Institut Laue-Langevin

Neutrons for society

Neutron based measurement techniques: unique tools in the non-destructive toolbox

30/11/2021

CNES: journée «CND - Détection d'endommagement et suivi en fabrication »

Contact us: industry@ill.eu
www.ill.eu/industry

caroline.boudou@ill.eu Industry Contact Officer

Institut Laue-Langevin (ILL)

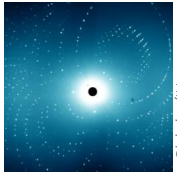
- 1 site (Grenoble)
- Managed by FR, DE, GB
- 11 member states
- Budget 2020: 102 MEUR
- 543 employees
- Since 1967

Source of neutrons to investigate materials, component and devices under varying conditions.

Magnetism, Biology and Health, Material and engineering, Chemistry, Crystallography, Soft matter
Particle and nuclear Physics
+ production of radio-isotopes

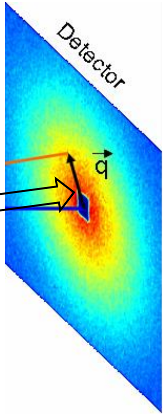
Neutron analytical techniques:
probing the intimate properties of materials

DIFFRACTION
Structure = arrangement of atoms and / or magnetic domains



Blakeley/ILL

Small angle Scattering
Statistics on the microstructure



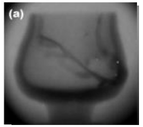
Grillo/ILL

source of neutrons or synchrotron X-rays

Beams of neutrons

Stanojev-Pereira

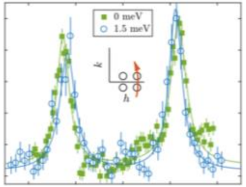
SAMPLE



Stanojev-Pereira

IMAGING
Morphological and Structural 3D info

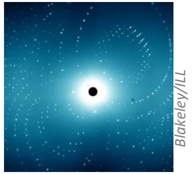
SPECTROSCOPY
Dynamics = movement of atoms, Electronic structure



Instruments = **beam lines**
Measurement periods **= beam time**

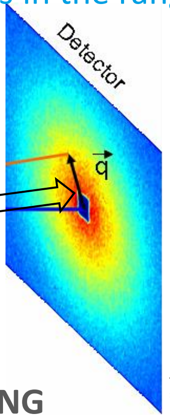
Neutron analytical techniques: contributing to material science and engineering challenges

DIFFRACTION
Phases analysis, strain/stress determination



Blakeley/ILL

Small angle Scattering
Statistical information on pores, inclusions, precipitates in the range of 1-600nm



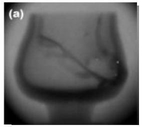
Grillo/ILL

source of neutrons or synchrotron X-rays

Beams of neutrons

Stanojev-Pereira

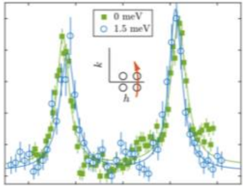
SAMPLE



Stanojev-Pereira

IMAGING
Defect detection, shape integrity, in-situ monitoring...

