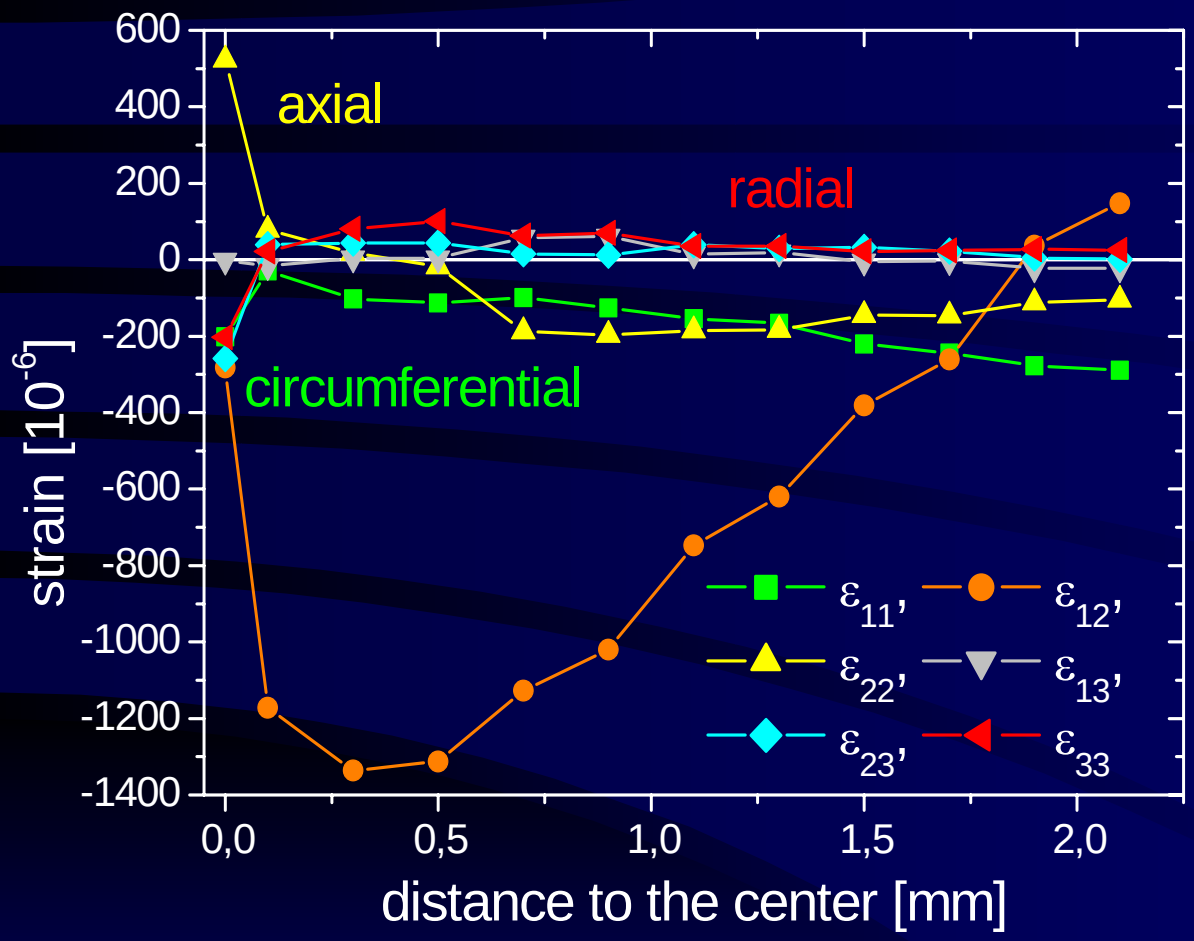
Example 2: High – Resolution Strain Analyses in the Bulk using Monochromatic High Energy SR

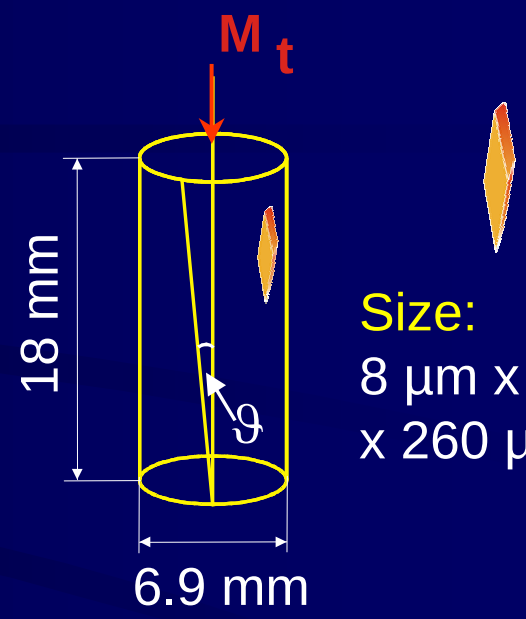


Gauge volume size: e.g. 8 µm x 5 µm x 260 µm

- set-up for strain analyses in the 2nd hutch of ID 11, developed by a co-operation between ESRF and Risoe National Lab., Roskilde, Denmark
- combination of high spatial and high angular resolution in bulk samples

