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**Real-time non-destructive characterization of
materials at the world's most powerful
microscopes**

01 February 2022
VBIK: Klubbmöte



KTH HOLDING AB



Co-founder & CEO

Ahmet Bahadır Yıldız

PhD from KTH Royal Institute of Technology within SwedNess: Swedish graduate school on neutron scattering. Metallurgical Engineer from RWTH Aachen. Working on materials and process optimization using experiments at large-scale facilities, since 2015.

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Co-founder & CSO

Peter Hedström

Professor in Materials Science at KTH Royal Institute of Technology. Conducting research in metallic materials at large-scale facilities, since 2002. Experience from conducting experiments at 10 different neutron and synchrotron X-ray facilities.

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Data scientist

Emil Österberg

Bachelor's degree in Vehicle Engineering from KTH. Pursuits Master's degree in Applied and Computational Mathematics at KTH in Mathematics of Data track. Previous working experience in numerical methods.

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>25
years

62

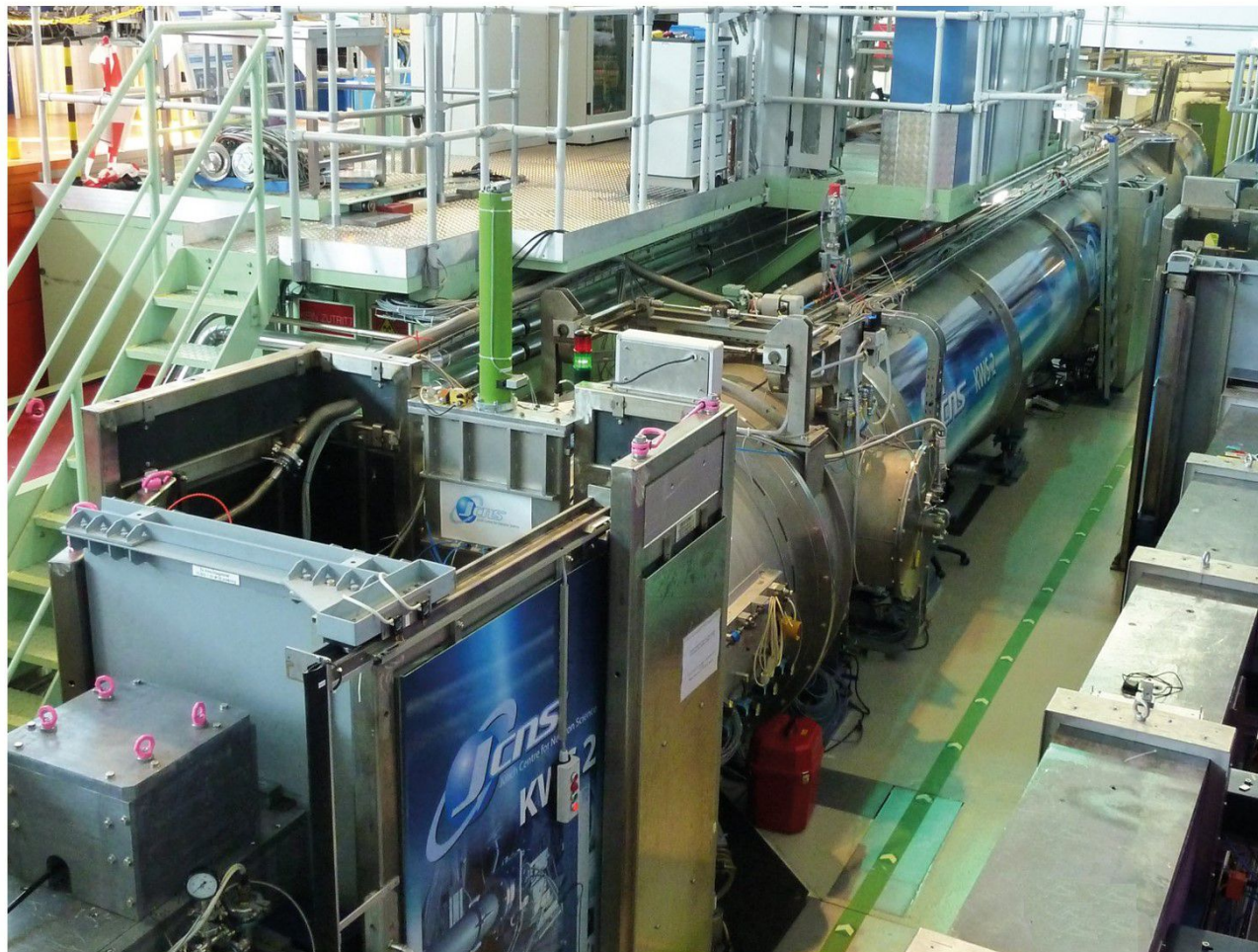
facilities

€450M

Around the globe

Annual operation
cost alone in
Sweden

Inside the facilities



/Introduction: Scatterin AB

- Why we need better materials and processes?
- Why we need characterization tools?
- What is missing in lab?
- Why we need Big Science?

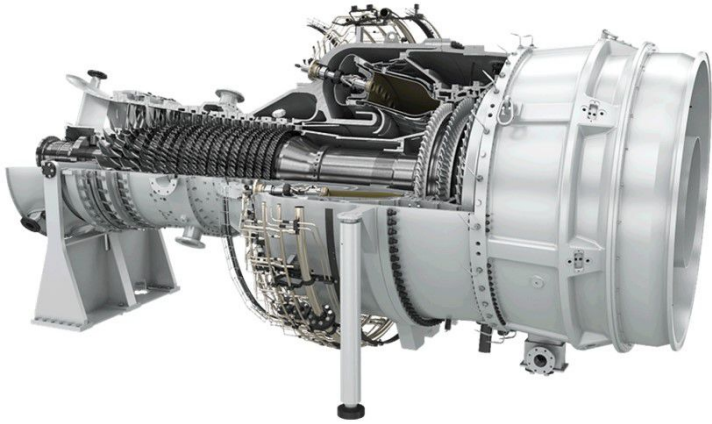
/Neutron and X-ray scattering techniques

- When neutrons and when X-rays?
- Application examples
- How to access to the facilities?

/Other side of the coin

- What we do at Scatterin AB?

Why we need innovations in materials and processes?