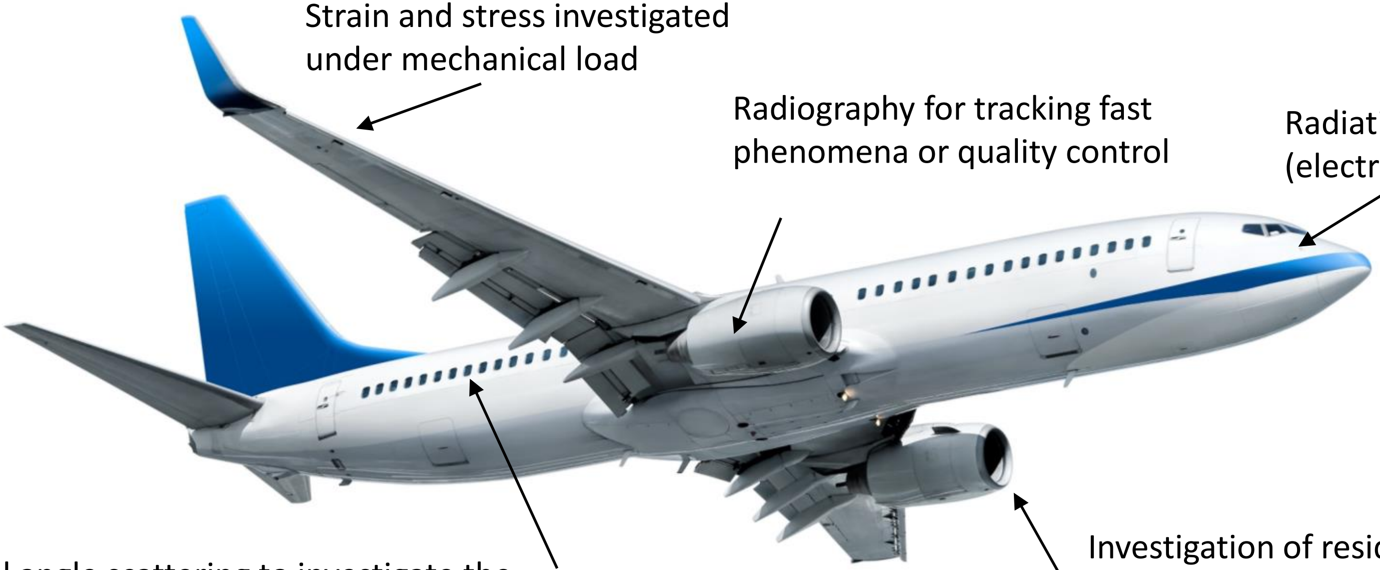
SPECTROSCOPY
Hydrogenated reactions, magnetism

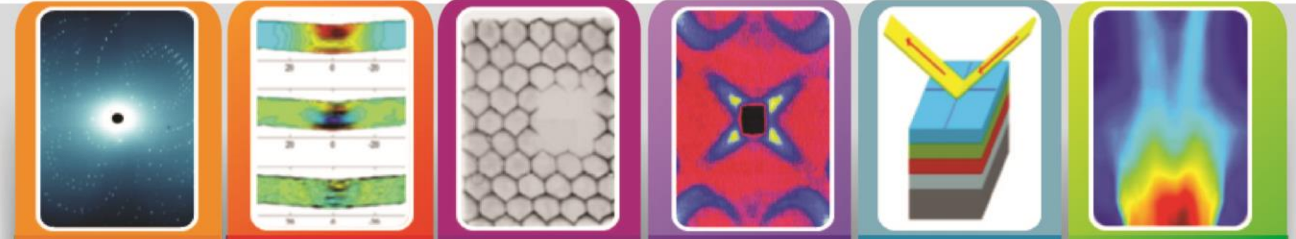


Instruments = **beam lines**
Measurement periods = **beam time**

Neutron based techniques: advanced tools in the NDT toolbox

⇒Develop, improve and benchmark on-line/on-site NDI techniques
⇒New materials, fabrication routes or post-treatment processes





	DIFFRACTION	STRESS SCANNING	IMAGING	SANS	REFLECTOMETRY	SPECTROSCOPY
		NON DESTRUCTIVE	**RADIOGRAPHY TOMOGRAPHY**	**SMALL ANGLE NEUTRON SCATTERING**		
	Position of atoms, crystal structure, magnetic phases, textures	Manufacturing processes, 3D printing, machining, welding, hardening, surface treatments, ageing	Visualisation of fluid, adhesive, polymer within metallic objects. Possible combination with x-rays	Structure of fluids, aggregates, vectorisation, nanostructures in solids, ageing of metals	Surface, interface, composition, thickness, roughness	Movements of atoms, dynamic analysis of molecules
Range of Investigation features	Atomic scale	From 60µm below surface up to several cm in metals	~ 10 µm to 1 mm	~1 nm to 1 µm	~ 1 nm to 600 nm in layers	Atomic scale
Typical size of sample	Few mm^3	From tens of mm^3 to 850kg	From cm to ~1m	Few mm^3	Few mm^3	Few mm^3
Industrial Sectors						