Strains in the Interior of a Full Cylinder subjected to Torsion, Swift Effect





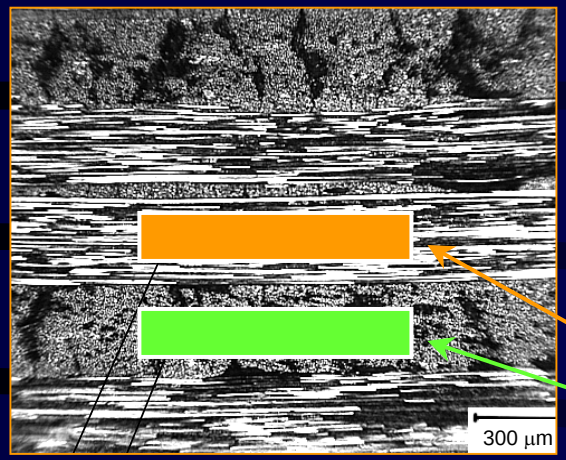
Size:
8 µm x 5 µm
x 260 µm

/R. Martins, U. Lienert, L. Margulies, A. Pyzalla 2001/

→ Strain analyses with high spatial and angular resolution
→ Analyses of a biaxial stress state
→ Possibilities for in-situ analyses (under mechanical and temperature loading)

Example 3: Applications for High resolution Strain scanning: Layers and Buried Interfaces

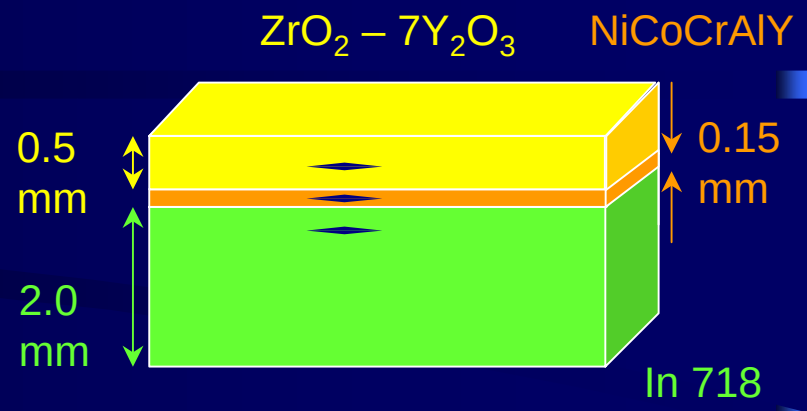
Layered Structures



SiC – SiC
orthogonal
fibers
0° / 90°

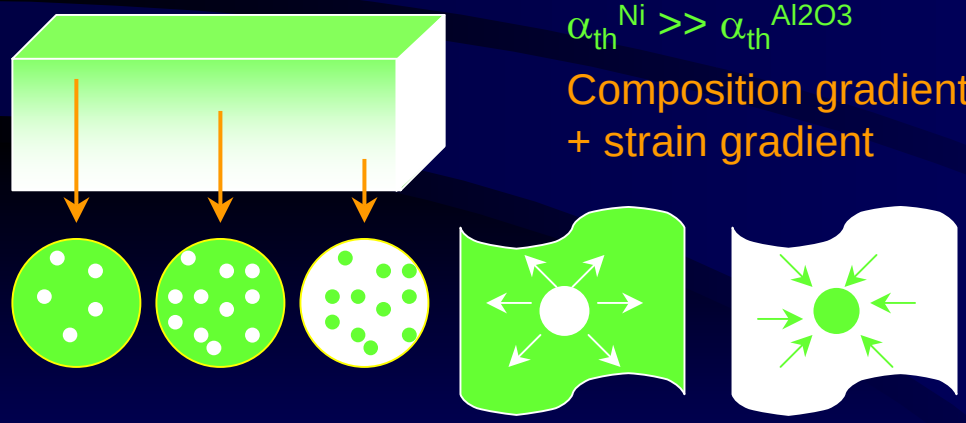
σ_{II}=230 ± 80MPa
$\sigma_{\perp}$=-30 ± 40MPa

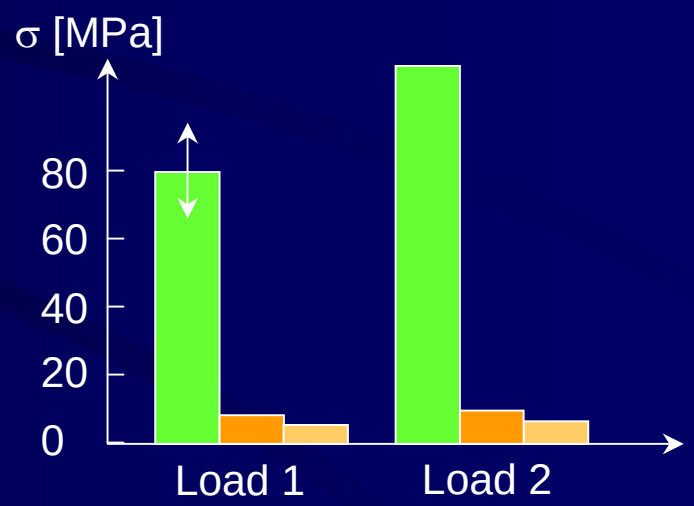
Thermal Barrier Coatings



Gradient Materials

Ni + Al_2O_3
$\alpha_{th}^{Ni} >> \alpha_{th}^{Al2O3}$
Composition gradient
+ strain gradient





→ Strain analyses with high spatial and angular resolution
→ Analyses near interfaces and in buried layers
→ Simultaneous determination of gradients in composition and stress

Strain and texture analyses with high local resolution “Strain scanning, texture scanning”

- scanning of strain / texture states of samples and components from the surface into the near-surface zone and the bulk, e.g. optimisation of processes (shot peening, hot extrusion, machining, steel drawing ..)
- scanning of strain distributions in critical zones of materials and small components, e.g. near interfaces, cracks (coatings, components with cold expanded holes, welds ..)
- ..

