SCIENCE LINK

Baltic Sea Region Programme 2007-2013
Part-financed by the European Union (European Regional Development Fund)

2nd Science Link Conference

Chamber of Commerce and Industry

10.06.2013, St. Petersburg

Part-financed by the European Union
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Neutrons and Synchrotron Radiation in Construction and Engineering

Dr. Marc Thiry, Industrial Liaison Officer
Helmholtz-Zentrum Geesthacht (HZG)
10.06.2013, St. Petersburg

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SCIENCE LINK

GEMS
German Engineering Materials Science Centre
Helmholtz-Zentrum Geesthacht
Zentrum für Material- und Küstenforschung

Content:

- The German Engineering Materials Science Center
- Neutrons and Synchrotron Radiation in Construction and Engineering
    - Methods and Examples
- Summary

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The German Engineering Materials Science Center

GEMS
German Engineering Materials Science Centre
Helmholtz-Zentrum Geesthacht
Zentrum für Material- und Küstenforschung

GEMS is a central user access platform for complementary research with photons and neutrons in engineering materials science.

- HEMS (High Energy Materials Science Beamline)
- IBL (Imaging Beamline)
- BioSAXS Beamline
- Neutron Source FRM II (SANS-1, REFSANS, STRESS-SPEC, contribution to ANTARES)

Instruments using synchrotron radiation are operated at DESY in Hamburg, instruments using neutrons are located at the outstation at the FRM II in Garching near Munich.

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High Energy Materials Science Beamline (HEMS)

- Texture determination
- Residual stress analysis
- Characterisation of nanostructures with high energy small-angle X-ray scattering (HE-SAXS) experiments
- Phase transformation analysis
- In-situ sample environments

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The German Engineering Materials Science Center

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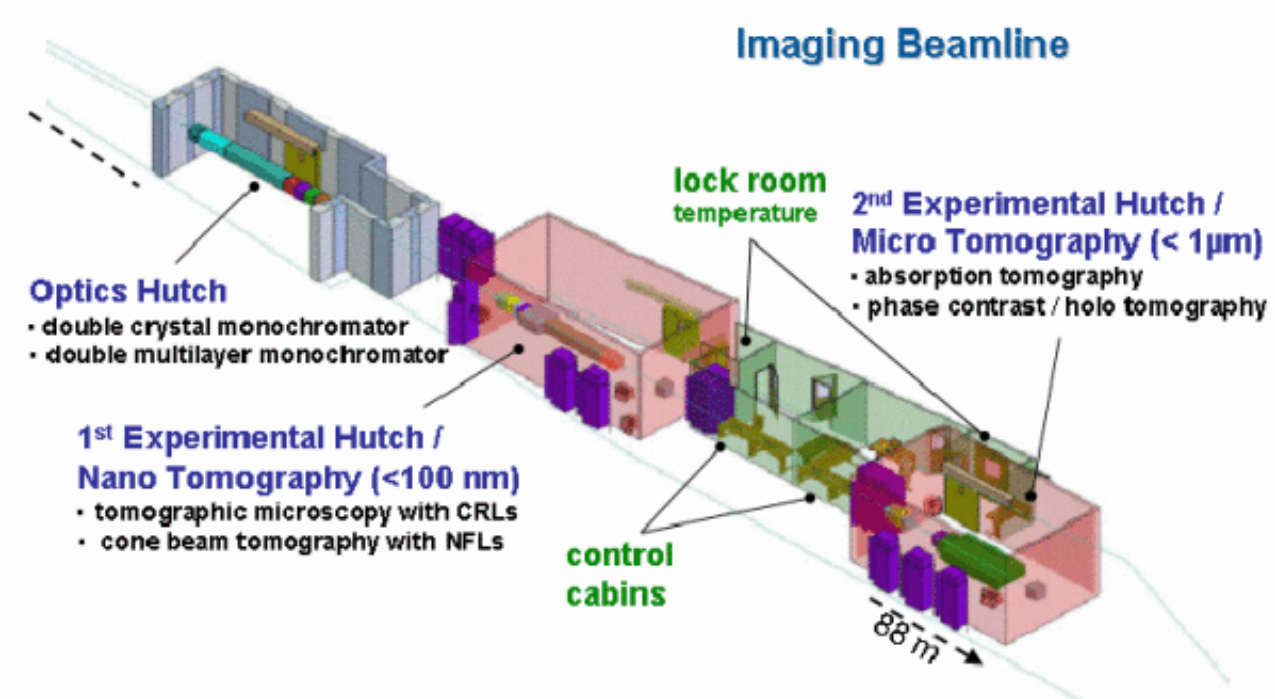
- IBL: Tomography of engineering materials



- BioSAXS: Structural research on soft matter samples (biomolecules, polymers, vesicles, micelles…)

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In situ sample environments

- Furnaces
- Cooling devices
- Tensile strain measurements
- Dilatometer
- Laser and stir welding environments

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GEMS – Neutron Experiments

FRM II:

- small angle neutron scattering (SANS1)
- reflectometer and evanescent wave small angle neutron spectrometer (REFSANS)
- residual stress analysis, texture determination (STRESS-SPEC)
- neutron computerised tomography (NCT) (contribution to instrument ANTARES)

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Additional GEMS services

- Custom in-situ sample environments (stir and laser welding, dilatometer)
- Sample Preparation (including FIB)
- Expertise of the Institute of Materials Research at HZG
- Integrated beamtime proposals for photons and neutrons
- Laboratory infrastructure: lab X-ray diffractometer (RöDi), Tomography (Nanotom), SAXS (Nanostar)
- Industry specific user support (data analysis, secrecy agreements)

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X-Ray tomography: 3-dimensional analysis of engineering materials

- X-ray Imaging has been used in medical diagnostics for over 100 years
- Development of the first anodes for clinical use in Hamburg around the end of the 19th century
- Many advancements have been made since in the use of X-rays in medicine as well as in materials science

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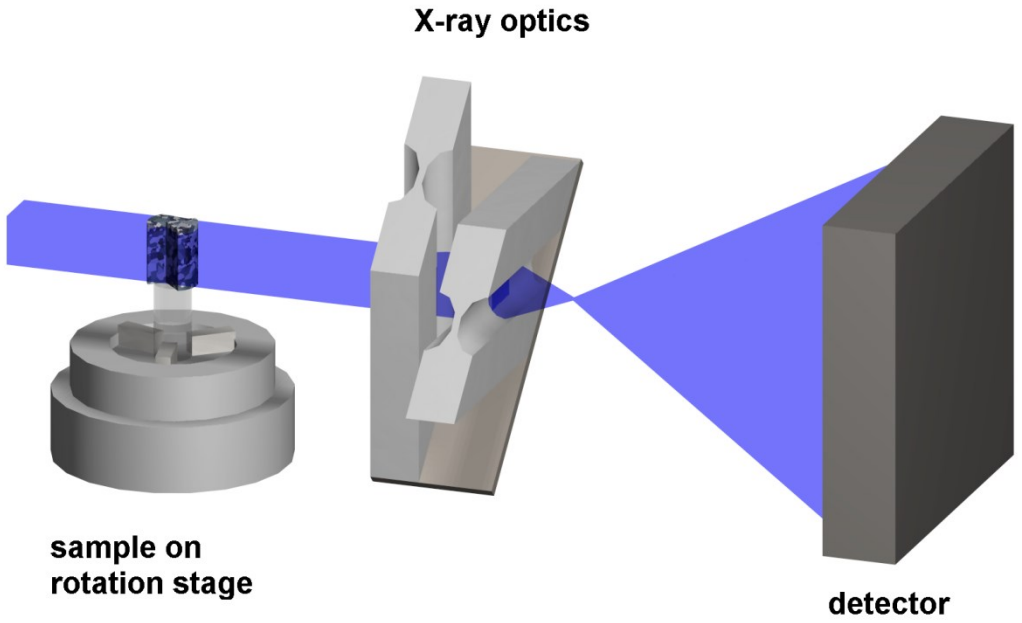
X-ray tomography: 3-dimensional analysis of engineering materials

- Rotation of the sample in the X-ray beam
- 3-dimensional image is created by computer software



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X-ray tomography: 3-dimensional analysis of engineering materials

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Helmholtz-Zentrum Geesthacht

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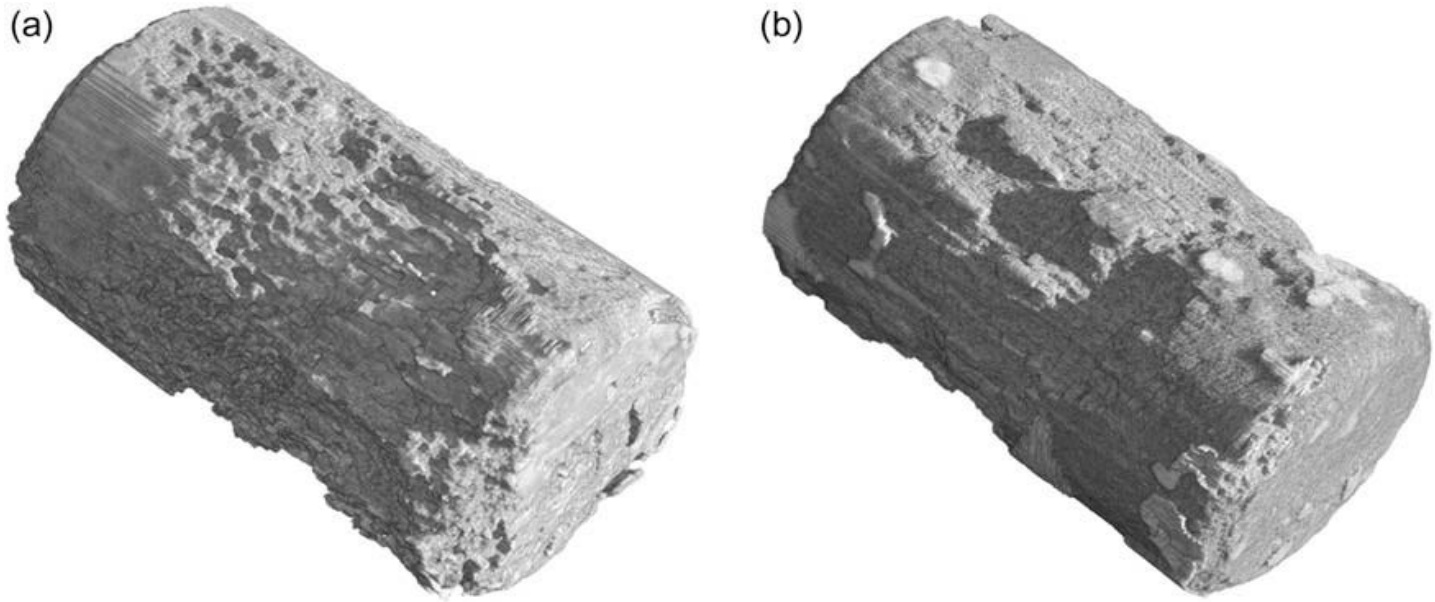
Corrosion morphology of *in vivo* corroded magnesium alloy



- Regular pattern of pitting corrosion
- Reduced, more uniform corrosion with singular deep pits

F. Witte, J. Fischer, J. Nellesen, C. Vogt, J. Vogt, T. Donath, F. Beckmann
Acta Biomaterialia **6** (2010), 1792-1799

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X-ray tomography: 3-dimensional analysis of engineering materials

German Engineering Materials Science Centre

Helmholtz-Zentrum Geesthacht

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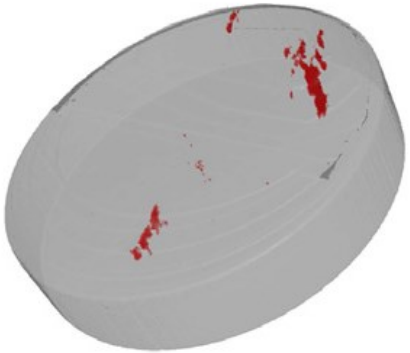
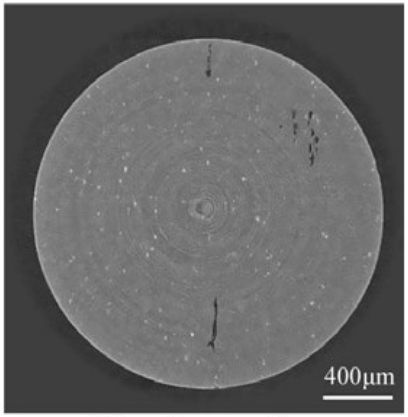
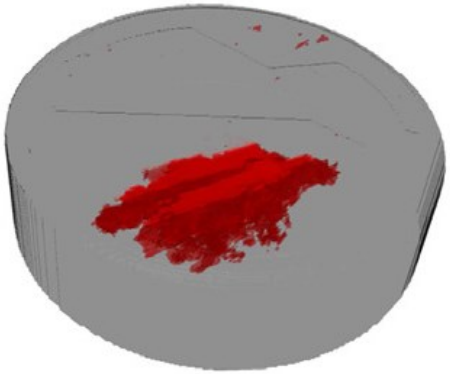
Study of intergranular corrosion in aircraft aluminium alloys using X-ray tomography