NEUTRONS FOR SOCIETY

Neutron diffraction

- Stress determination

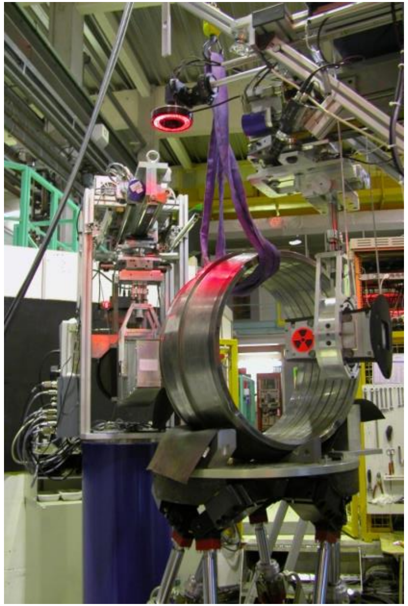
  profiles and maps obtained after sevreral cm of metal

- (powder and single crystal) diffraction

  organisation of atoms and in-situ evolution

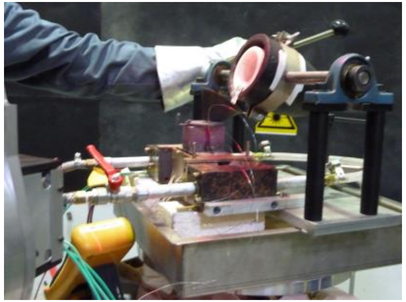
Stress Analyser at the ILL: SALSA

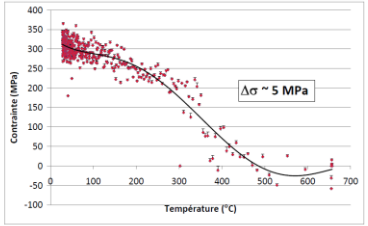
BIG
large components



1m / 750kg

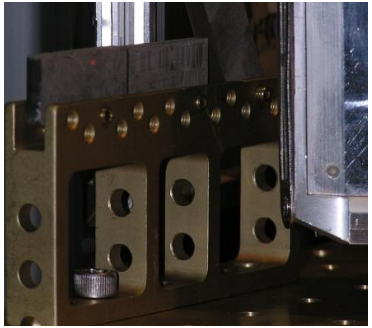
FAST
In-situ studies

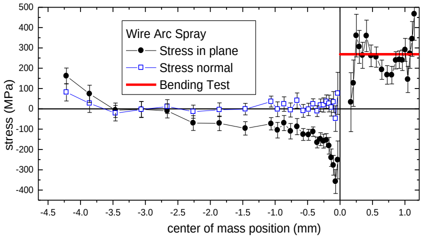




1s

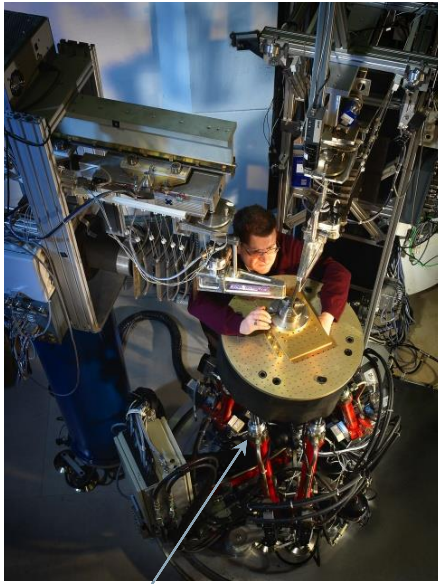
HIGH RESOLUTION
Surfaces & Interfaces





40 microns

Stress Analyser at the ILL: SALSA



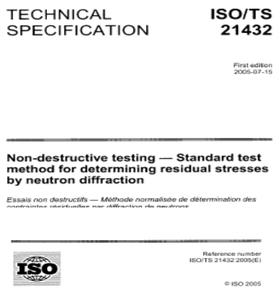
Sample positioning stage (hexapod): can handle 750 kg samples with positioning accuracy of about 5 µm.

Non-destructive stress scanning for the determination of stress in real-size components

=> Residual stress mapping allows deep investigations on: **manufacturing processes, 3D printing, machining, welded areas, hardening processes, ageing, surface treatments**.