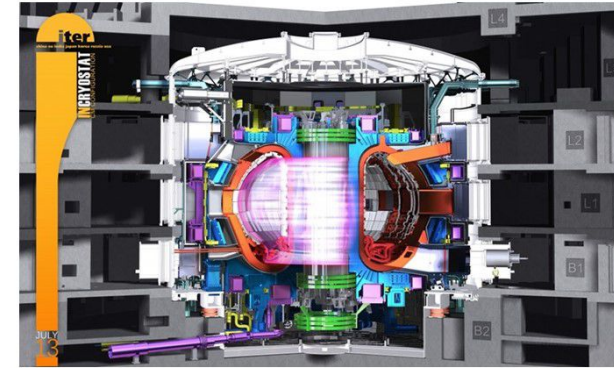
<https://www.siemens-energy.com/global/en/offerings/power-generation/gas-turbines/sgt-800.html>



<https://www.kugellager-express.de/deep-groove-ball-bearing-inch-r10-w7-14-open-oiled-15-875x34-925x7-14-mm>



https://www.youtube.com/watch?v=JN_Mrq_nxE&ab_channel=SecoTools

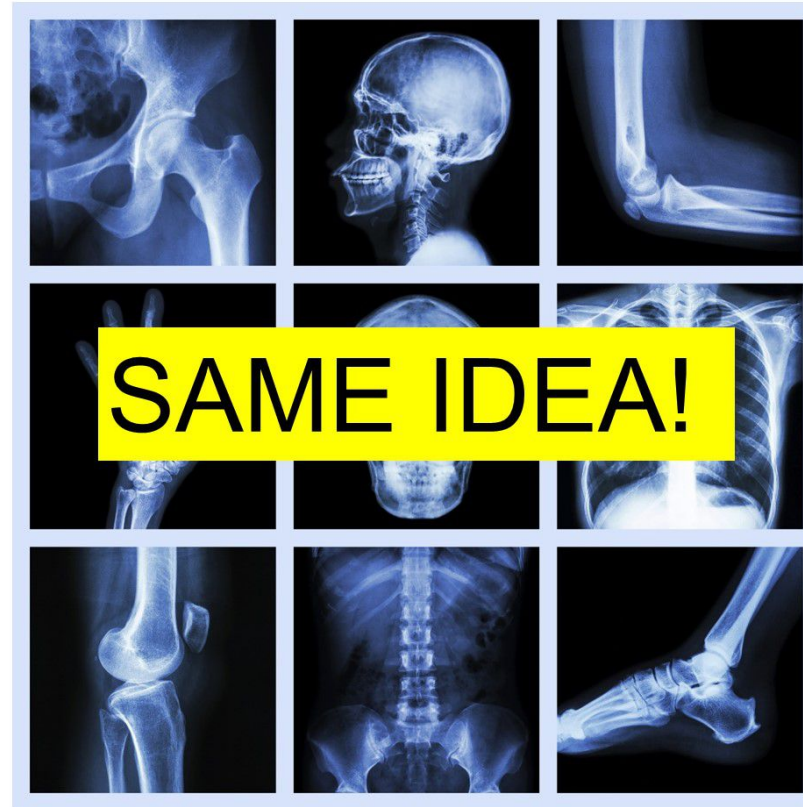


Fusion reactor

https://www.iter.org/img/resize-900-90/www/content/com/Lists/WebText_2014/Attachments/230/tkm_cplx_final_plasma2013-07.jpg

- Applicational requirements: temperature/deformation/voltage/fuel/conductivity/corrosion...
- Cost
- Supply chain problems
- Environmental footprint/requirements: Emissions, recyclability..

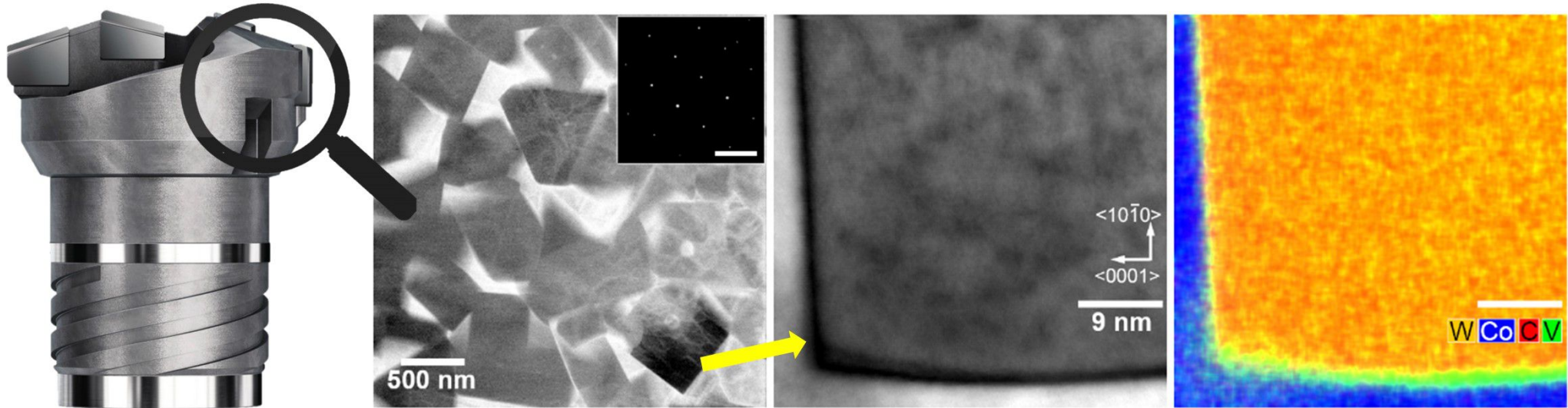
Why we need characterization tools?



<https://cvccessentials.files.wordpress.com/2015/03/491912865.jpg>

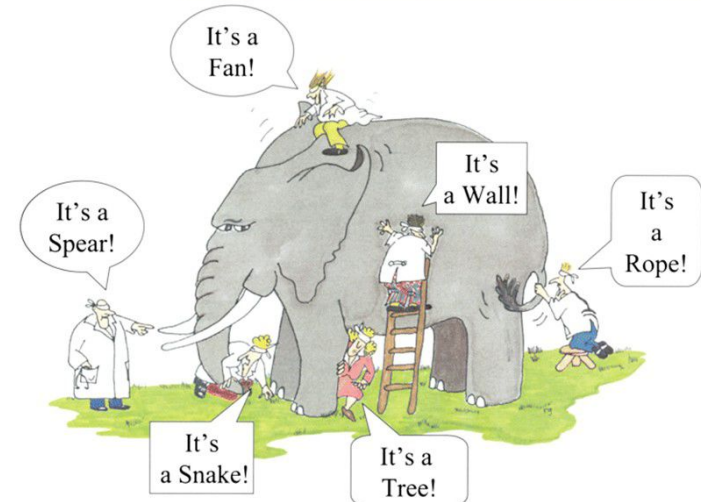
- Knowledge-based materials and process development

What is missing in lab?