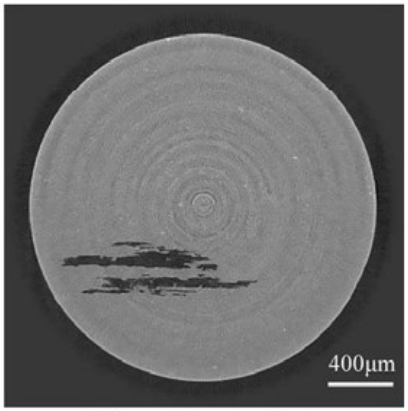
48 h



58µm from surface



168 h



115µm from surface

S.P. Knight et al. / Corrosion Science 53 (2011) 727–734

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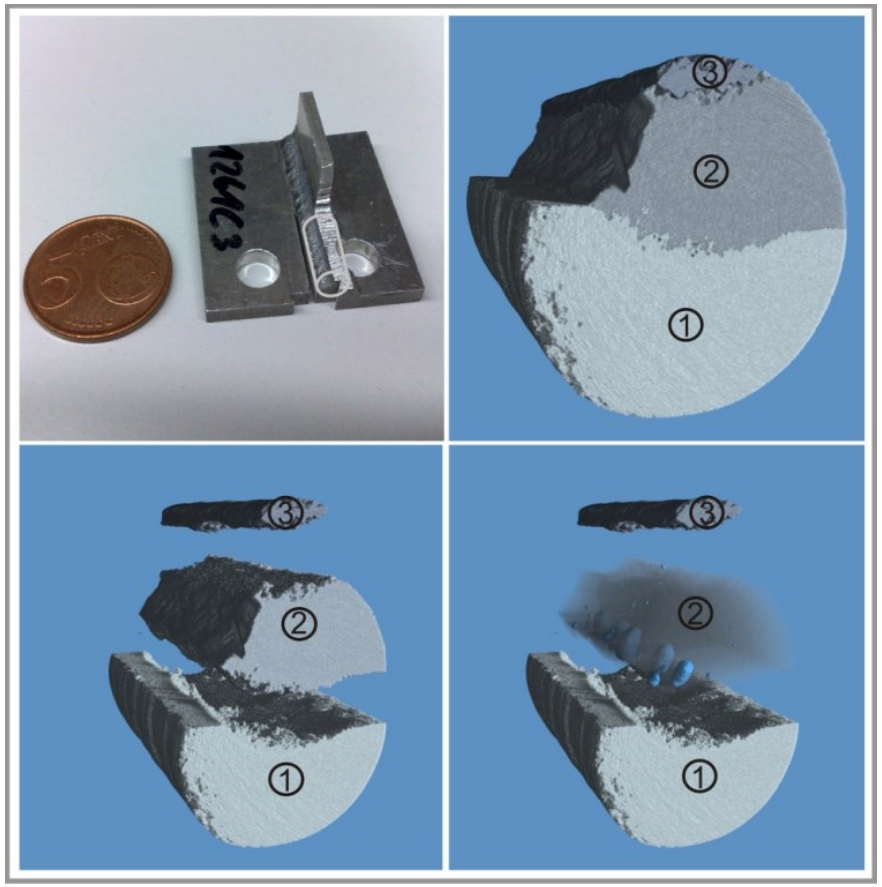
X-ray tomography: 3-dimensional analysis of engineering materials



Pores in Al laser beam welds

→ very high density resolution achieved, different Al alloys can be distinguished

J. Herzen et al., *Proc. of SPIE* **7078,** 70781V (2008)

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Neutron radiography

Pyrotechnology in Space:

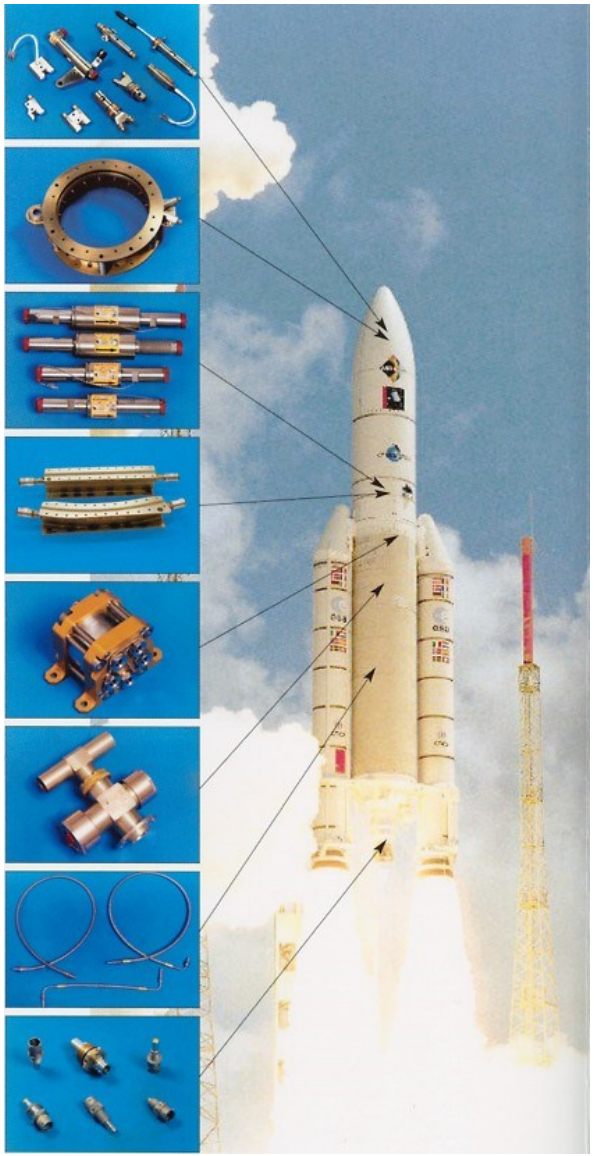
Ca. 800 devices on board of

ARIANE 5

for separation of components

e.g. cable cutters for steel ropes of up to 10 mm tickness.

Devices include modern explosives (e.g. Hexogen)



Quality assurance:

Every single device has to be tested before launch for

- Sufficient density
- Homogeniety

of the explosive

=> Neutron Radiography

- Large penetration
- Good contrast for light elements, esp. hydrogen rich materials

Significant portion of the production for Ariane 5
checked at GeNF since 2004

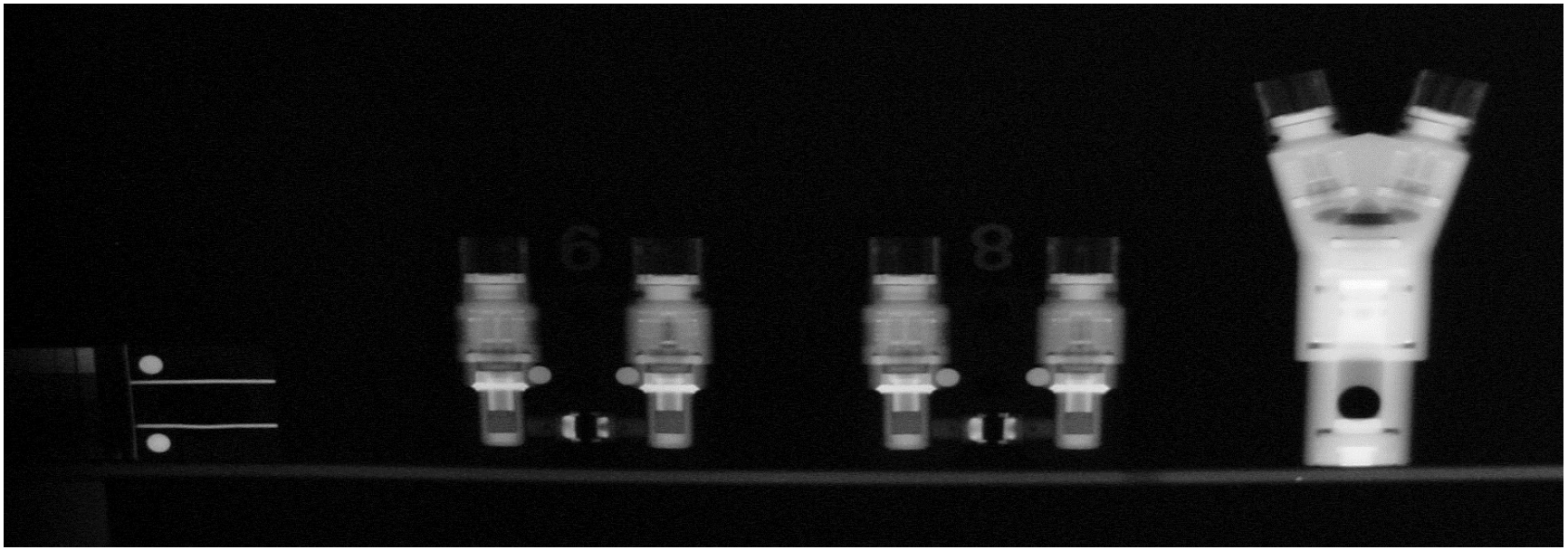
Part-financed by the European Union (European Regional Development Fund)

J. Vollbrandt, H.-W. Schmitz, GKSS; Fa. Dassault Aviation, Argenteuil, France

Neutron radiography

Initiators

Cable cutter



Advantage of Neutrons:

- Large penetration depth
- Sensitivity for light elements

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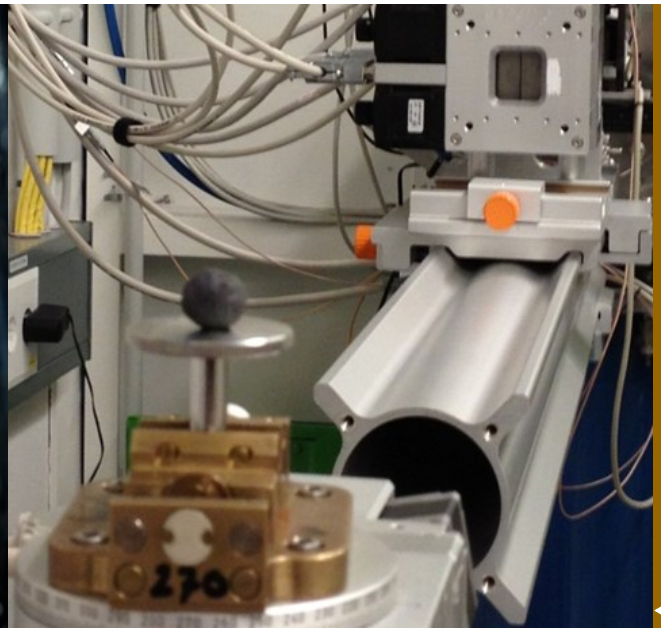
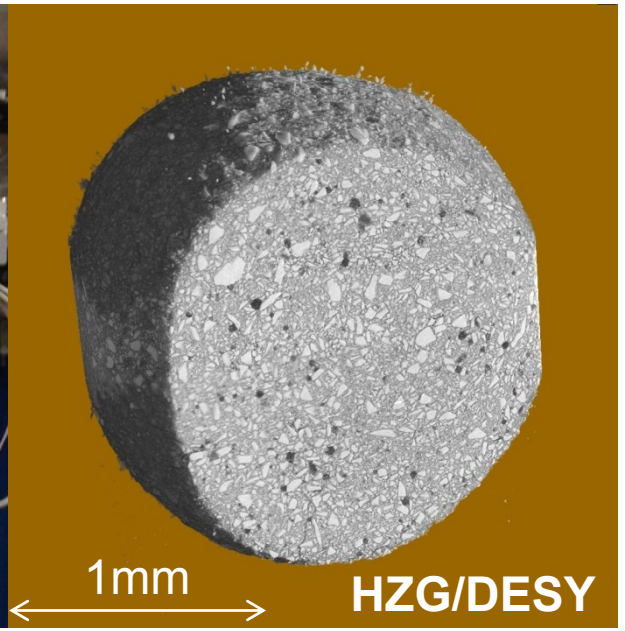
Microtomography of iron ore pellets

(LKAB)

- The use of additive mixes in pellets production leads to increases in steelwork's productivity while energy use, wear and slag are reduced.
- Knowledge about porosity and grain sizes in the pellets will help in further development and optimization of the product