- Features:
  - Gauge volume : ~ mm^3
  - Scan can start from about 60 µm below the surface up t several cm in the piece examined
  - Investigation possible after about:
    - 6 cm of steel,
    - 30 cm of Al,
    - 7 cm of Ti
    - 4 cm of Ni
    - 6 cm of Cu
  - In situ studies are possible (furnace, cryostat, test rig)
  - Typical uncertainty: 50 MPa

EASI-STRESS


TECHNICAL SPECIFICATION

ISO/TS 21432

First edition 2005-07-15

Non-destructive testing — Standard test method for determining residual stresses by neutron diffraction

Essais non destructifs — Méthode normalisée de détermination des

ISO

Reference number ISO/TS 21432:2005(E)

© ISO 2005

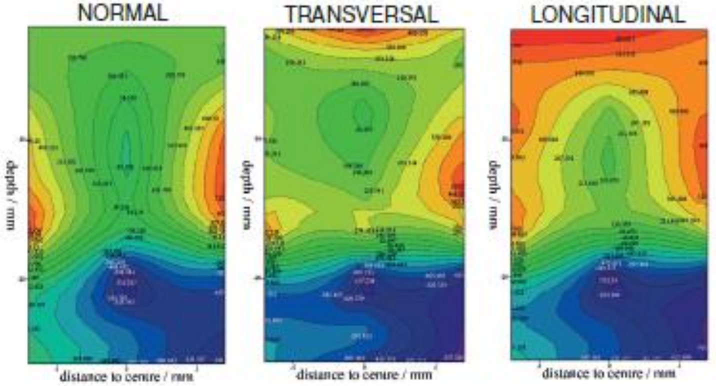
2012 CEN ISO/TS 21432 has been accepted as a standard by AFNOR

Neutron diffraction: stress determination

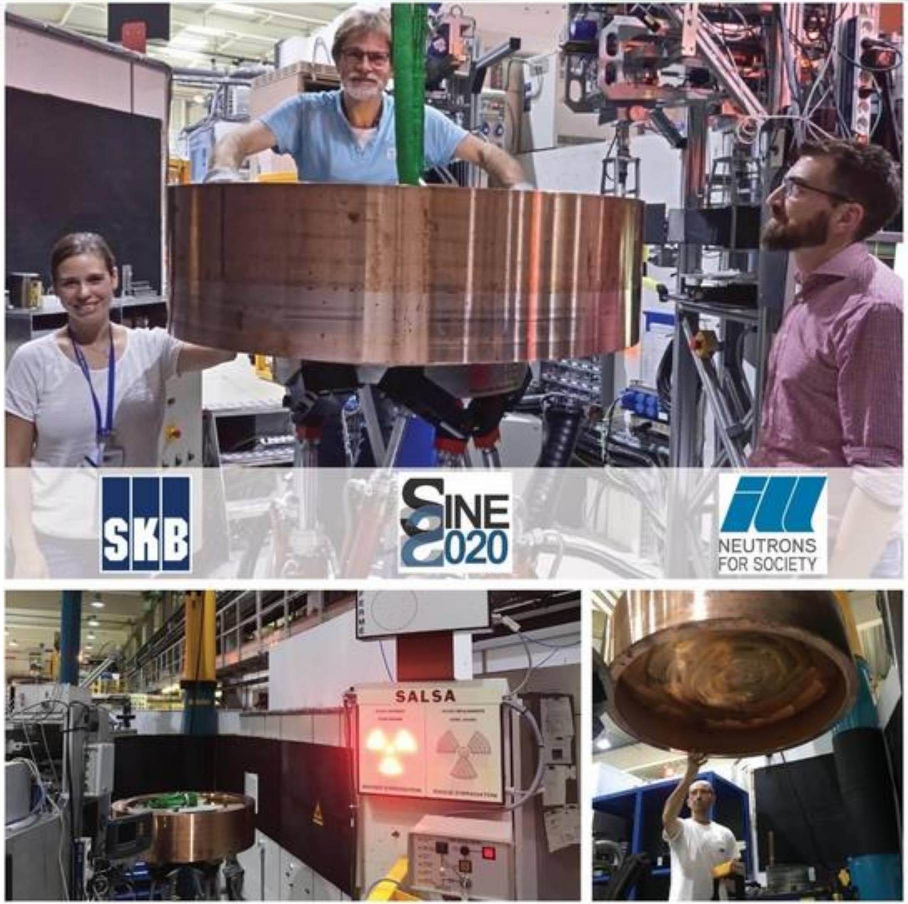
Investigation of residual stress in the thickness (4 cm) of the welded region (FSW)