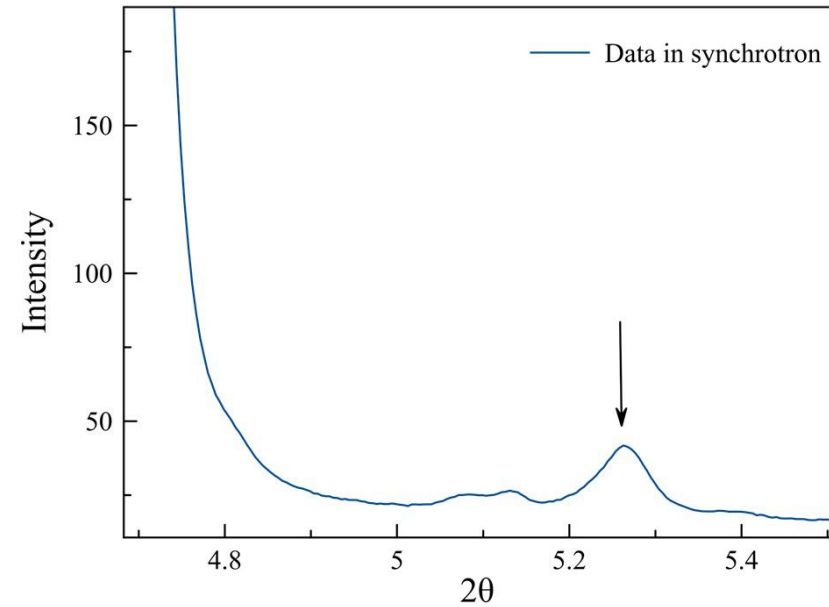
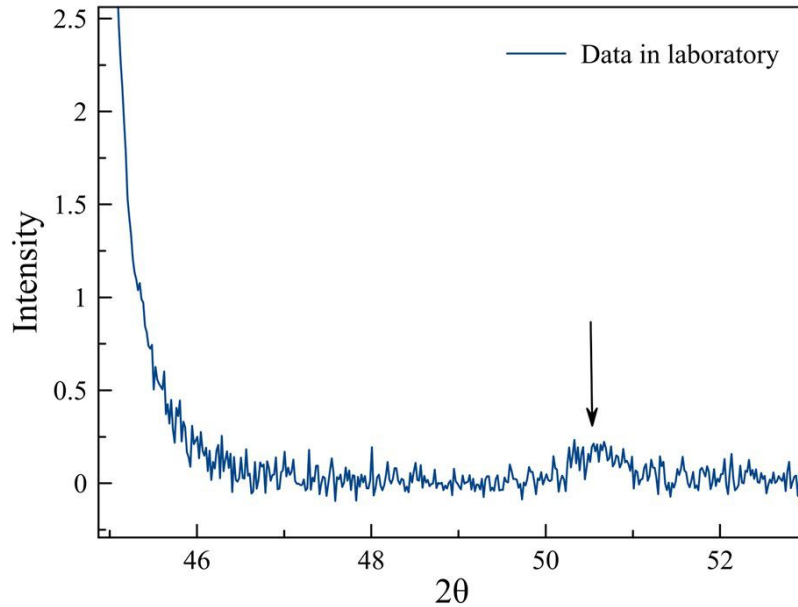


http://sandvik.ecbook.se/se/en/solid_round_tools/

- Destructive: Requires sample preparation
- Complete quantitative view: very small probe volume
- Post-mortem: we check after process/service finished
- Slow



Why we need Big Science?



- Better resolution and sensitivity: time and spatial (nanometer scale resolution)
- Higher penetration: 10 μm vs 7 cm in steel
- Speed: 1.5 h vs 5 ms
- Non-destructive
- In-situ experiments: See what happens to material/component during processing & service



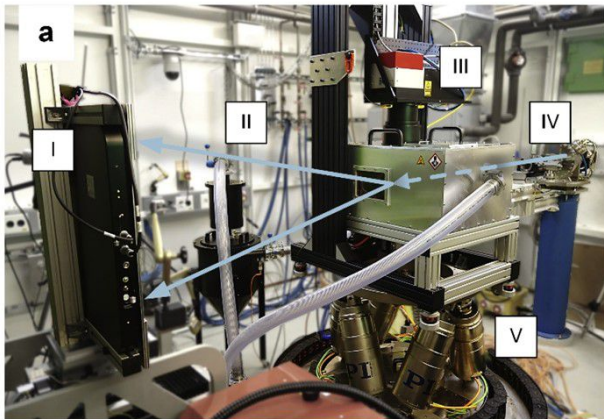
Anders Kaestner: https://www.youtube.com/watch?v=VESMU7JfVHU&ab_channel=AndersKaestner

In-situ experiments

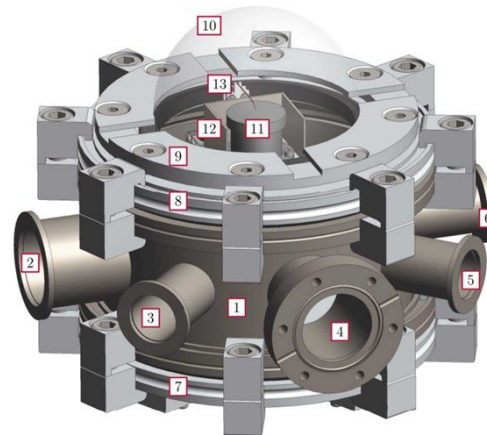
- Temperature range: 10 K to 1700 °C
- Heating and cooling rates: 4000 K/s heating and 2500 K/s cooling
- Load: up to 100 kN
- Gas options: Air, inert, vacuum, reactive
- Specific devices: Need-driven, tailored devices are possible



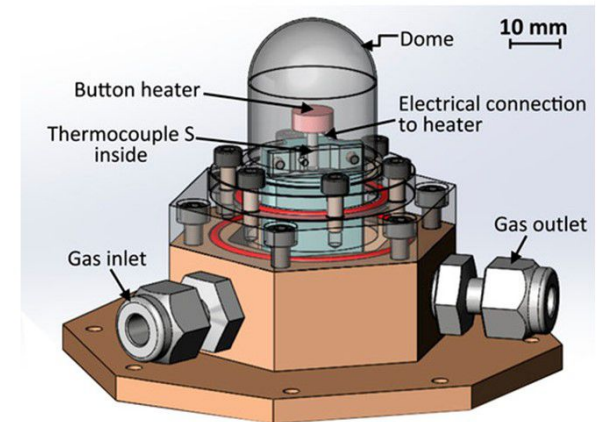
High temperature deformation setup
<https://j-parc.jp/researcher/MatLife/en/se/image/bl19ht01.jpg>



In-situ setup for metal additive manufacturing.
F. Schmeiser, et al. Additive Manufacturing 32 (2020) 1010282