Methods, Requirements

- slit systems or optics (e.g. multi-layers, lenses) in the primary beam
- slit systems and / or optics in the secondary beam
- large range of available energies
  e.g. scanning in grazing incidence: very-near-surface stress/texture states
  high energy synchrotron radiation: bulk strain / texture states (<~200 keV)
- near-surface-zone: energy tuning (resolution in the in-depth direction)
- fast detection systems (e.g. 2D – detectors)
- Handling of samples / components allowing for heavy components, complex shaped components, movements with high lateral and angular precision
- “customer – friendly” data analyses software for engineers

Example 4: IGBTs in power electronics („HIMRATE“)

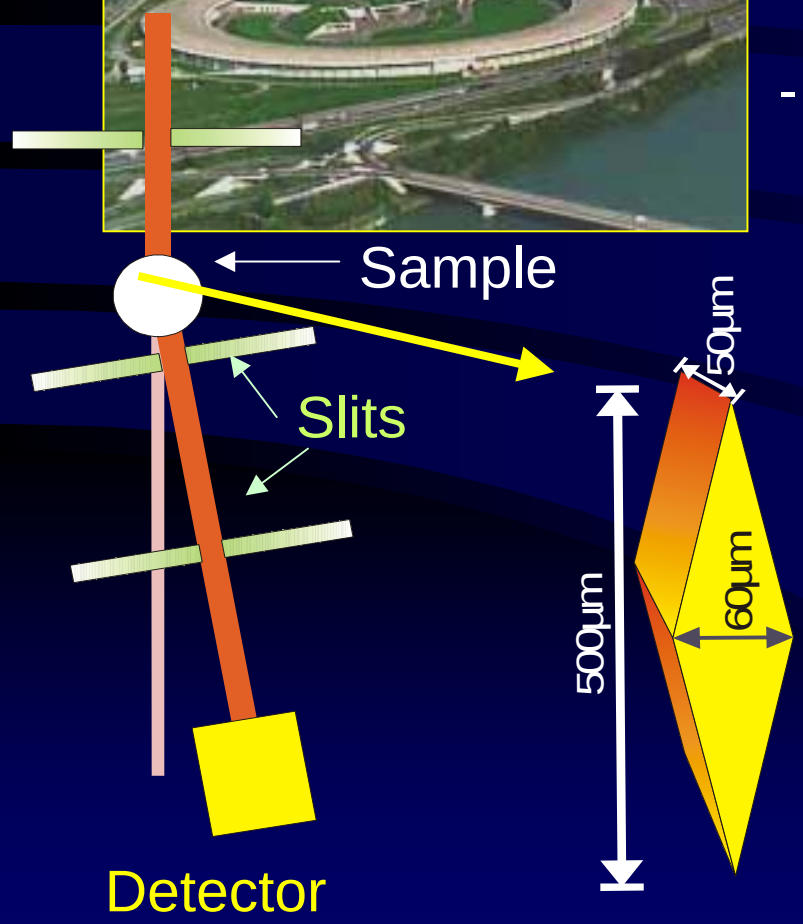
Unpublished results

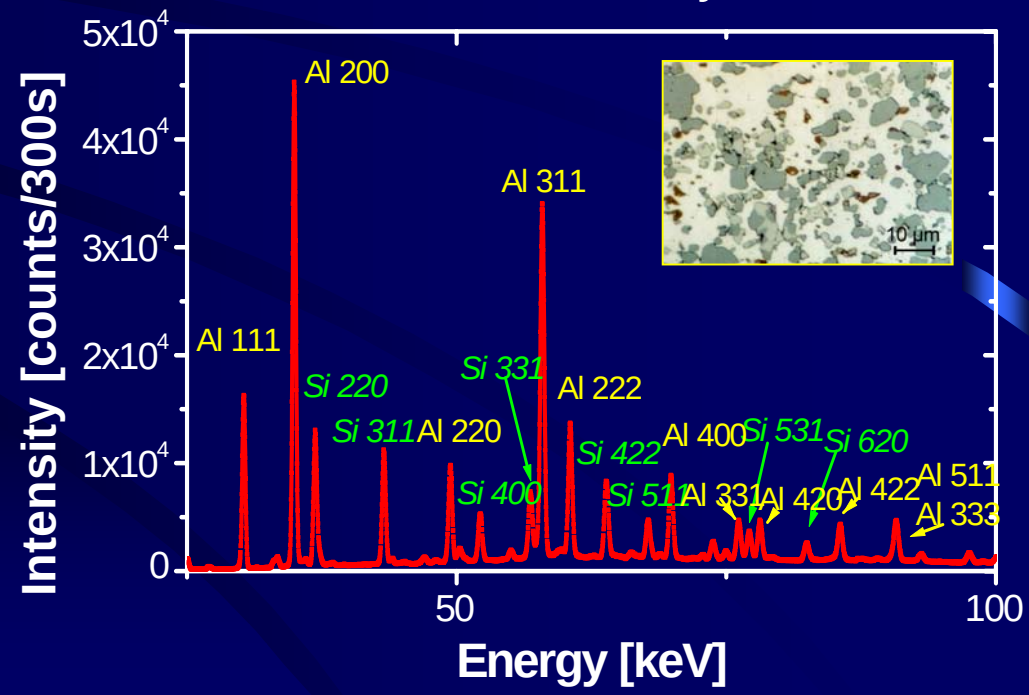
Example 5: In-situ Strain Analyses Using White High Energy Synchrotron Radiation