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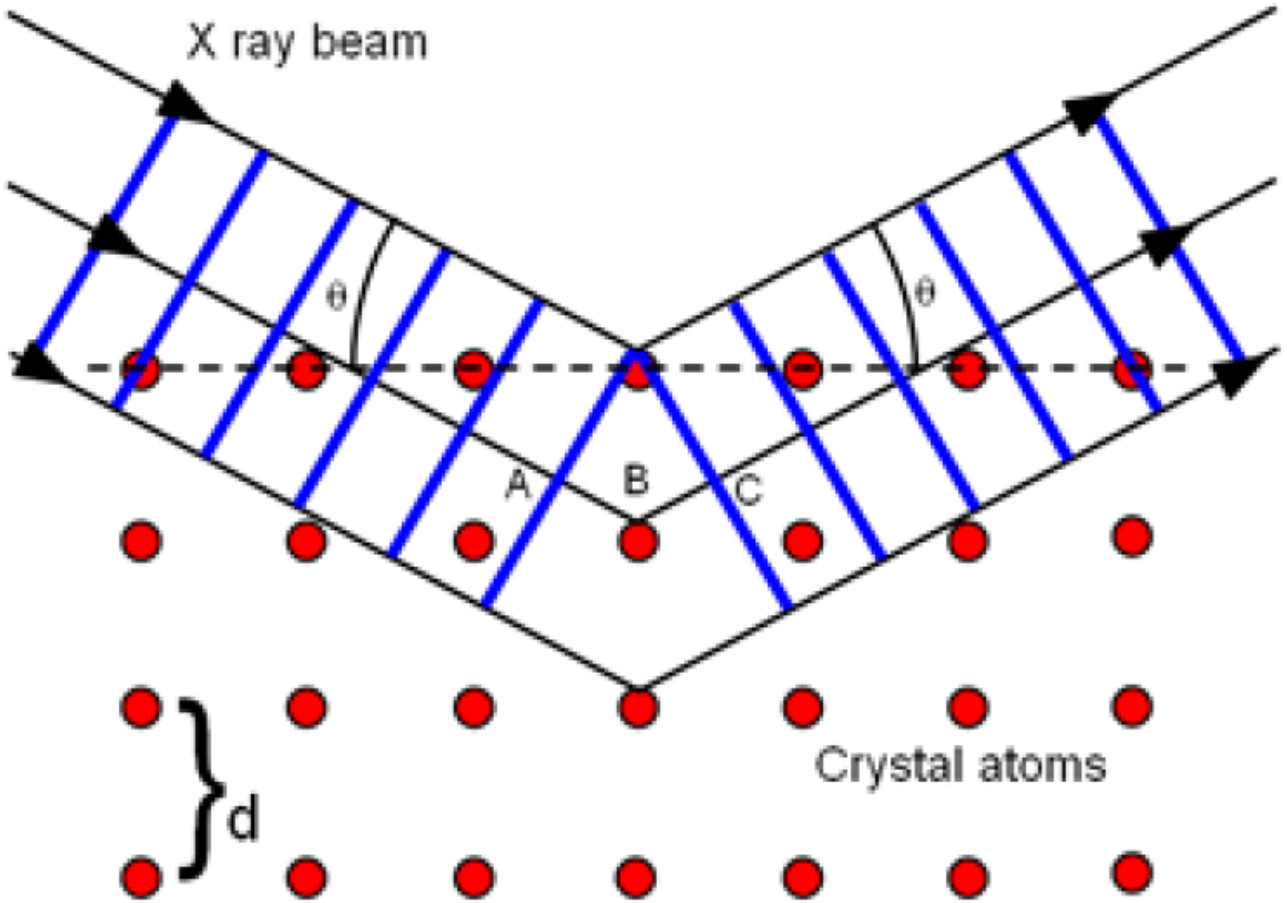
X-Ray and Neutron Diffraction



Interplanar distances are of same length as X-ray wavelength

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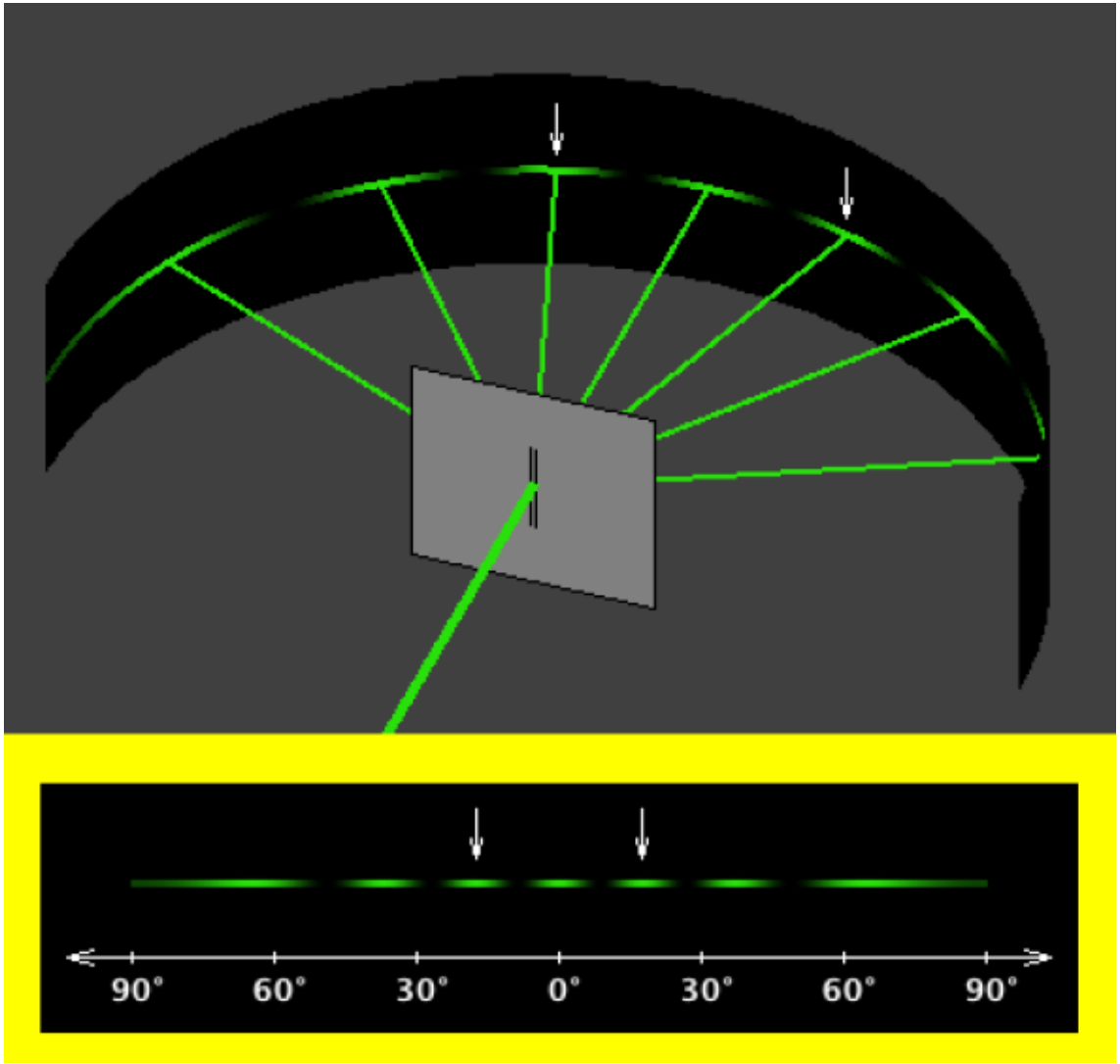
X-Ray and Neutron Diffraction

Interference of light at a double slit



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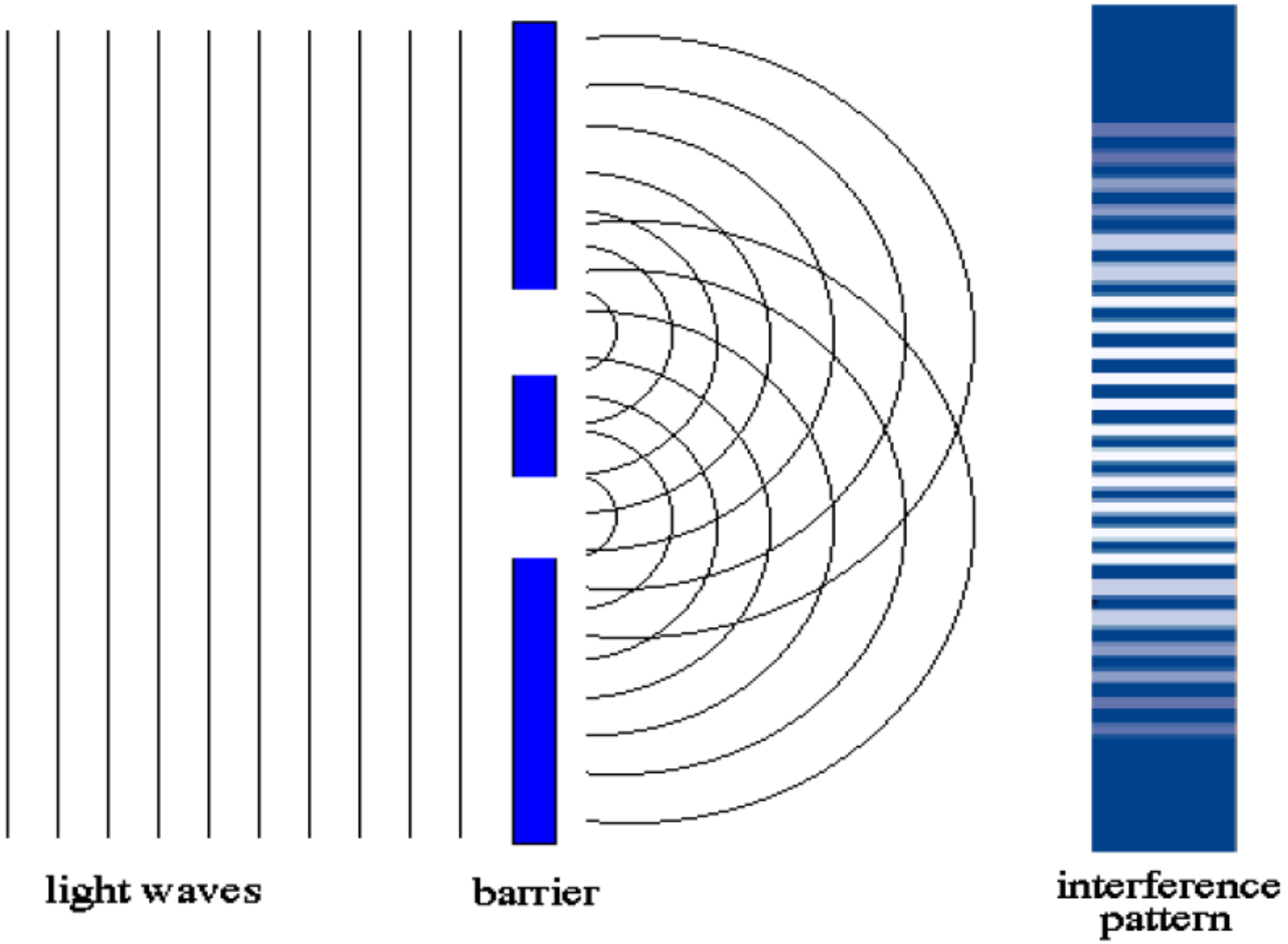
X-Ray and Neutron Diffraction

Interference:



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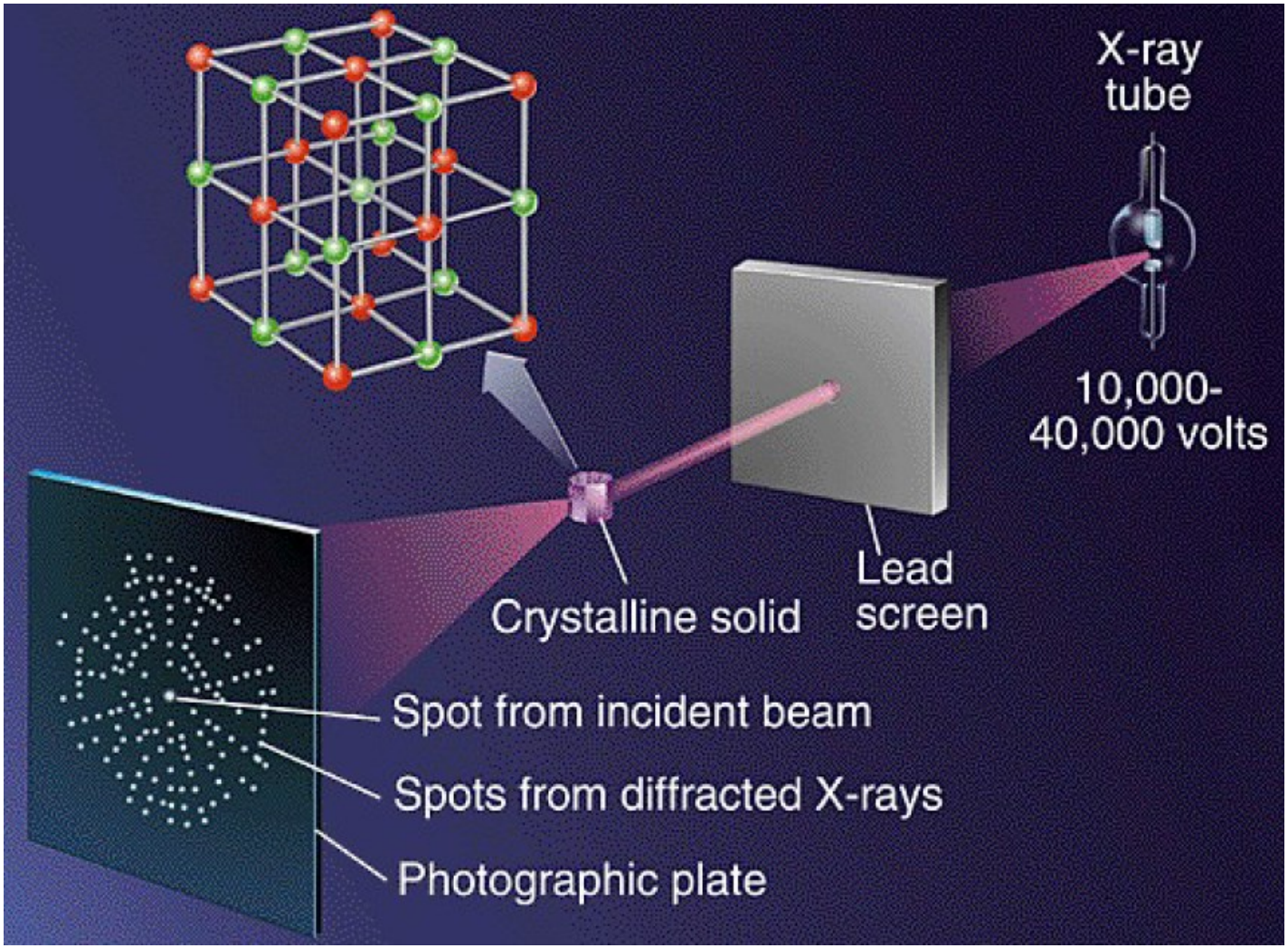
X-Ray and Neutron Diffraction



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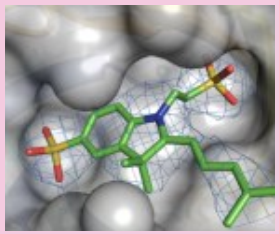
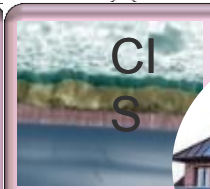
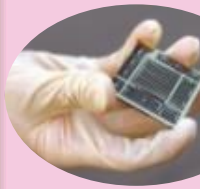
INDUSTRIAL APPLICATIONS





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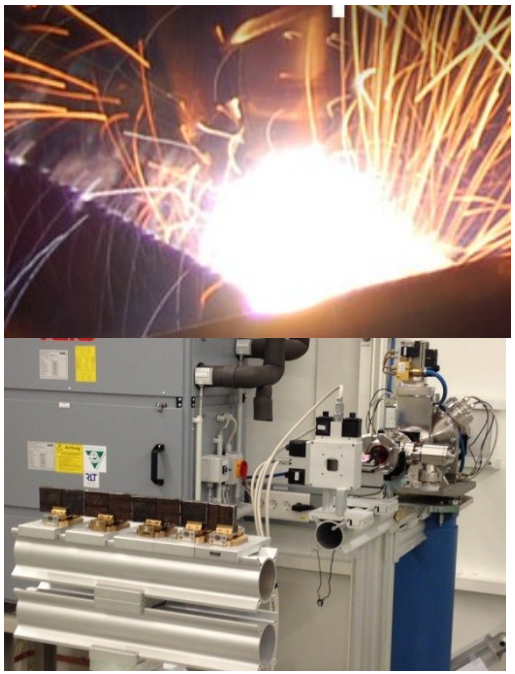
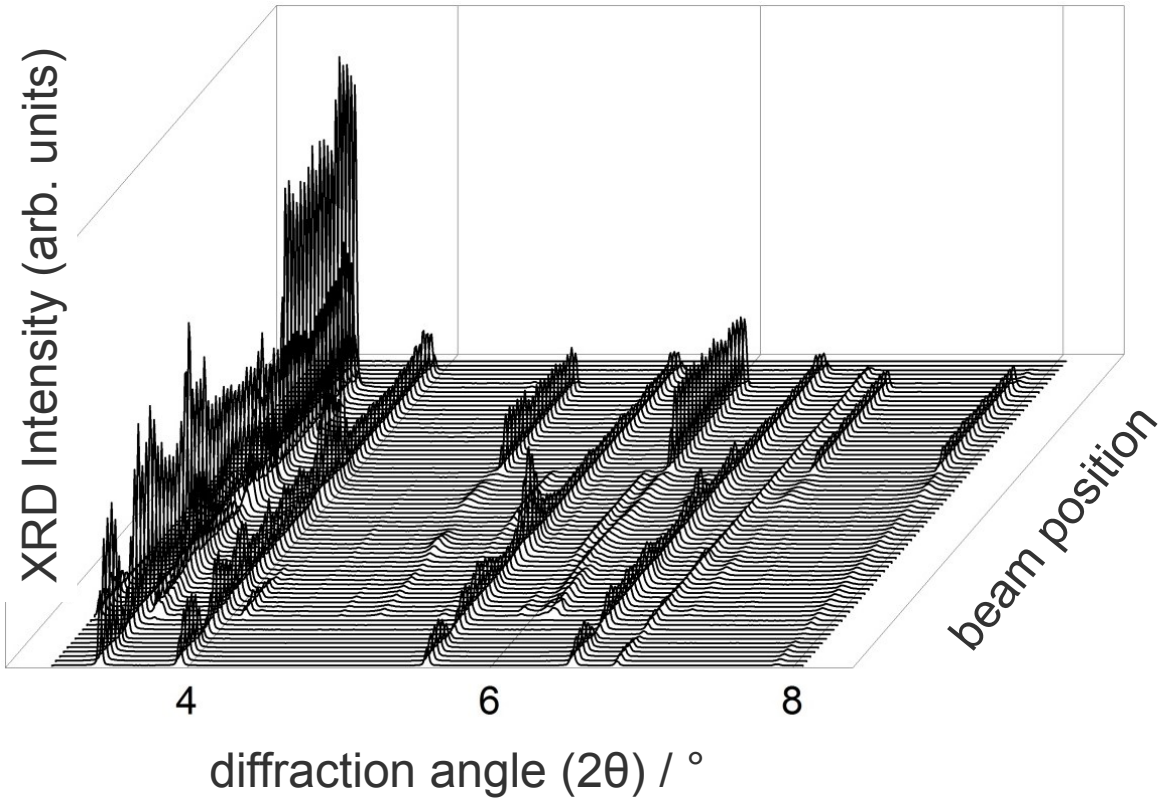
Phase analysis and residual stresses in welded steel plates – Ykkösmetalli

- The influence of welding of on residual stresses in steel plates used for tunnel drillng machines in mines was investigated by XRD at **P07 (HEMS by HZG/DESY)**

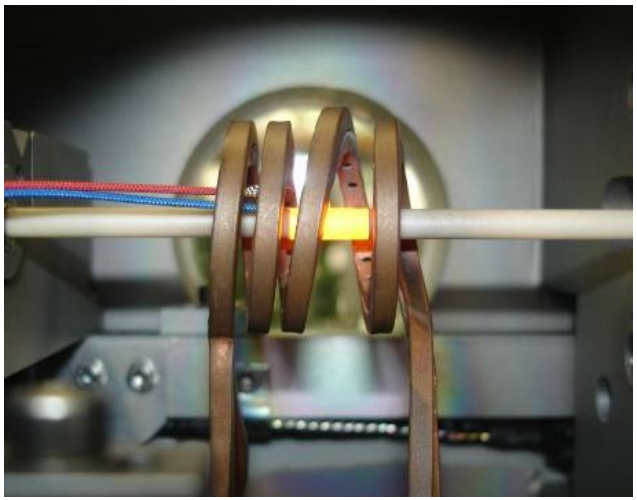


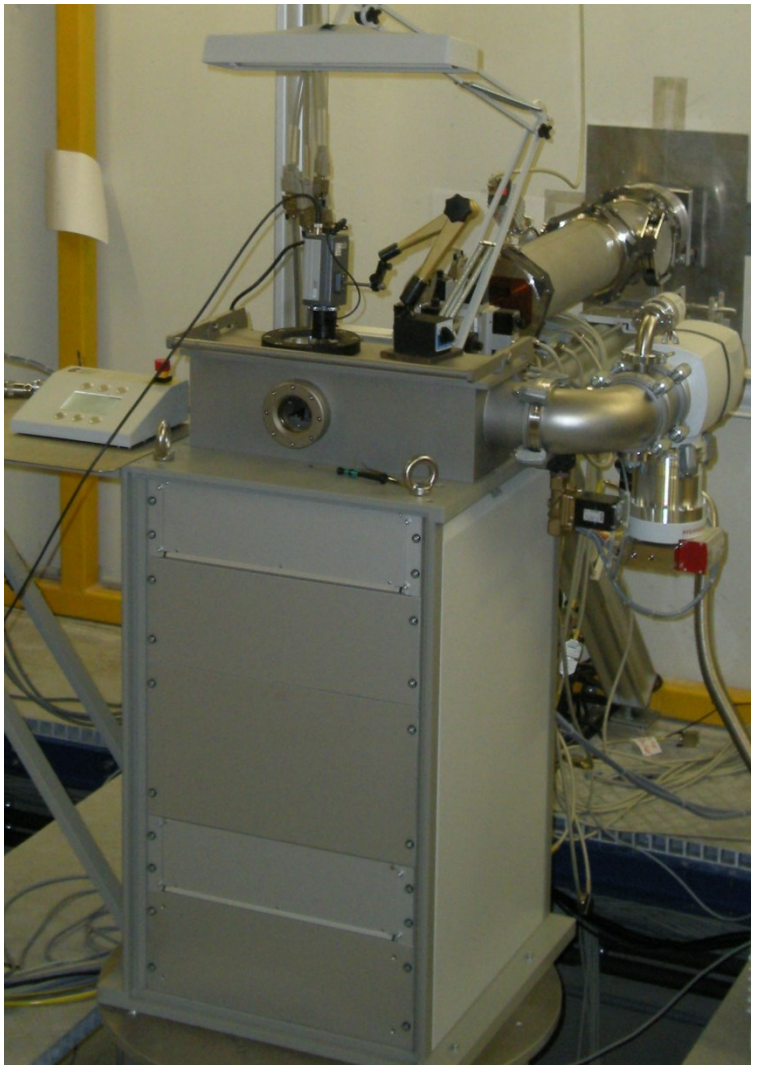


Part-financed by the European Union
(European Regional Development Fund)

Dilatometer for *in situ* studies of phase and microstructure transformations

- commercial dilatometer (Bähr 850 A/D)
- induction heating:
  – max. heating rate 4000 K/s
  – max. cooling rate 2500 K/s (hollow samples)
- DSC unit
- deformation units (25 kN)