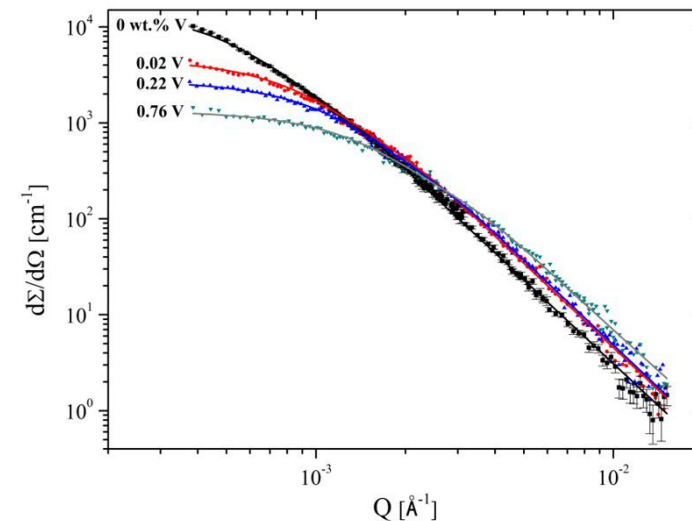
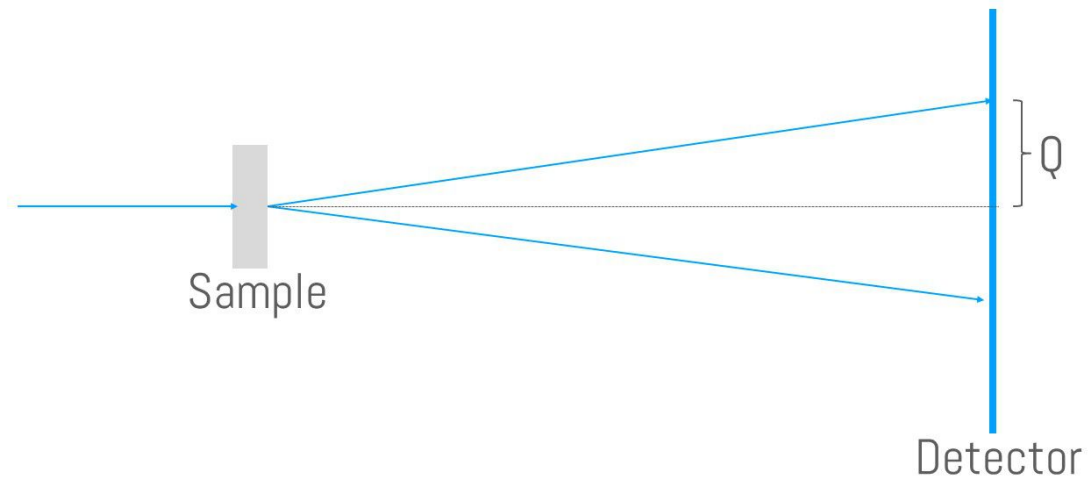
In-situ setup for electric field and temperature
M Nentwich, J. Synchrotron Rad. (2021). 28, 158–168



Reactor for in-situ experiments
M.-I. Richard Review of Scientific Instruments 88, 093902 (2017)

Non-destructive analysis & Large components





Detector detects => Scattering vector (Q) & Count

$$\text{Size of a structure} \approx \frac{2\pi}{Q}$$

$$Q = \frac{4\pi}{\lambda} \sin(\theta)$$

- Small structure => High Q range
 - Interplanar spacing => Diffraction!(Wide angle scattering)
- Small & ultra small angle scattering => Dimensions <1 nm to several μm

Neutron and synchrotron X-ray techniques

Diffraction => Crystallography

- Crystal structure & Phase identification and fraction
- Reaction sequences and kinetics (incubation periods, precipitation sequence)
- Residual stresses & Strain/stress partitioning between the phases: temperature-dependent elastic constants
- Defect evolution e.g. dislocation density
- Detection range: >0.x vol%

Small angle scattering (SAXS and SANS) => Compositional (scattering) variations

- Quantification of precipitates, porosity and particles (low volume fractions can be detected)
- Size distribution and volume fraction
- Size range: nm to μm

Imaging and tomography (CT) => Compositional (absorption) variations

- Reconstruction of sample volume for analyzing cracks, porosity, inclusions
- Size range: Sub-micron to cm

* Compared to lab: faster, higher resolution, higher sensitivity (better signal to noise ratio), and bulk averaged data

* They can also be done **simultaneously in conditions mimicking production and service** (temperature, time, load can be simulated).

Neutrons vs X-rays

They see and interact with matter differently.

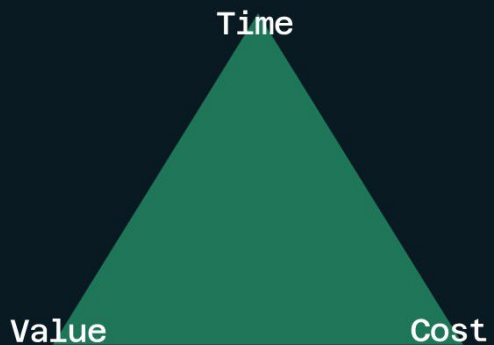
- Scattering contrast
 - Neutrons have magnetic scattering (ferrite)
- Penetration dept
- Imaging: resolution
 - Synchrotron X-rays: $< \mu\text{m}$
 - Neutrons: a few tens of μm

Synchrotron X-rays

- Easier to get
- Faster acquisition
- Smaller beam size (nm-range resolution in mapping)
- Problems in gauge volume



https://www.psi.ch/niag/CulturalHeritageEN/igp_f345611c157211931a0e673009a60c6a_XBudplusBud.jpg



Road to knowledge & innovations

Key question: What are the needs and requirements?