White high energy synchrotron radiation

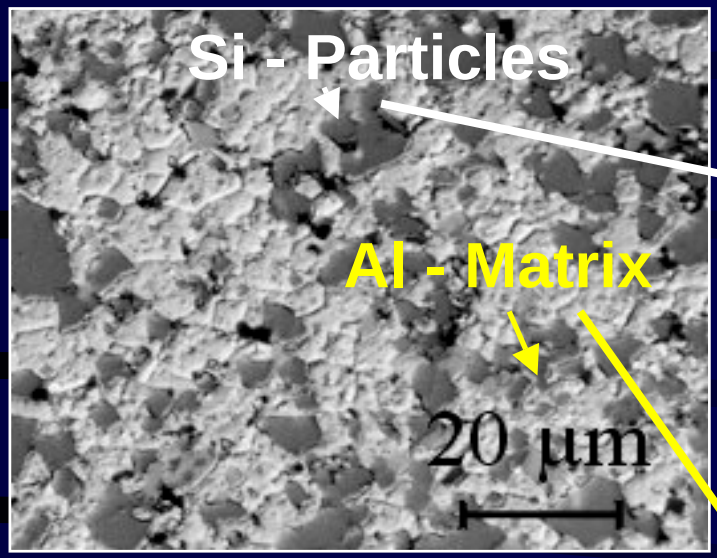
$n\lambda = 2d \sin\theta$, $\lambda = hc / E_{X\text{-}ray}$

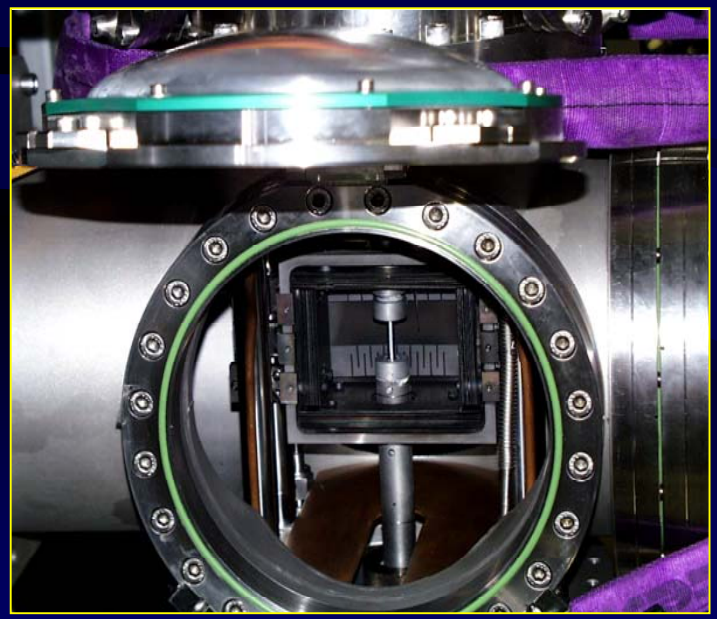
- high photon flux
- high photon energies
  → high penetration depth
- complete spectrum
  → phase analyses, texture analyses, residual stress simultaneously