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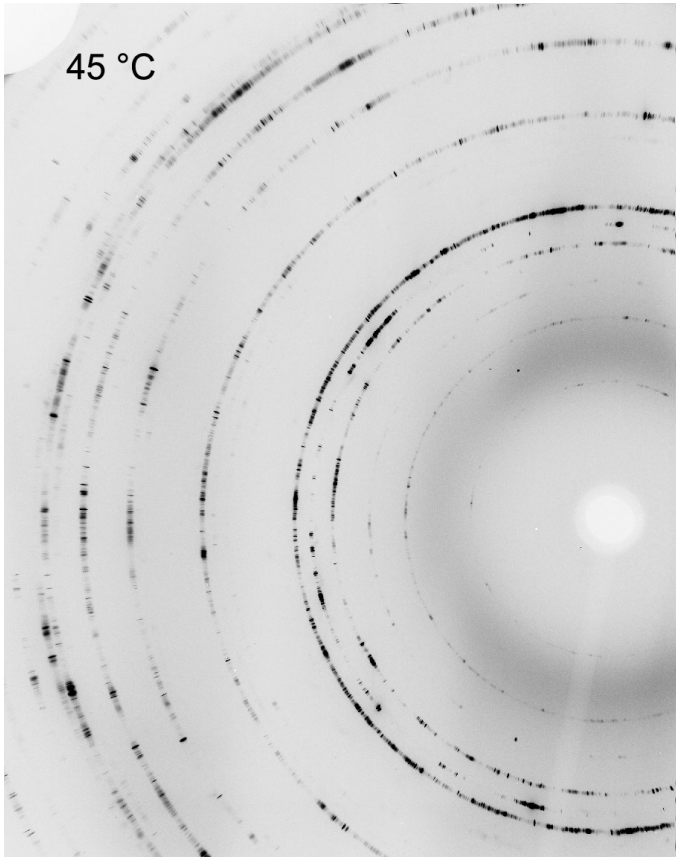
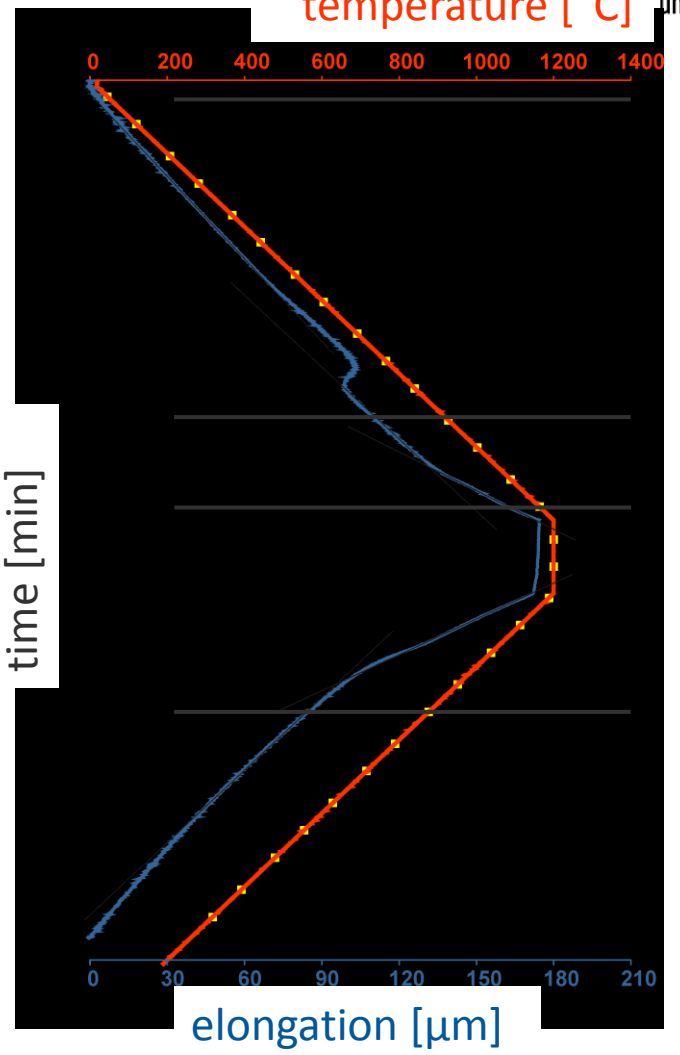
Dilatometer for *in situ* studies of phase and microstructure transformations





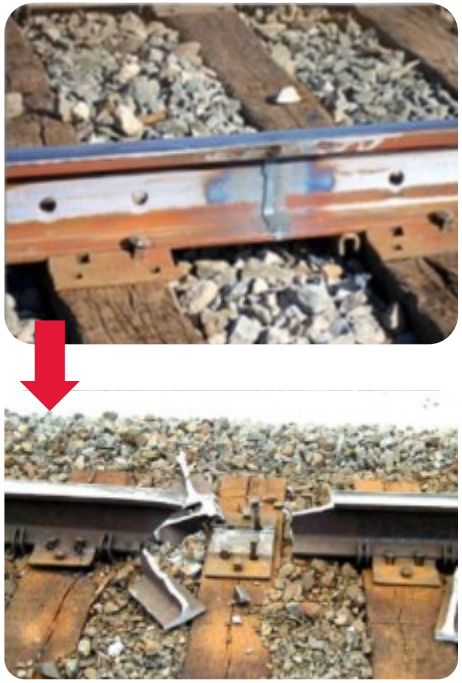
Ti-43Al (at.%), cast material, synchrotron experiment

Part-financed by the European Union (European Regional Development Fund)

A. Stark, M. Oehring, F. Pyczak, A. Schreyer, Adv. Eng. Mater. 13 (2011) 700–704

Stress and strain analysis

Hydrogen embrittlement



Service failure originating in a weld.
Source: Journal of Wheel/Rail Interaction

Residual stresses are almost always present in technical parts.

They superpose the load stresses occurring under service conditions.

Residual stresses can be adjusted in the manufacturing process "residual stress-engineering".

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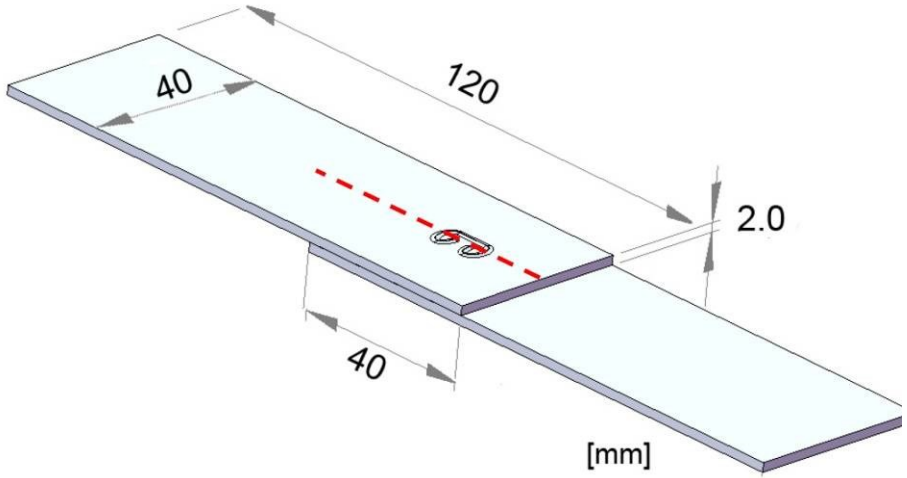
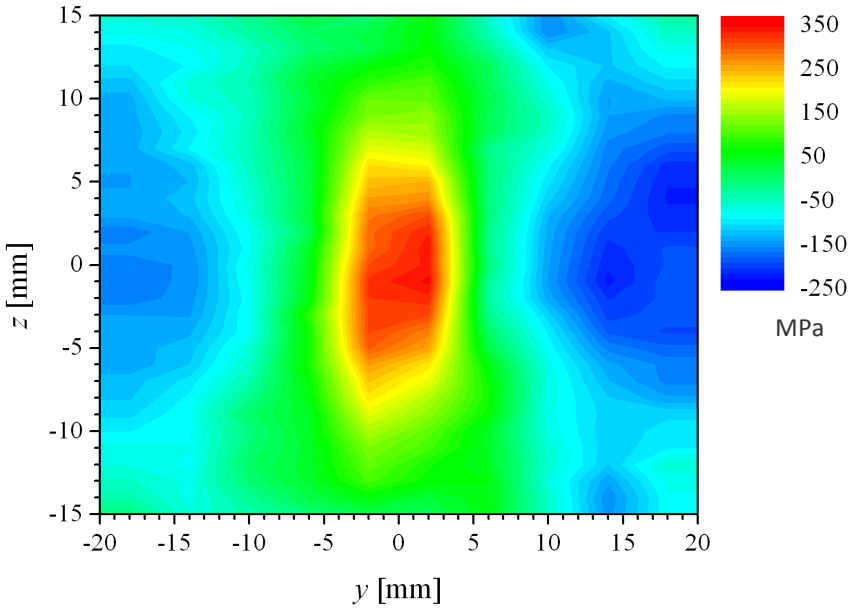
X-ray diffraction on laser spot welds

Problem: Depth resolution required for residual stress analysis with high-energy X-rays in overlap joints to distinguish between the two sheets



„conventional“ stress map



Solution: Conical Slits

With X-ray beam diameter <50 µm depth resolution <1 mm possible

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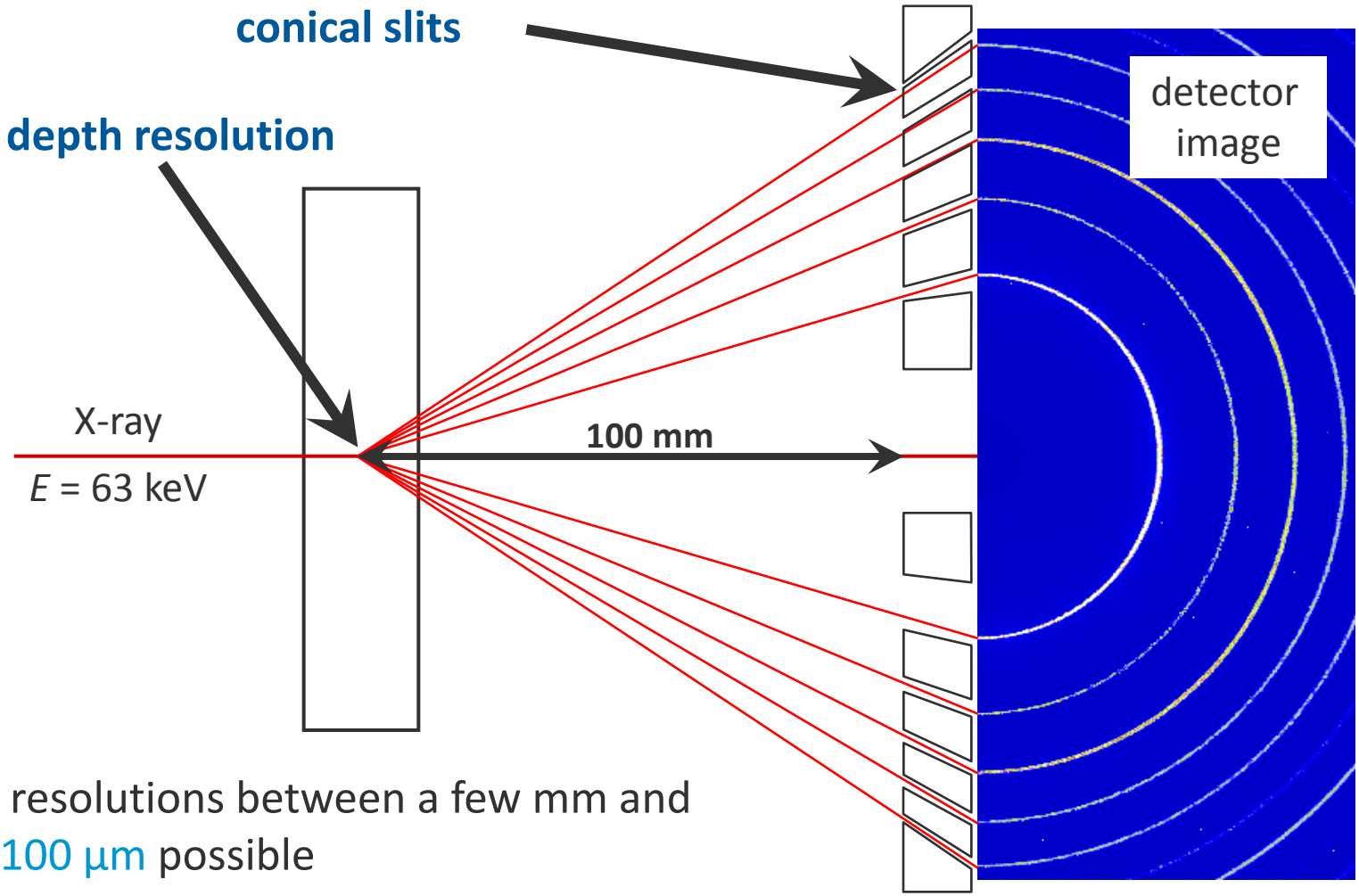
Depth resolution with conical slits



depth resolutions between a few mm and a few 100 µm possible

Part-financed by the European Union (European Regional Development Fund)

T. Fischer, P. Staron, A. King, N. Schell

Residual stress properties for high strength lightweight components

- **Volkswagen** develops new light weight components for engines
- Some aluminium alloys require a heat-treatment step for the hardening process
- Following heat-treatment, a high level of residual stress is produced which can decrease the lifetime of the component
- Simulations have been made to predict the stresses and increase the reliability in the durability prediction