Example: High-voltage components

Problem/question

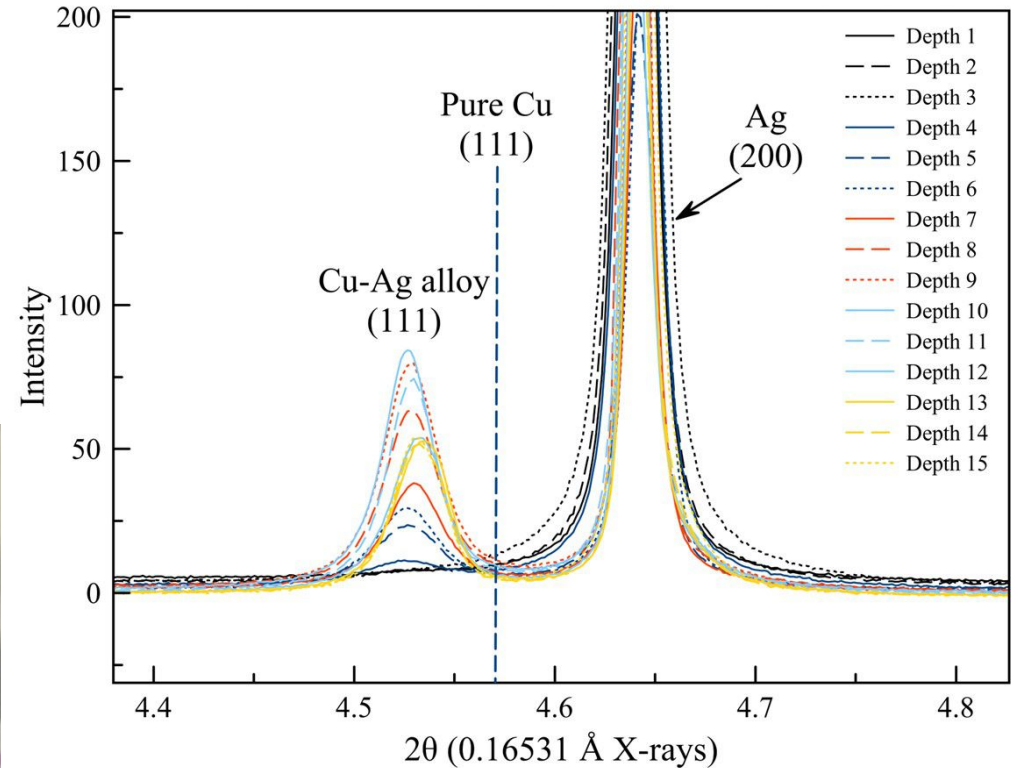
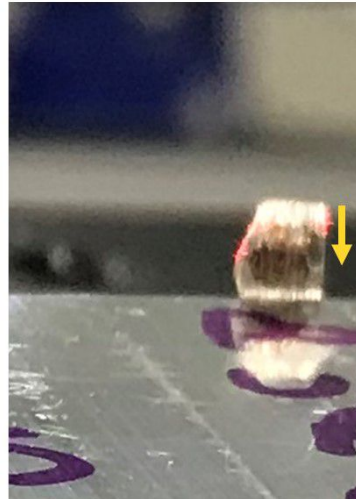
- Formation of unknown abrasive phase in high-voltage contact joints

Solution

- High-energy synchrotron X-ray diffraction

Benefit

- Phase is identified
 - Formation mechanism is revealed
 - Oxide formation is found in mapping
-
- Measurement to final report in 1 week



Example: High-voltage components

Problem/question

- Formation of unknown abrasive phase in high-voltage contact joints

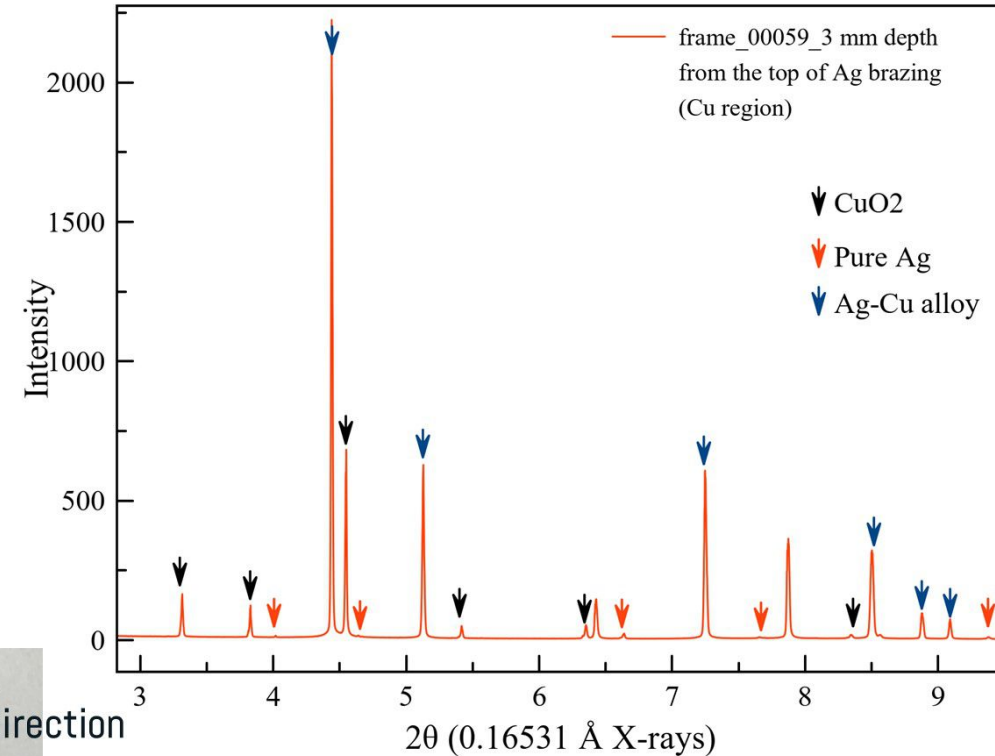
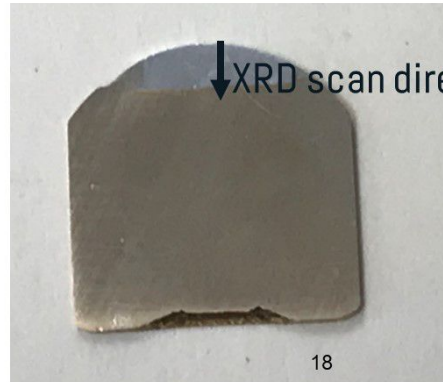
Solution

- High-energy synchrotron X-ray diffraction

Benefit

- Phase is identified
- Formation mechanism is revealed
- Oxide formation is found in mapping

- Measurement to final report in 1 week



Example: Welding in large component

Problem/question

- How does strain/stress develop with welding?
- Do we have harmful/brittle phases?