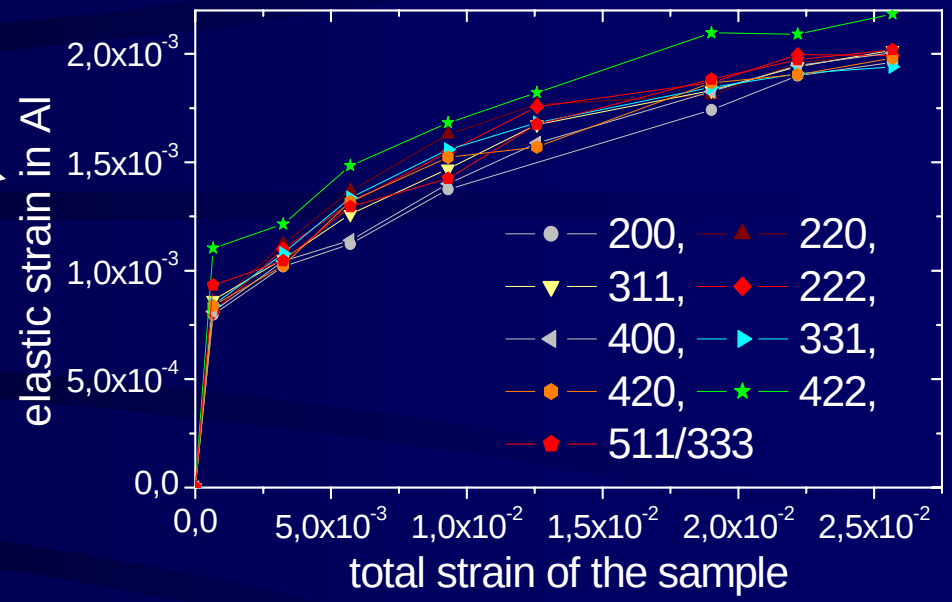


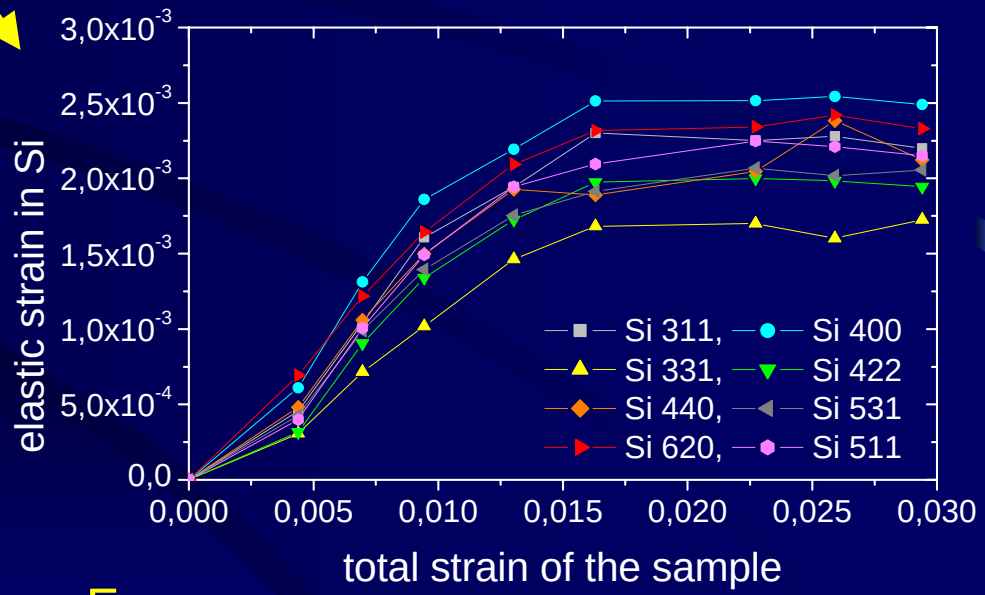


In-situ tensile Tests: AlSi25Cu4Mg1, 250°C



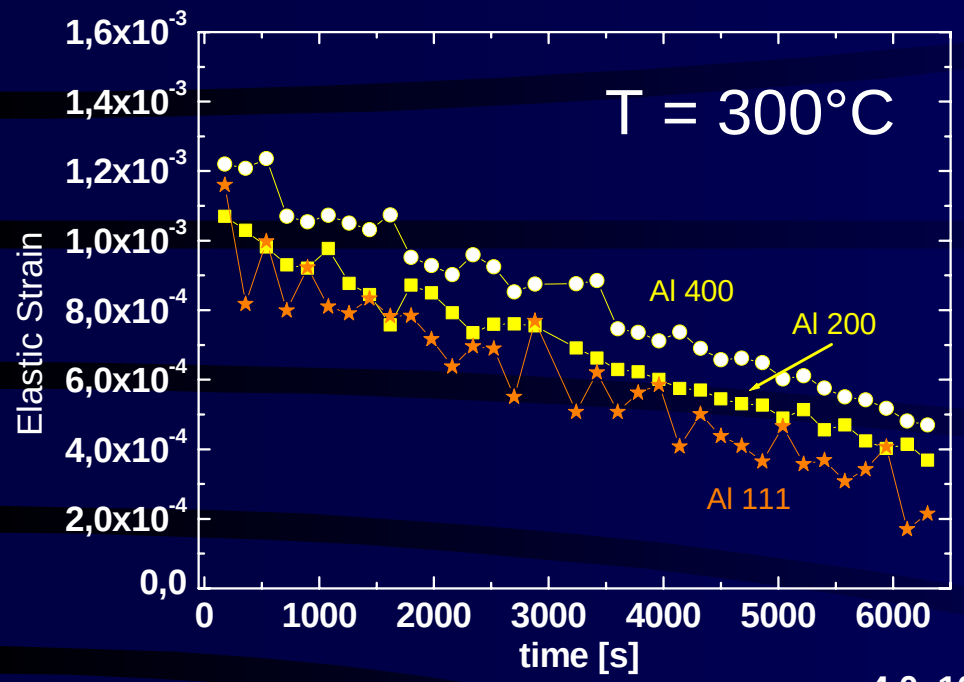






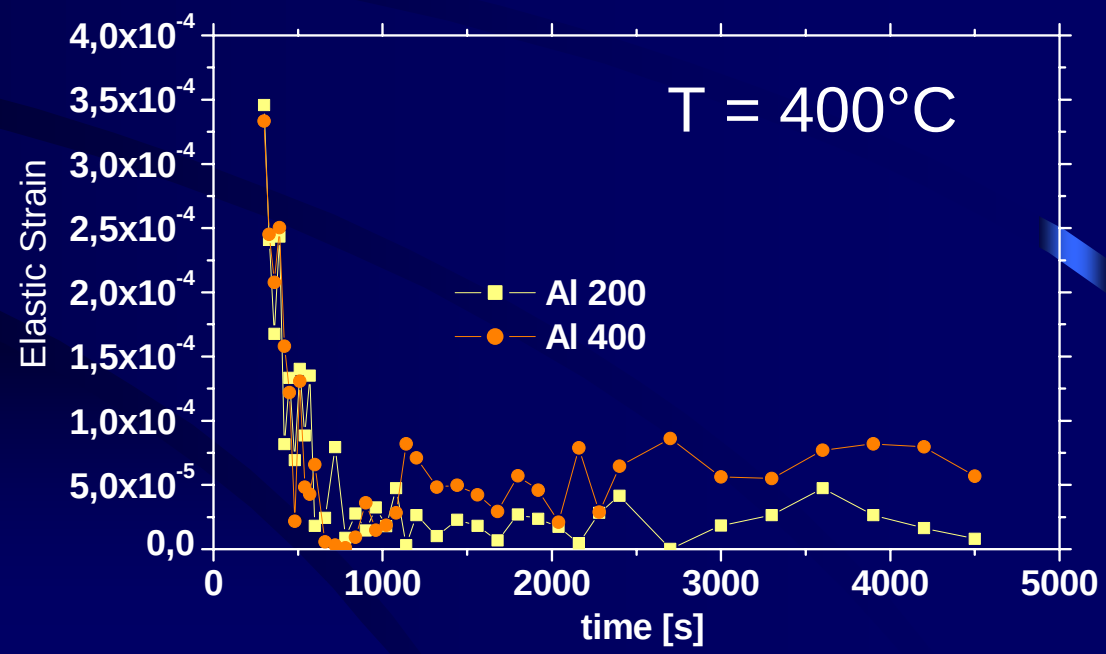
Coop. with A. Jacques et al., Nancy, France

Time - resolved Investigations



- Stress relaxation tests during tensile loading at elevated temperature
- material: AlSi25Cu4Mg1
- sample elongation: 1000 µm ~ 3,5 %
- acquisition time per spectrum app. 10s to 30s

- elastic strain in matrix and particles accessible at the same time
- in future even lower data acquisition times due to changes to the detector electronics and shielding



Further future applications of in-situ and time-resolved strain/texture analyses

- monitoring of „real processes“ such as welding, hardening, machining, ..
- layer growth, e.g. in PVD – processes, high temperature corrosion, ..
- degradation of materials, components e.g. during corrosion in aqueous media, hot gas corrosion, fatigue, thermal fatigue, ..
- solution and precipitation processes (e.g. in Al-alloys, Mg-alloys)
- phase transformations (e.g. in steels, multiphase materials with interdiffusion..)
- crystallization, recovery, recrystallisation processes ..
- ..

Methods, Requirements

- sample evironments for thermal and / or mechanical loading
- enough space at the beamline for setting up complex sample environments
- fast detection systems, read-out and data transfer systems: e.g. 2D-detectors multiple-detector systems
- long-term stability of the beam and the beamline components
- on-line and real-time data analyses for experiment control

Example 6: Fast in-situ tomography

Unpublished results

Combined SR Experiments

- complex materials and components where a multitude of properties has to be characterized simultaneously or at certain local spots
- strain and profile analyses: e.g. in strongly deformed materials
- strain and texture analyses: e.g. strains in highly textured materials such as thin layers, texture and intergranular stress formation, ..
- tomography + strain analyses: e.g. strain fields near cracks, strain fields near delaminations, strain fields around individual fibres and fibre bunches, ..
- strain / texture analyses and small angle scattering: changes in stress fields due to precipitation or solution (d_0 – problem remains), strain fields in materials with certain particle contents, e.g. dispersion hardened alloys (ODS – superalloys), ..
- texture and spectroscopy: influence of chemical composition on grain orientation ..