Part-financed by the European Union
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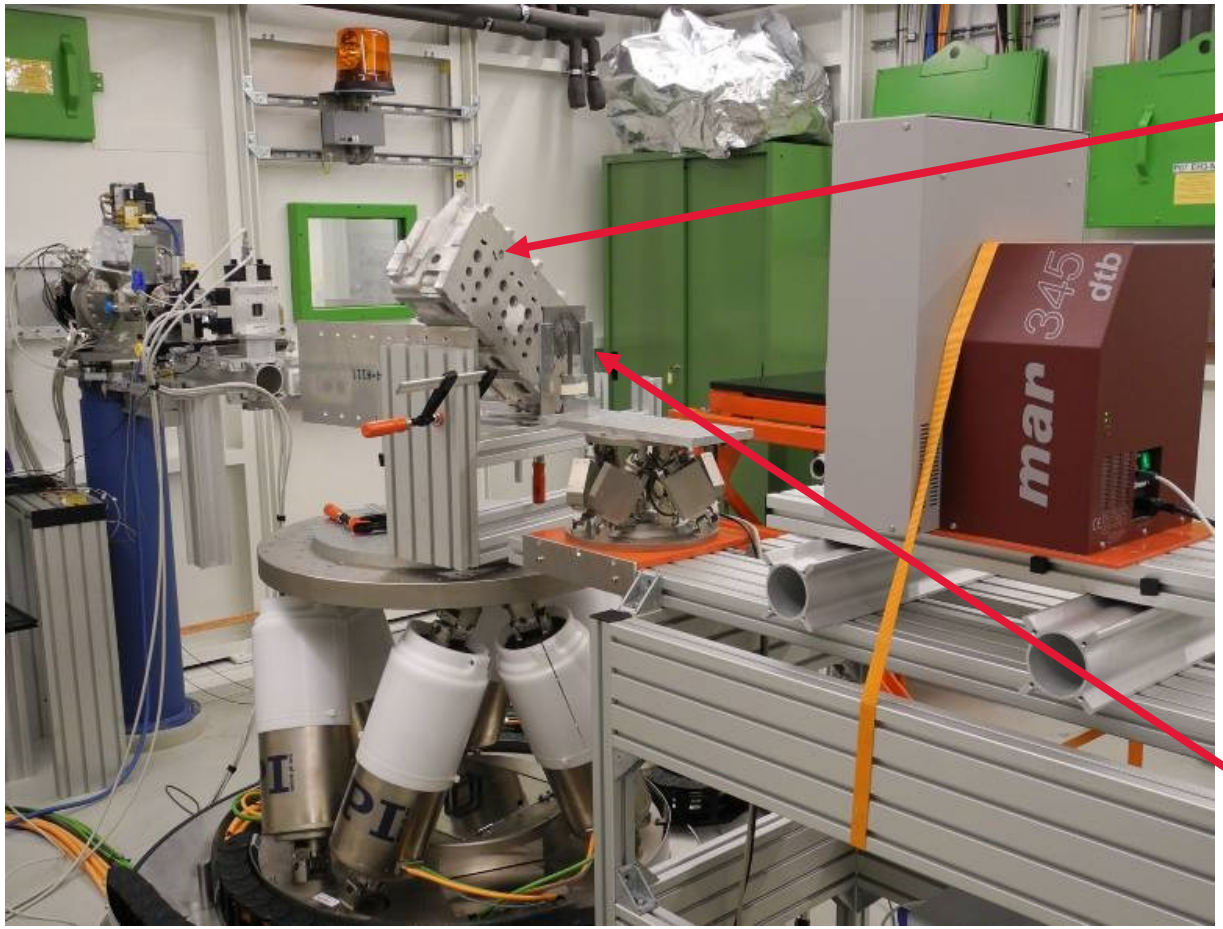
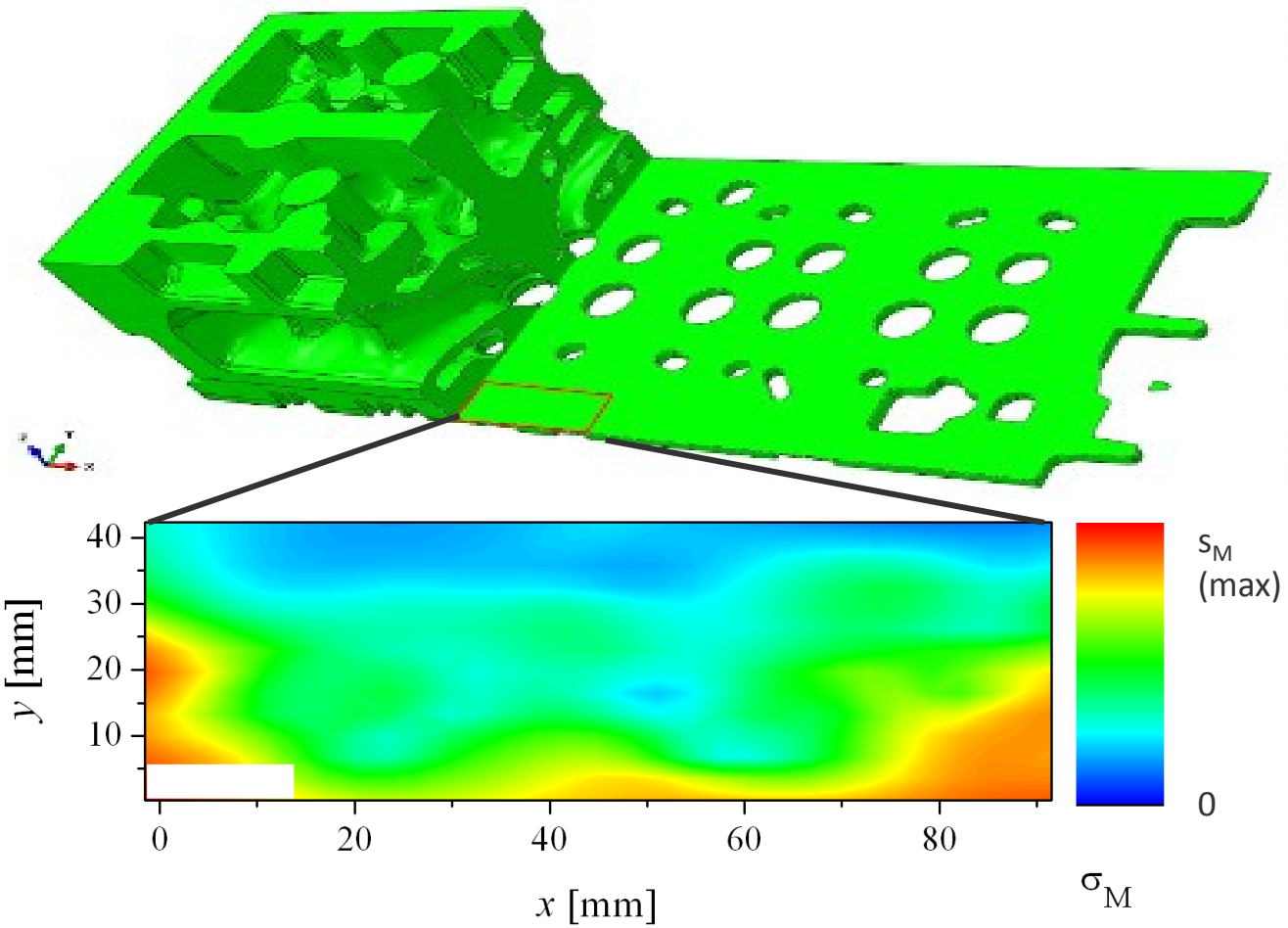
Aluminium alloy sample mounted at HZG beamline HEMS at PETRA III (DESY):



Depth resolved residual stress measurements were performed using the conical slit technique.

Part-financed by the European Union (European Regional Development Fund)

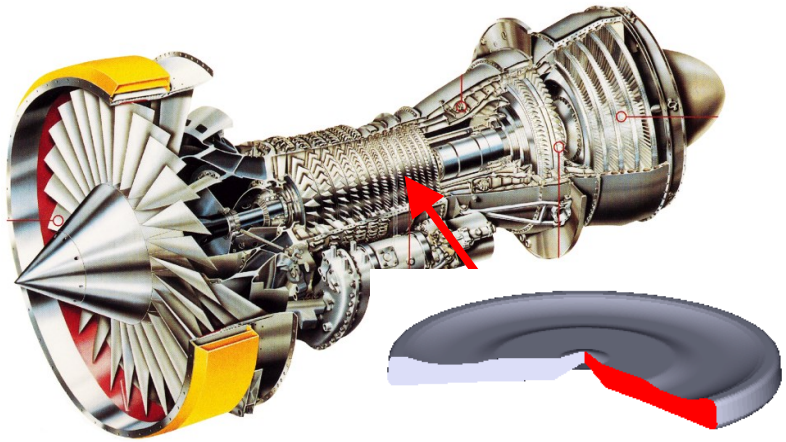
Depth resolved stress pattern:

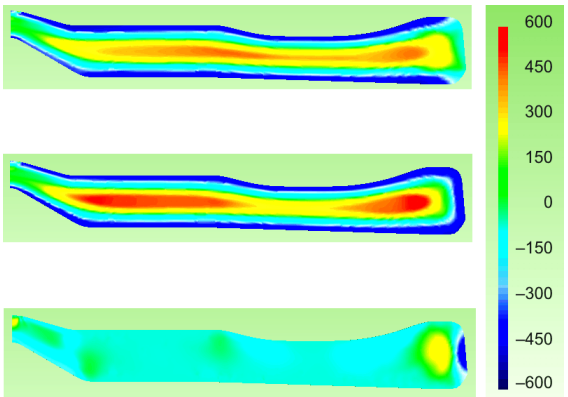


Experimental results of the project in Science Link validated the simulations made by Volkswagen

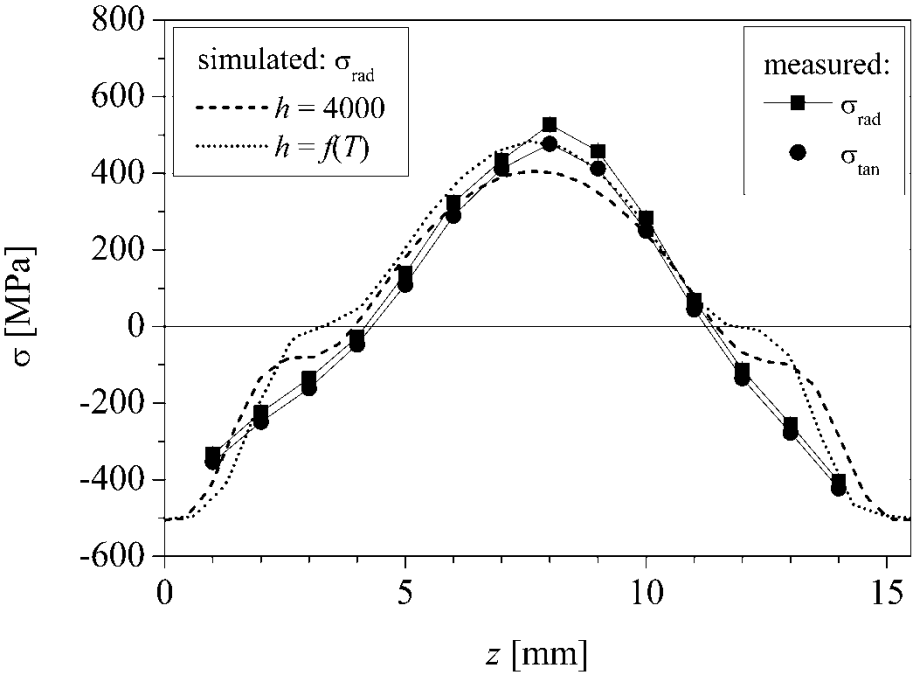
Part-financed by the European Union (European Regional Development Fund)