Solution

- High-energy high-resolution synchrotron X-ray diffraction
- Non-destructive measurement

Benefit

- Revealed formation of a brittle phase inside the weld
- Mapped strain/stress gradient in 2D
- Identified optimum welding parameters
- Input for simulations

Example: Metal cutting tools

Problem/question

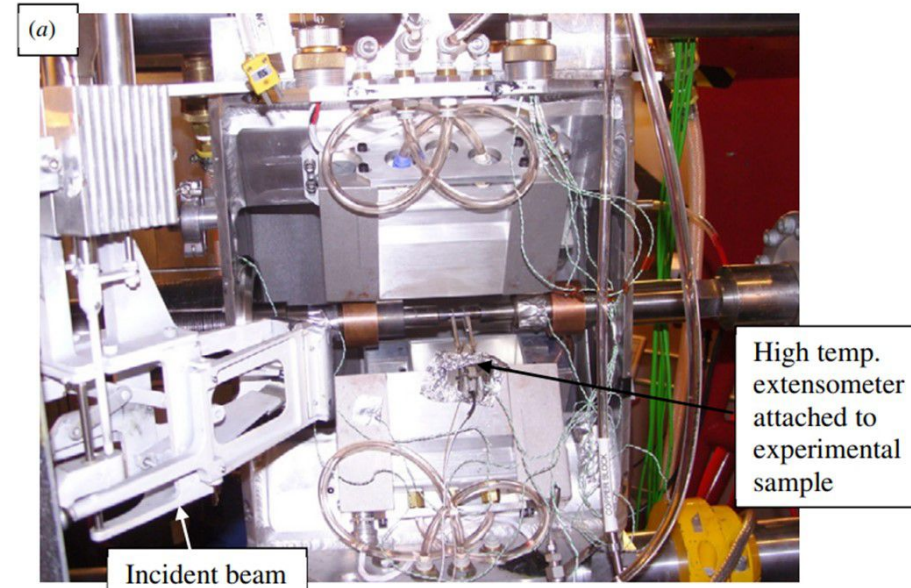
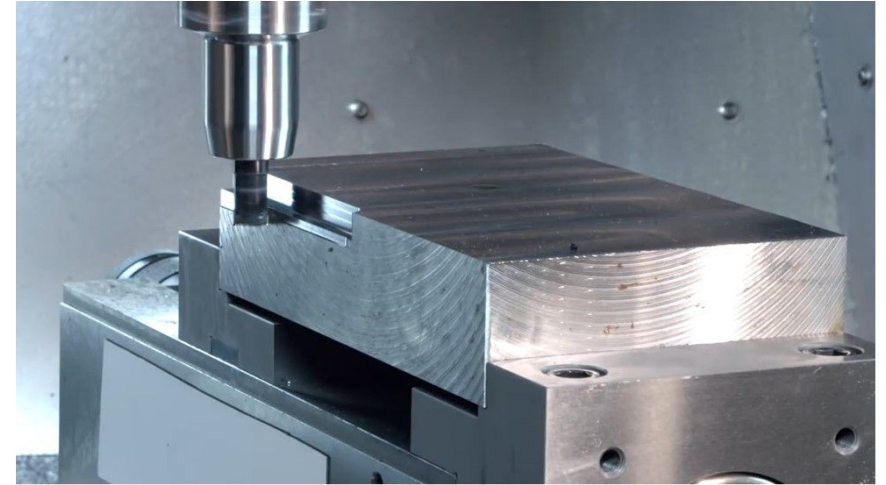
- Why some hard metal grades have longer tool life?
- What happens to tool at >700 °C under ~ 1 GPa load?

Solution

- In-situ diffraction at operation temperatures under compressive load
- In-situ SANS at operation temperatures

Benefit

- Simulated service conditions: every details of the material/component
- See what happens during service without interruption
- Create foundation for future developments
- Input for simulations



Example: Bearing component

Problem/question

- Structure evolution under the surface of bearing component
- How is the gradient? How do defects evolve?

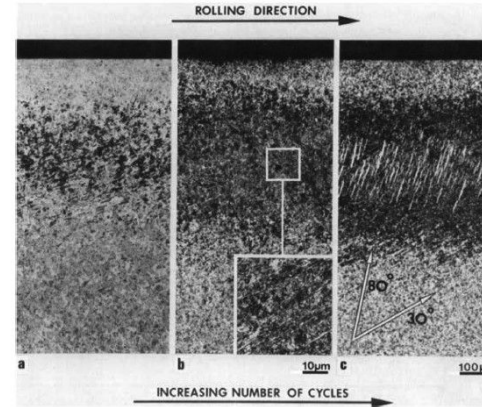
Solution

- High-energy high-resolution synchrotron X-ray diffraction

Benefit

- Phase evolution is identified & quantified
- Defect evolution is quantified
- Answer: what brings better performance?
- Input to simulations

- Measurement to results in 2 days



Swahn H, Becker PC, Vingsbo O. Martensite decay during rolling contact fatigue in ball bearings. Metal Trans A. 1976 Aug;7(8):1099-1110.



<https://www.kugellager-Express.de/deep-groove-ball-bearing-inch-r10-w7-14-open-oiled-15-875x34-925x7-14-mm>

Example: Heat treatment

Problem/question

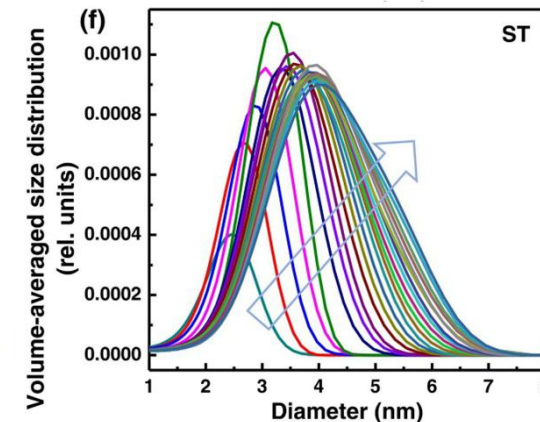
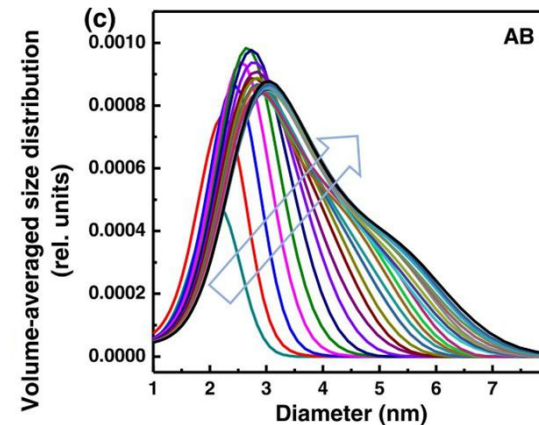
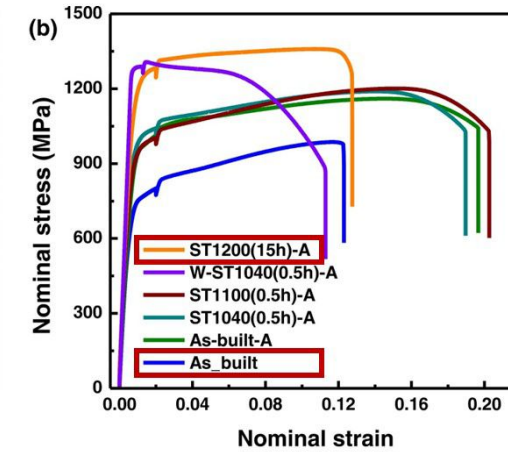
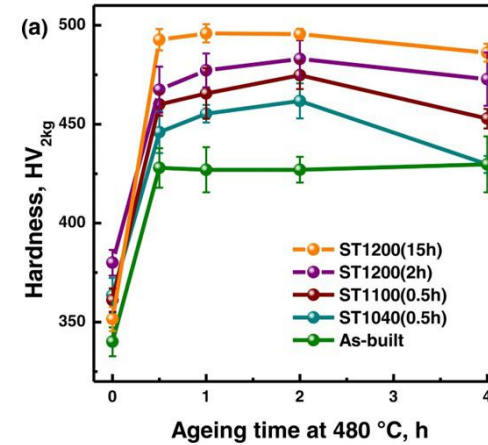
- Reason behind different mechanical performance after heat treatment?
- How to tailor the structure for the best performance?