- SKB, Sweden
- a 850 kg piece made of copper

Stress maps for the three principal components of the stress tensor



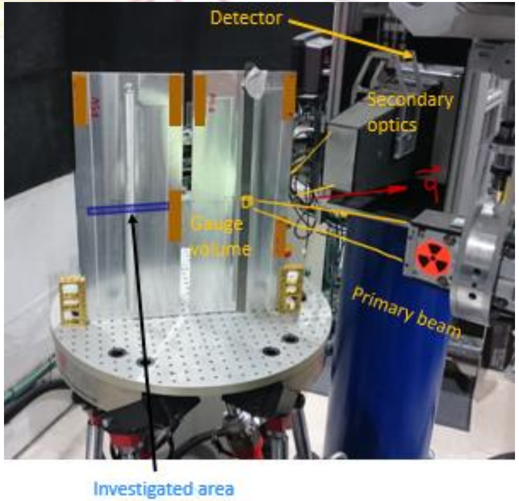
(values in MPa, maps not built from the SKB measurements)

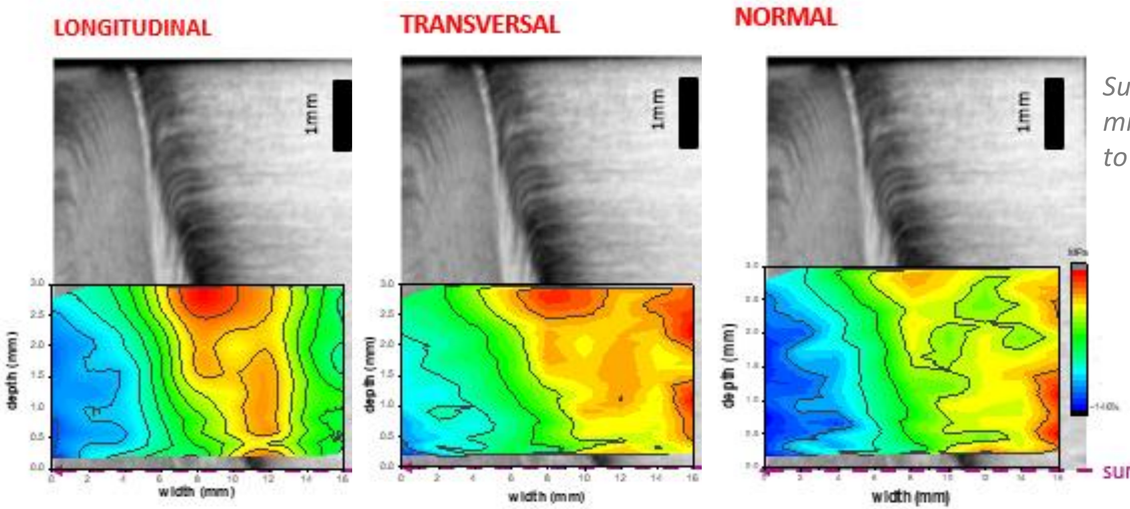


SINE2020 project receives funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 654000.

Stress determination on FSW at SALSA

- Near surface and through thickness **stress mapping**
- **non-destructive**
- full stress tensor (absolute values confidential)





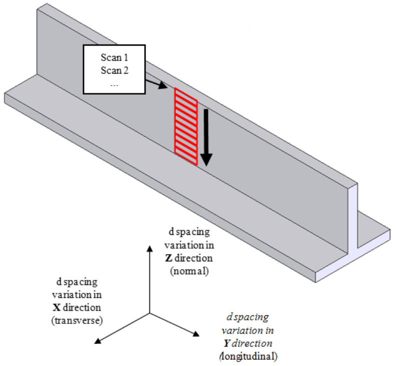
surface

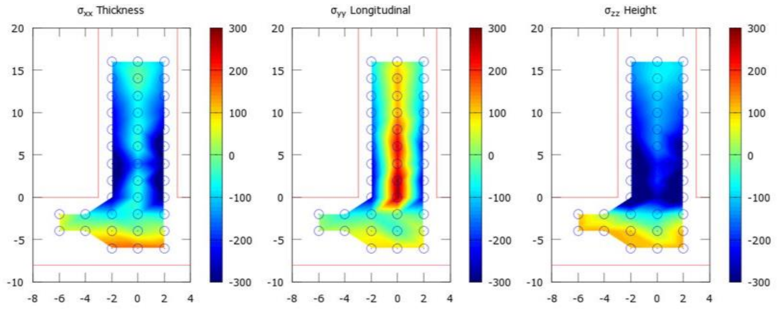
Superimposition to a micrography of a section to ease the reading

Absolute values: confidential!