Residual stresses in forged aircraft turbine compressor discs





Simulated stress pattern



Simulated and measured stress distribution

Stress measured by neutron diffraction

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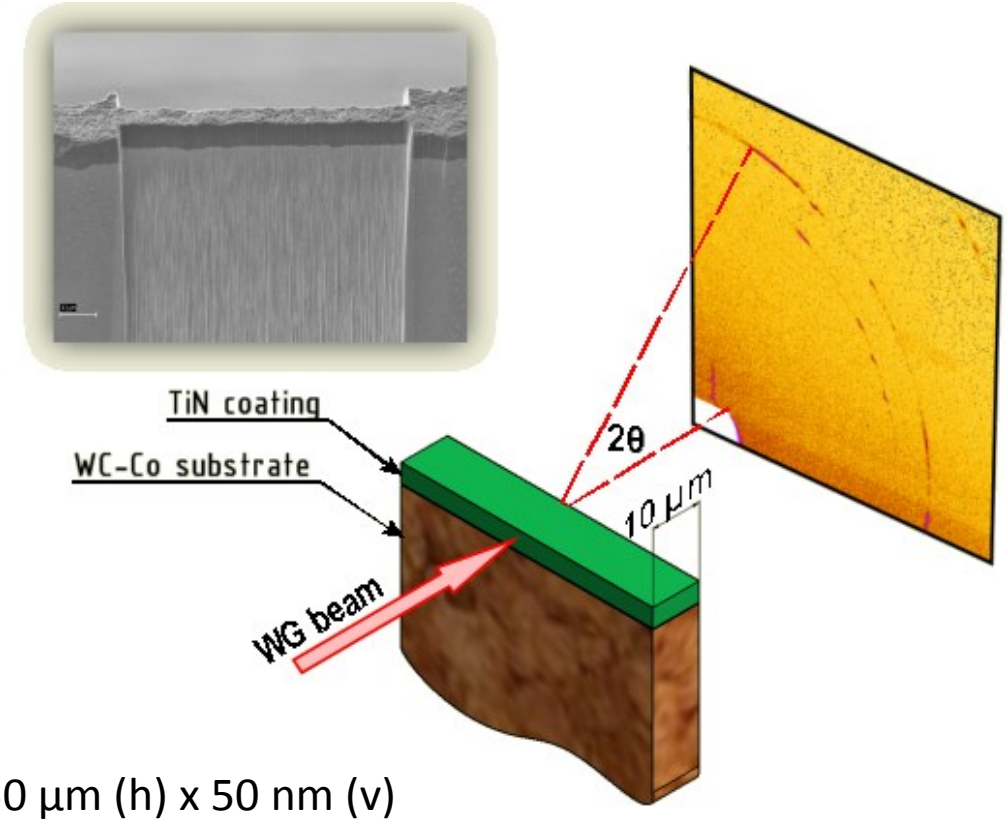
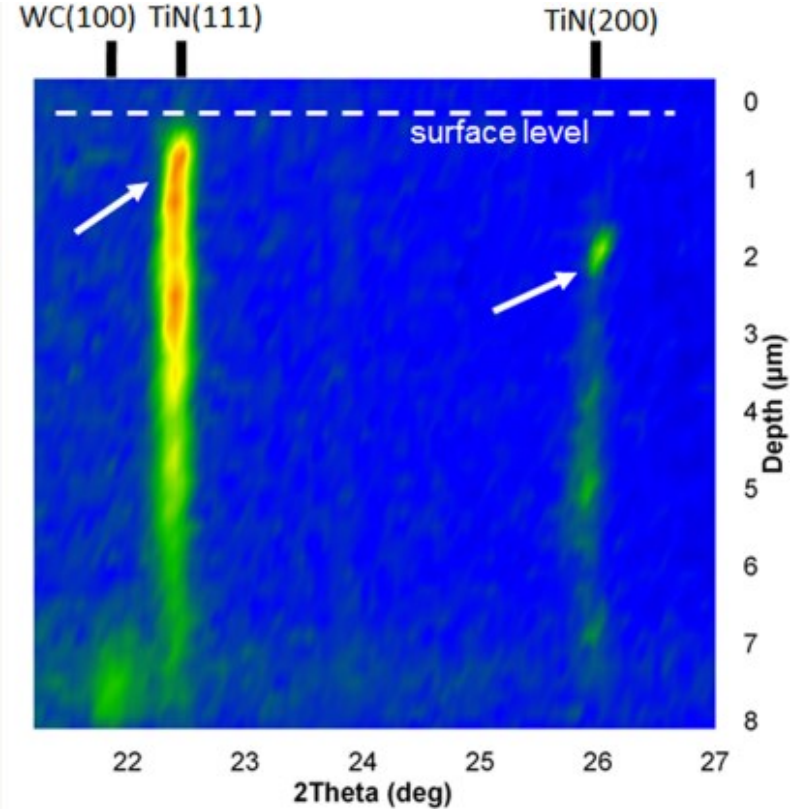
Residual stress in TiN coating on WC-Co





50 µm (h) x 50 nm (v) waveguide beam

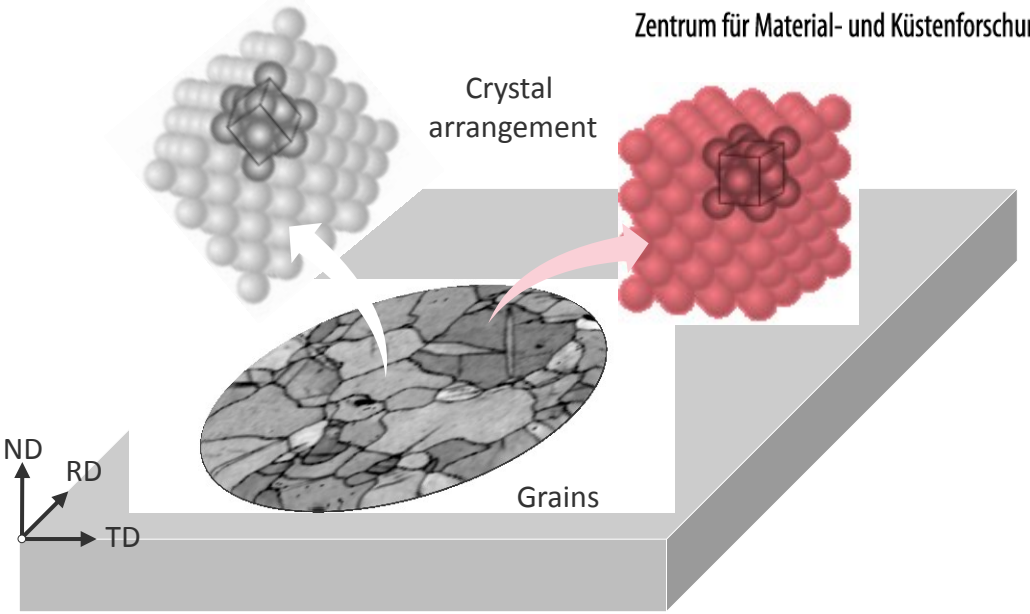
variation of microstructure and strain with depth, important for:

- performance of high-performance coated tool
- stability / adhesion of hard coating

C. Krywka, H. Neubauer, M. Priebe, T. Salditt, J. Keckes, S. V. Roth, M. Müller
J. Appl. Cryst. **45**, 85-92 (2012)

Part-financed by the European Union (European Regional Development Fund)

Texture analysis



Many technical important materials,

such as metals, ceramics and some plastics, are polycrystalline.

Texture assessment describes the orientation distribution of the crystallites.

All properties* influenced by the anisotropy of the constitutive crystals depend on the texture.

**i.e. elastic, plastic, electrical, magnetic, thermal…*

Part-financed by the European Union
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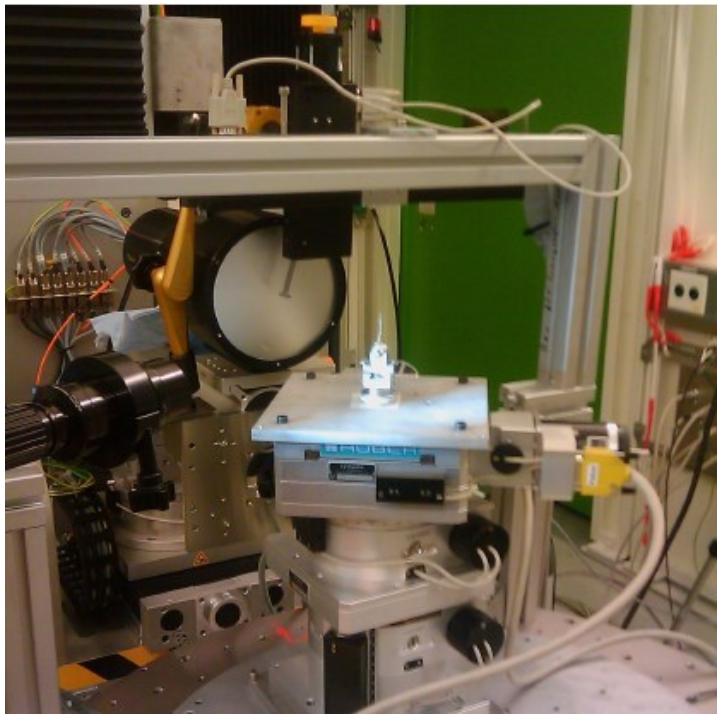
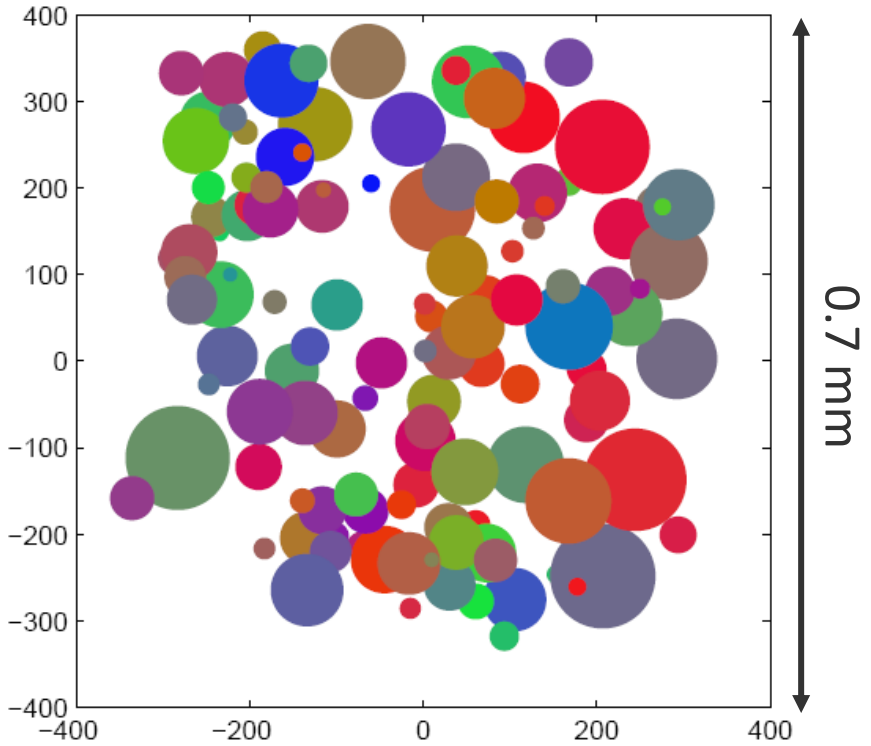
First grain mapping experiment with *in situ* tensile testing





accuracy: grain position 30 µm, orientation 0.15°, grain volume 35 %

Jette Oddershede, Søren Schmidt, Ulrik Lund Olsen, Bettina Camin, Torben Fischer, Norbert Schell (2011)

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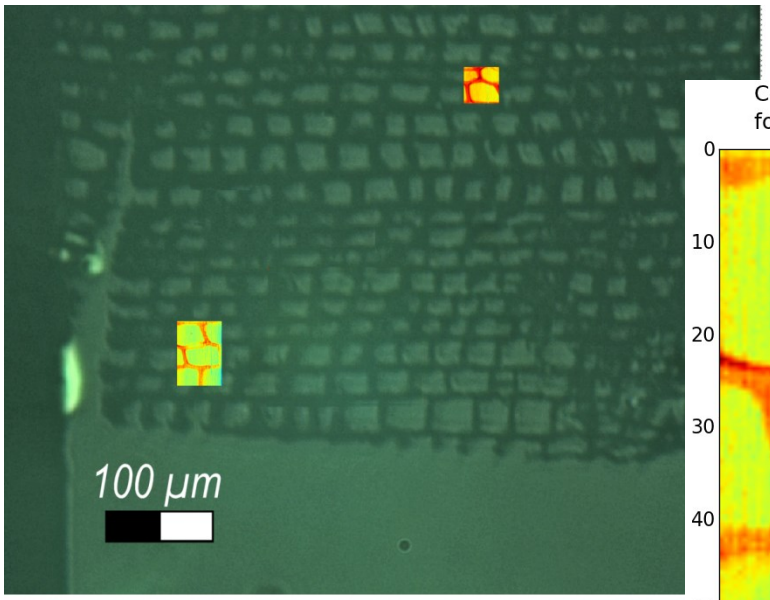
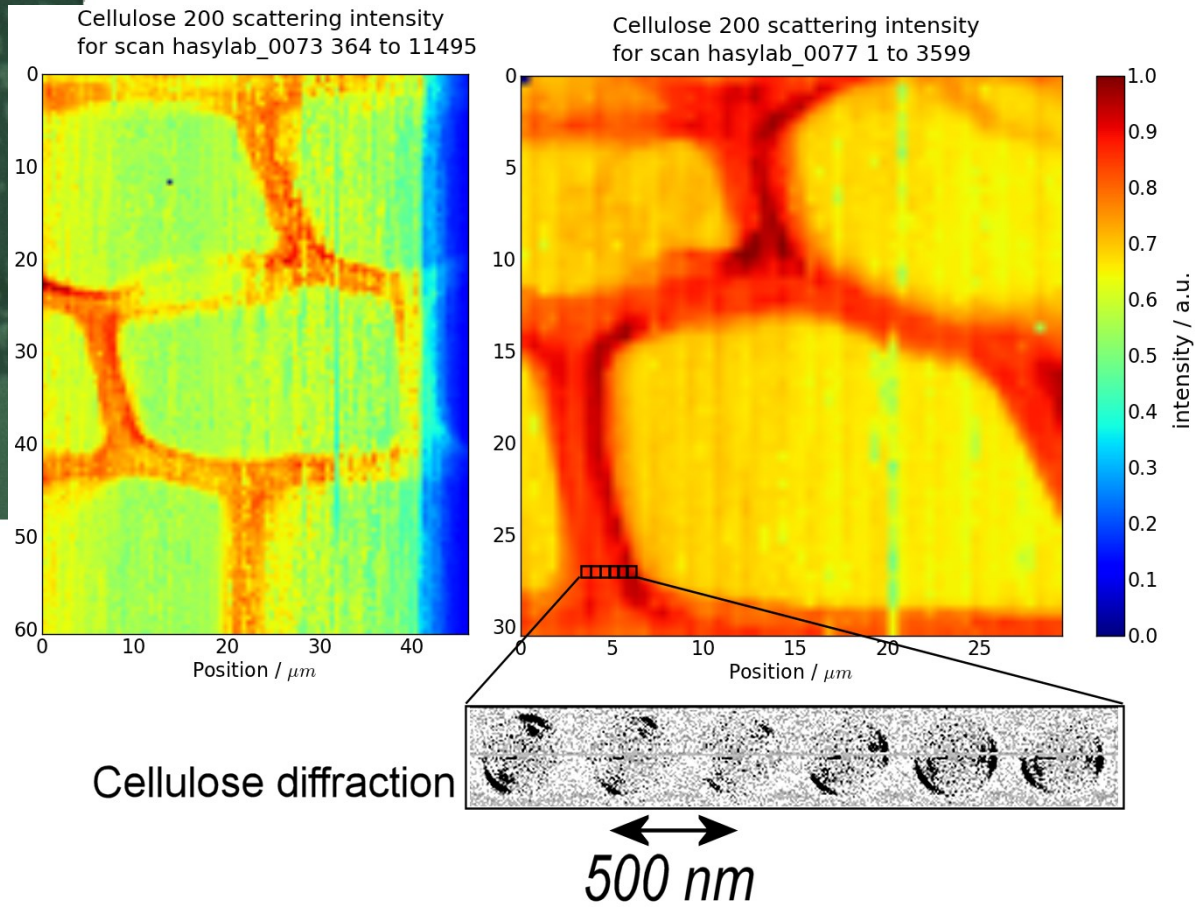
Nanostructure of the wood cell wall