Solution

- Synchrotron X-ray diffraction
- In-situ SANS at heat treatment temperatures (480 °C)

Benefit

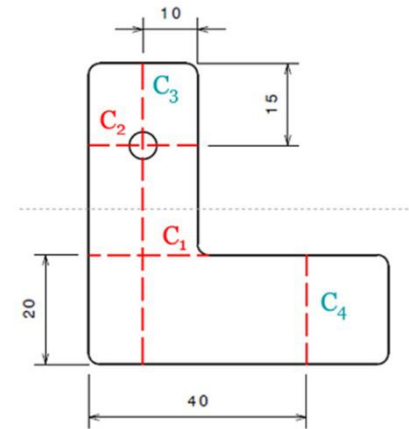
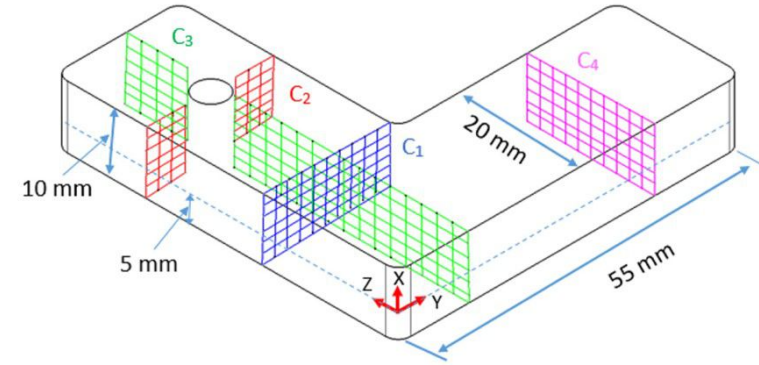
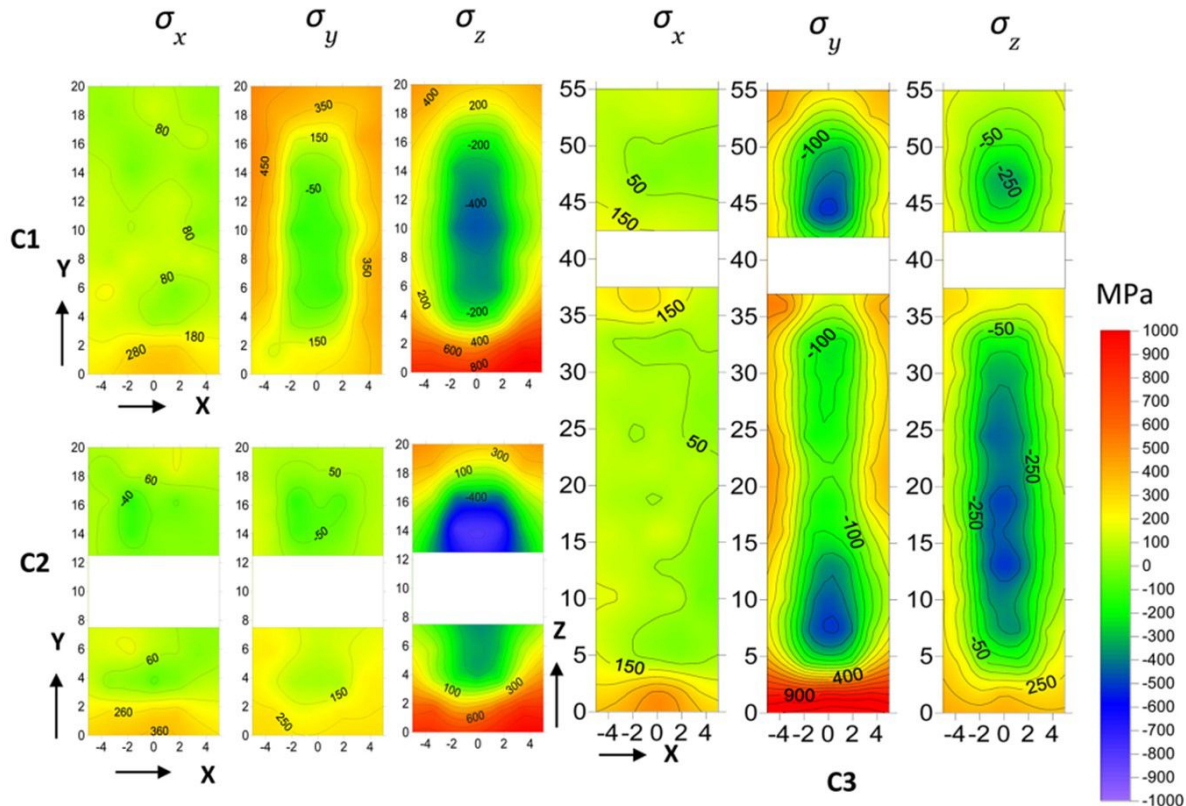
- Subtle changes in the structure identified
- Optimized performance: Excellent combination of strength and ductility (ultimate tensile strength ~1340 MPa and uniform elongation ~10.5 %)
- Optimized heat treatment parameters
- Input for mechanical modelling



Example: Mapping residual stress distribution

Residual stress distribution in additively manufactured component

- Strain/stress for three orthogonal sample directions
- $2 \times 2 \times 2 \text{ mm}^3$ gauge volume
- Non-destructive measurement



Example: Real time analysis of soldering

Structural evolution during soldering of Cu-6Ni/Sn-0.7Cu/Cu-6Ni joint

Synchrotron X-ray imaging up to 240 °C

Formation and growth of:

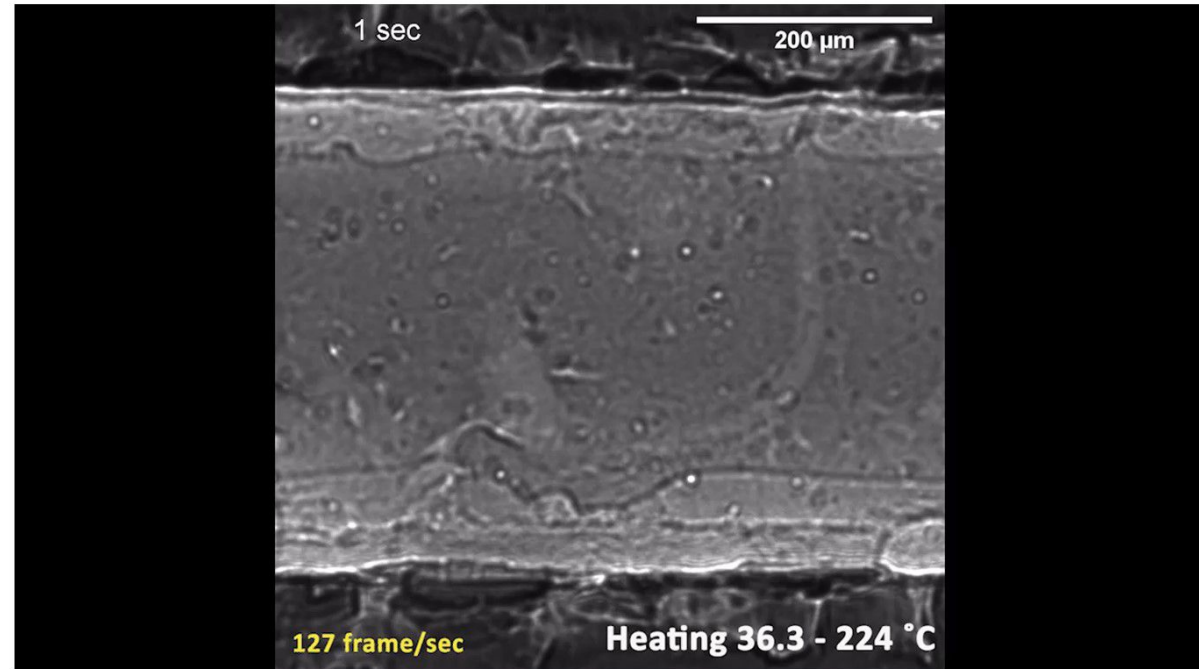
- Intermetallic compound (IMC)
- Voids
- Cracking

The cross-section can be mapped by XRD:

- Phase identification & quantification
- Stress/strain quantification

Benefits

- Understand each detail of soldering
- Optimize soldering parameters



How to access to the facilities?

Academic access

- Free
- We need to publish the results (results will be open to public)
- Competition: Write proposal and wait for the results
- Applications 2 times a year
- Application to experiment about a year

Industrial access

- Paid
- Fully confidential
- Fast: application is possible anytime

Special programs

Other side of the coin



Terabytes of data



Months of analysis



Project cost

Own data analysis software: In-house developed pipelines

Powerful analysis software for X-ray and neutron scattering data

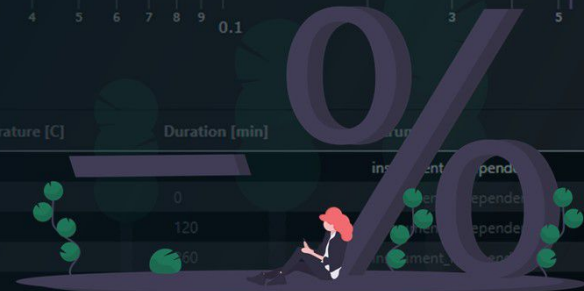


10X faster access to results



No competence bottleneck

| Data title | Composition | Temperature [C] | Duration [min] | Instrument | Date added | Experiment date |
|--------------------------|-------------|-----------------|----------------|------------------------|------------|-----------------|
| 1_1859199-256999_031 | Steelaging | 330 | 0 | instrument_independent | 11/29/2021 | |
| 1_3011102-256999_001.dar | Steelaging | 330 | 0 | instrument_independent | 11/29/2021 | |
| 1_3011102-256999_012.dar | Steelaging | 330 | 120 | instrument_independent | 11/29/2021 | |
| 1_3011102-256999_003.dar | Steelaging | 330 | 60 | instrument_independent | 11/29/2021 | |
| 1_3011102-256999_012.dar | Steelaging | 330 | 60 | instrument_independent | 11/29/2021 | |
| 1_3011102-256999_003.dar | Steelaging | 330 | 20 | instrument_independent | 11/29/2021 | |



Reduced cost

Synchrotron X-rays and neutrons as a service.

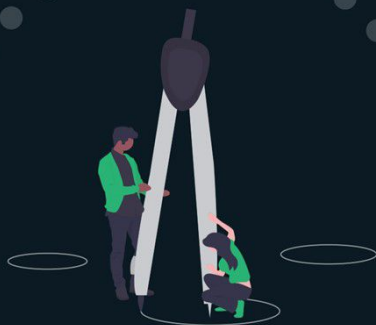
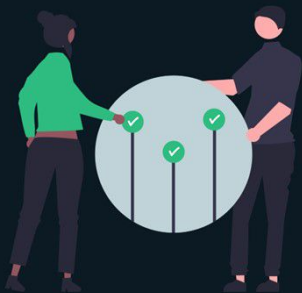


We offer beginning-to-end service for high-quality and cost-effective materials analysis.

01

Planning

We meet, understand your problem, and choose the most suitable solution: neutrons, X-rays, or maybe we can solve the problem at the lab. Experimental plan, lead time, and deliverables are defined.



02

Preparation

We perform the necessary preparations, including samples, sample environment, and sample container.

03

Measurement

We perform the experiments: diffraction, small-angle scattering, or tomography.



04

Analysis

We analyze the data, work together with you for the interpretation of the results, and finalize the project as it is planned.

Conclusions

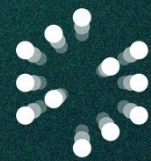
- Sustainable development requires novel materials and processes
- Knowledge-based innovations

Big Science: Neutron and synchrotron X-ray facilities

- Better resolution and sensitivity
- Higher penetration (7 cm in steel) and speed
- Non-destructive
- In-situ experiments (See what happens to material/component during processing & service)

Scatterin AB

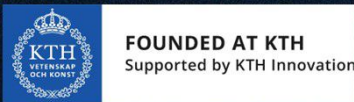
- Focuses on **industrial needs**.
- Key question: What are the needs and requirements?
- **Industrially viable** Big Science: Cost, time scale, impact



SCATTERIN

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