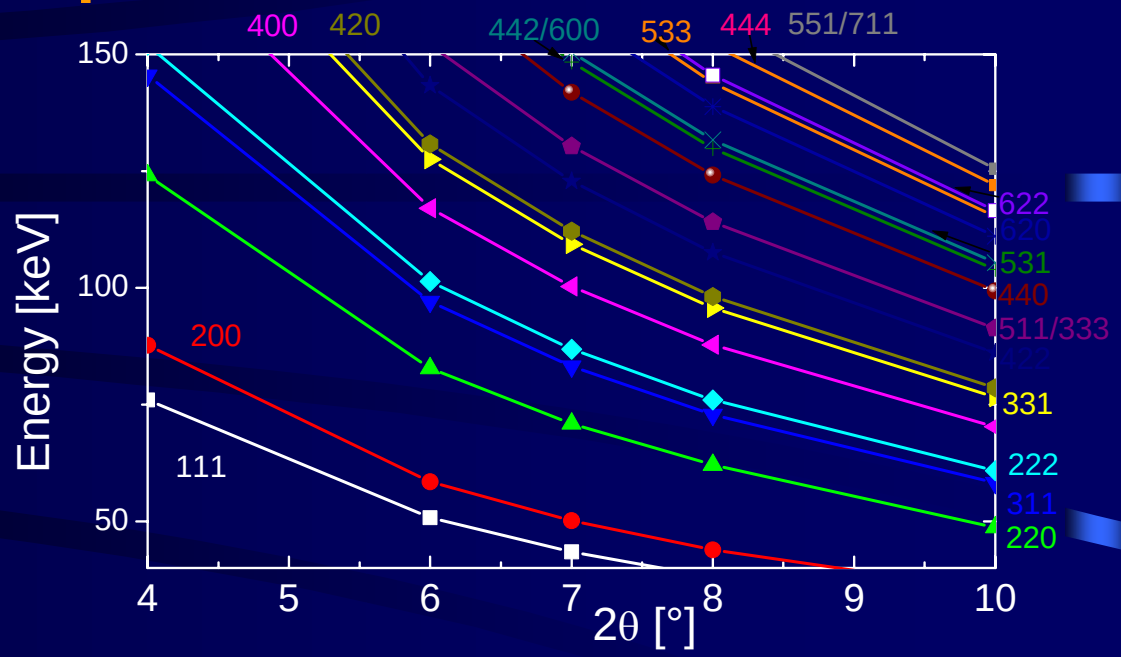
Methods, Requirements

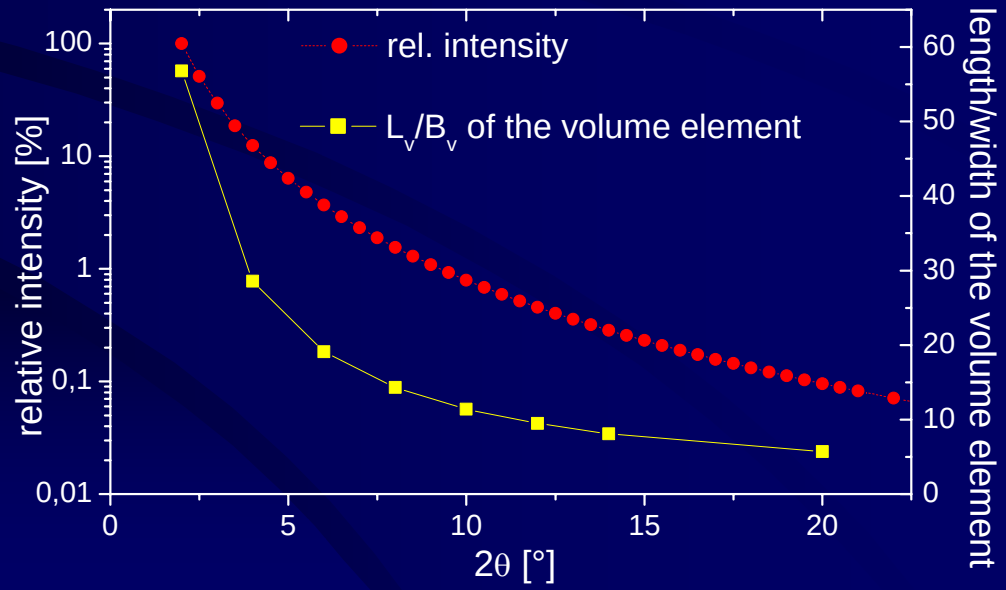
- beamlines that enable the use of both methods, e.g. strain analyses and tomography, small and wide angle scattering
- experimental set-ups including detector systems that allow simultaneous analyses with different methods or a fast switching between the methods

White High Energy Synchrotron Radiation: further possibilities I

multip[illegible]ector systems

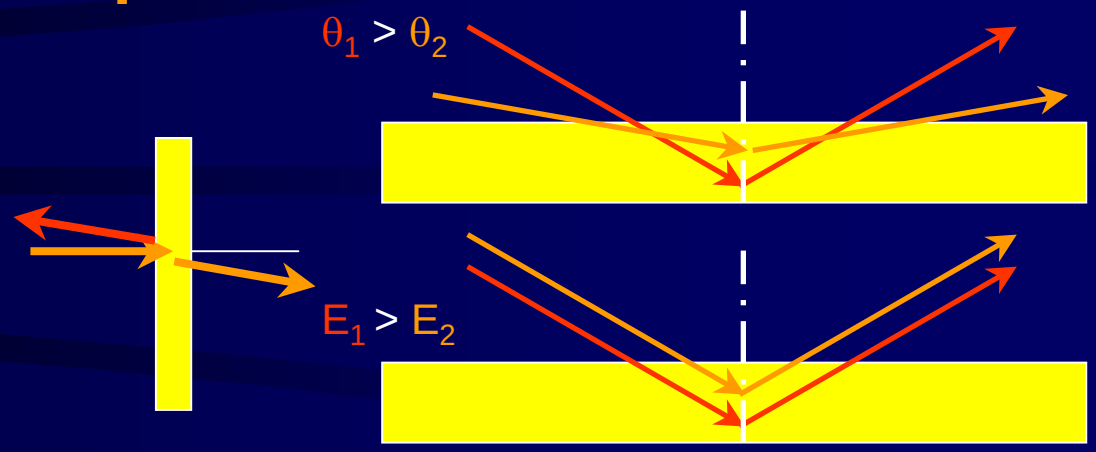
- increa[illegible] speed
- increa[illegible] information
- b[illegible] reachable for h[illegible]ergies