S. Storm, M. Ogurreck, C. Krywka, M. Müller (2012)

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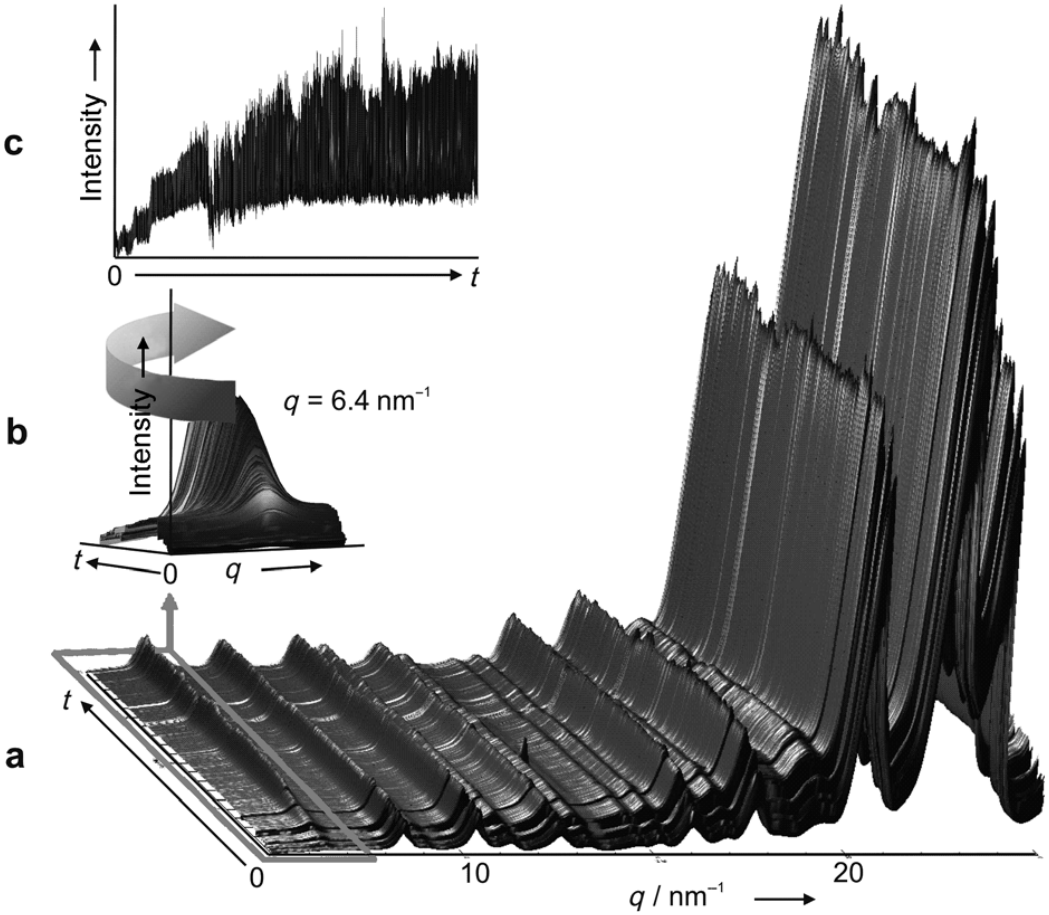
Investigation of the setting of cements by time resolved XRD

Cement represents a complex colloidal suspension consisting of reacting crystalline and amorphous phases.

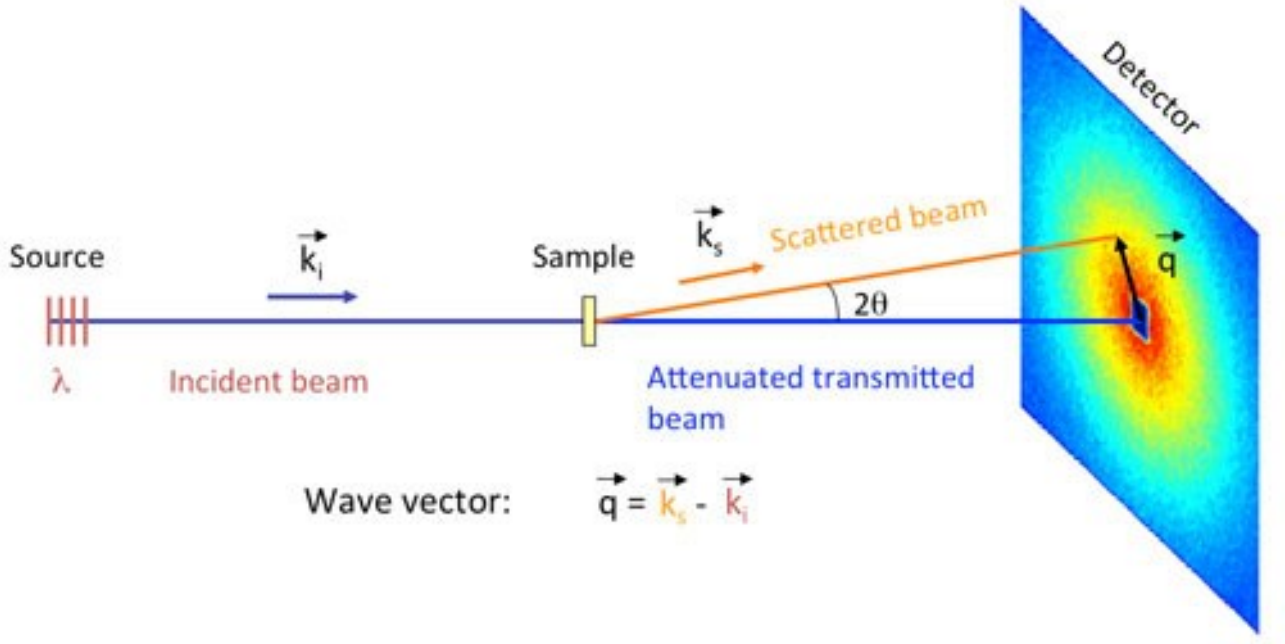
XRD enables the study of cement hydration on the millisecond timescale.



M. Schlegel, A. Sarfraz, U. Müller, U. Panne, F. Emmerling, Angew. Chem. Int. Ed. 2012, 51, 4993 –4996

Part-financed by the European Union (European Regional Development Fund)

Small angle scattering



- Available with neutrons and X-rays
- Useful for structure analysis of hard and soft matter in the nanometer scale

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Investigation of agglomeration of nanoparticles in polymer resins

By Small angle neutron scattering (SANS), the amount and size of aggregations of nanoparticles in solution as well as in polymer matrices can be examined

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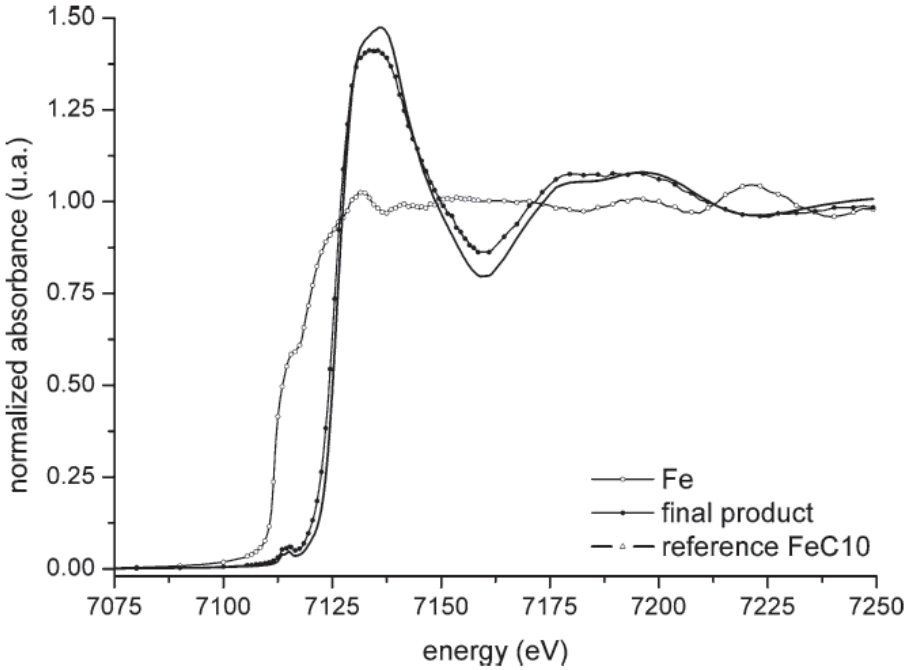
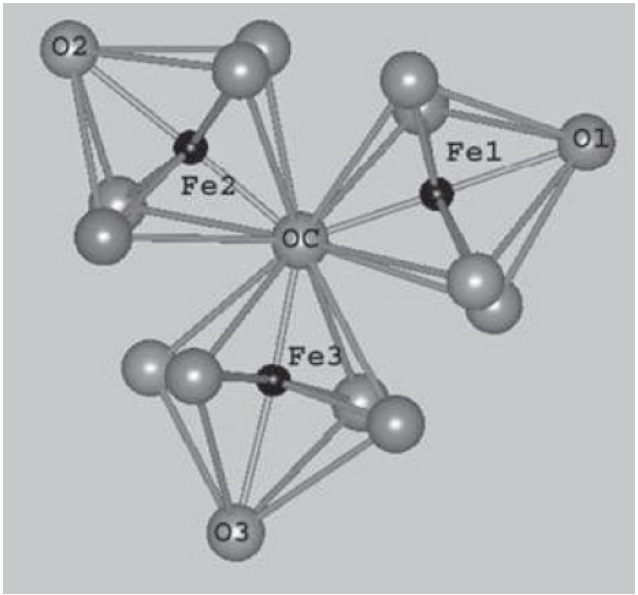
1. W. Reimers (editor), A. R. Pyzalla (editor), A. K. Schreyer (editor), H. Clemens (editor,) Neutrons and Synchrotron Radiation in Engineering Materials Science, Viley VCH, **2008**, p. 245.

Examination of passivation of iron surfaces by XAS





Chemical surface passivation of iron artefacts by carboxylates. Absorption energies match reference substance.

Part-financed by the European Union (European Regional Development Fund)

F. Mirambet, S. Reguer, E. Rocca, S. Hollner, D. Testemale Applied Physics A, 2010, 99 (2), 341-349.

Possibilities of X-ray absorption spectroscopy in engineering and materials science:

- Corrosion studies of various materials
- Corrosion protection
- Other chemical surface treatments

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Summary:

Synchrotron- and neutron sources offer a broad field of solutions for scientific problems in construction and engineering:

- **X-Ray and Neutron diffraction** – texture, phase analysis, residual stress
- **Tomography** – 3D imaging
- **X-ray absorption spectroscopy** – chemical surface treatments, corrosion

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Thank you

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Marc.Thiry@hzg.de

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