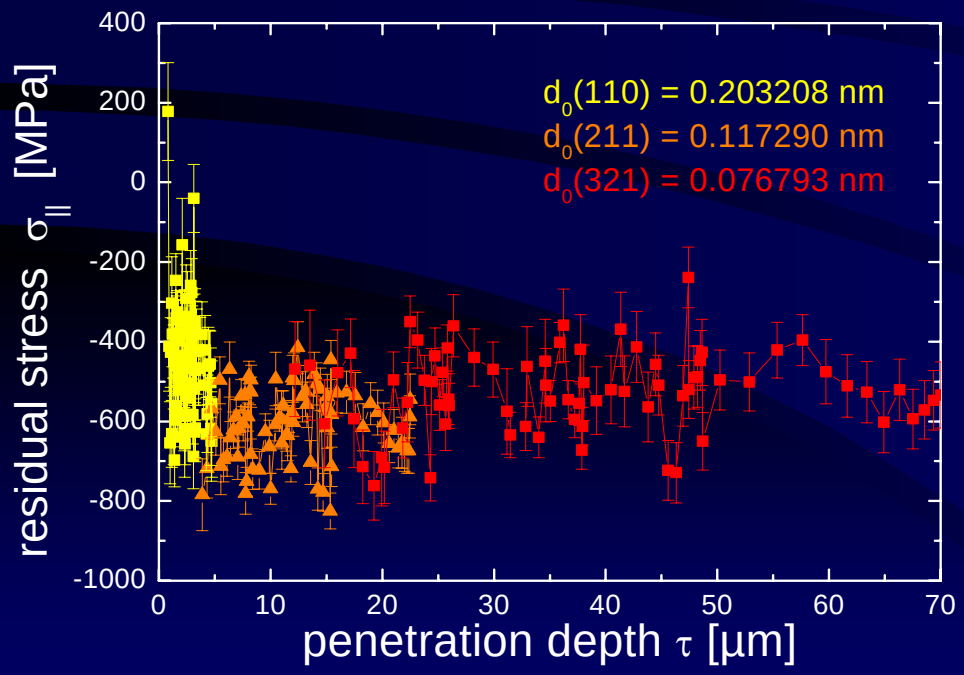
White High Energy Synchrotron Radiation: further possibilities II

- analyses in reflection mode using energies up to 60 keV
- non-destructive analyses of strain gradients in the near – surface - zone

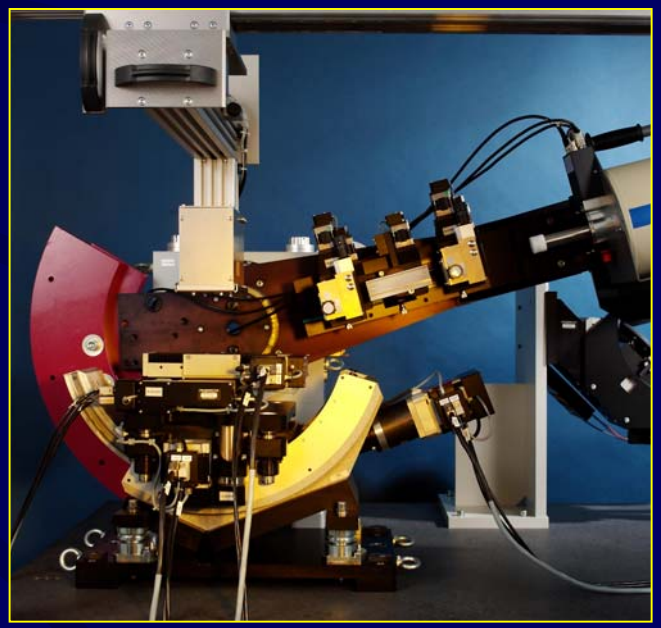


/G. Brusch, W. Reimers 1997/

Shot peened (90°) steel sample



/C. Stock, Ch. Genzel, W. Reimers 2001/



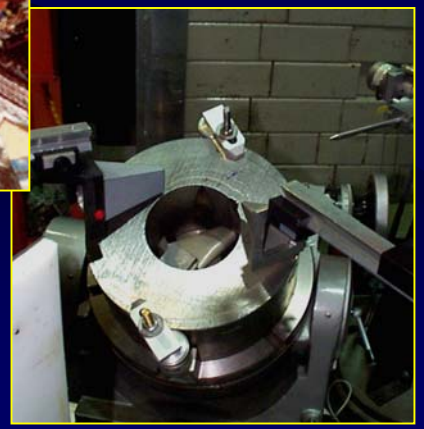
/C. Genzel, C. Stock, W. Reimers 2002/

Neutrons and HESR: Complementary Methods

- Wavelength
- Volume element shape and size
- Absorption
- Flux

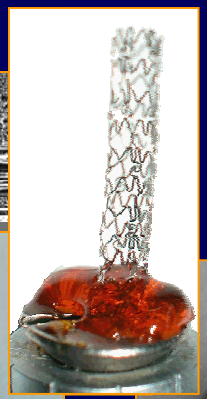
Neutrons

- high Z – materials
- coarse grained materials
- large components
- global textures
- low symmetry crystal structures
- …



HESD

- low Z – materials
- layered structures, buried interfaces
- small, regular shaped components
- in - situ analyses during thermal and mechanical loading
- time-dependent processes
- ..



Example 7: Characterisation of Sauropod Bones

Unpublished results

Conclusions and Outlook

In-situ Determination using Materials Behaviour using HESR

- high photon flux ⇒ short data acquisition time, small gauge volume
- high parallelism ⇒ small and well defined gauge volume
- large available energy range ⇒ penetration depth defined by photon energy, measurements at the surface and in the bulk

⇒ **microstructure, texture, strain / stress analyses in engineering materials and components**

⇒ **in-situ studies of material behaviour under external loading**

Outlook

⇒ strain and stress mappings at the surface / in the bulk of samples

⇒ stress / strain analyses in small components

⇒ strain distribution within individual grains, grain interactions

⇒ in-situ experiments: e.g. loading, annealing

⇒ ‘on-line’ data analyses (Rietveld, res. multi – reflection analyses)

⇒ combined experiments: e.g. texture+stress, strain+tomography

Strain and Texture Analyses using HESD

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