Stress scanning @SALSA: additive manufacturing

- IREPA & University Cranfield: Ti64 CLAD versus WAAM





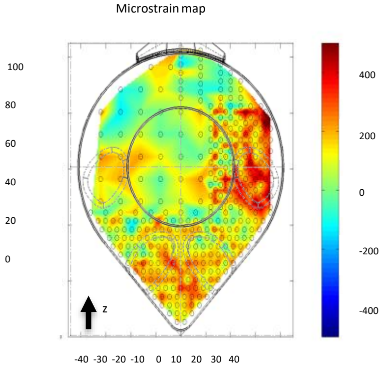
- ✓ Stress is balanced in the system sample + base plate
- ✓ Access to joint region: deep stress gradient
- ✓ CLAD has lower residual stresses than WAAM

→ Benchmark of simulations

- THALES & Renishaw: Aluminium SLM

- ✓ High strain gradient: -100 to 400 με
- ✓ Non-symmetric gradient:
  - Influence of laser scanning strategy more than geometry
  - Principal directions?

→ Stress gradient not related to geometry but to printing strategy!



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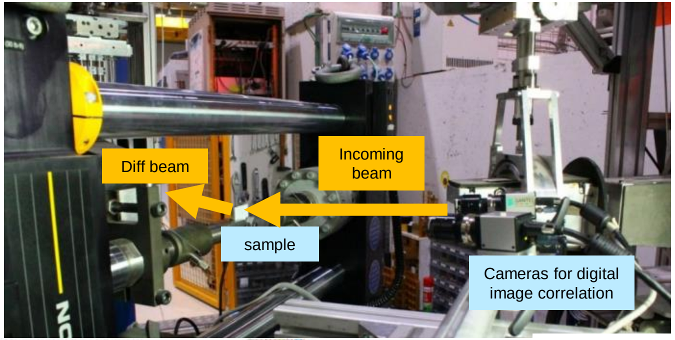
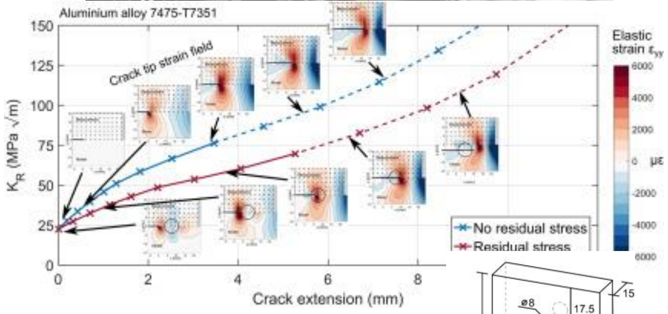
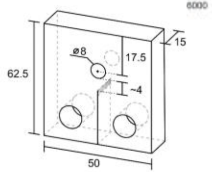
Additive manufacturing: In-situ characterization

In-situ crack growth in WAAM Ni-superalloy IN-625

- Load Rig (50KN)
- Surface (DIC) and bulk (ND) strain field around crack during loading.
- Effect of crack orientation: elastic strain distribution is anisotropic

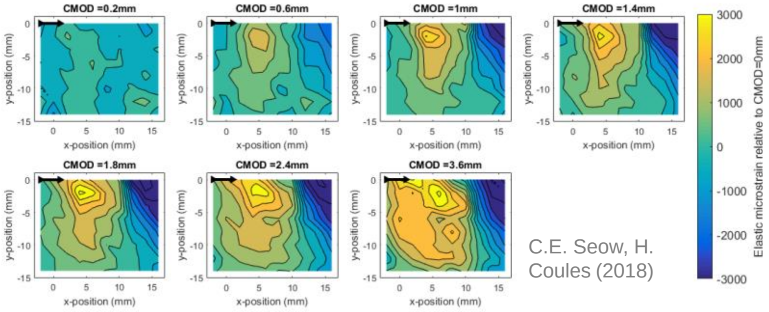






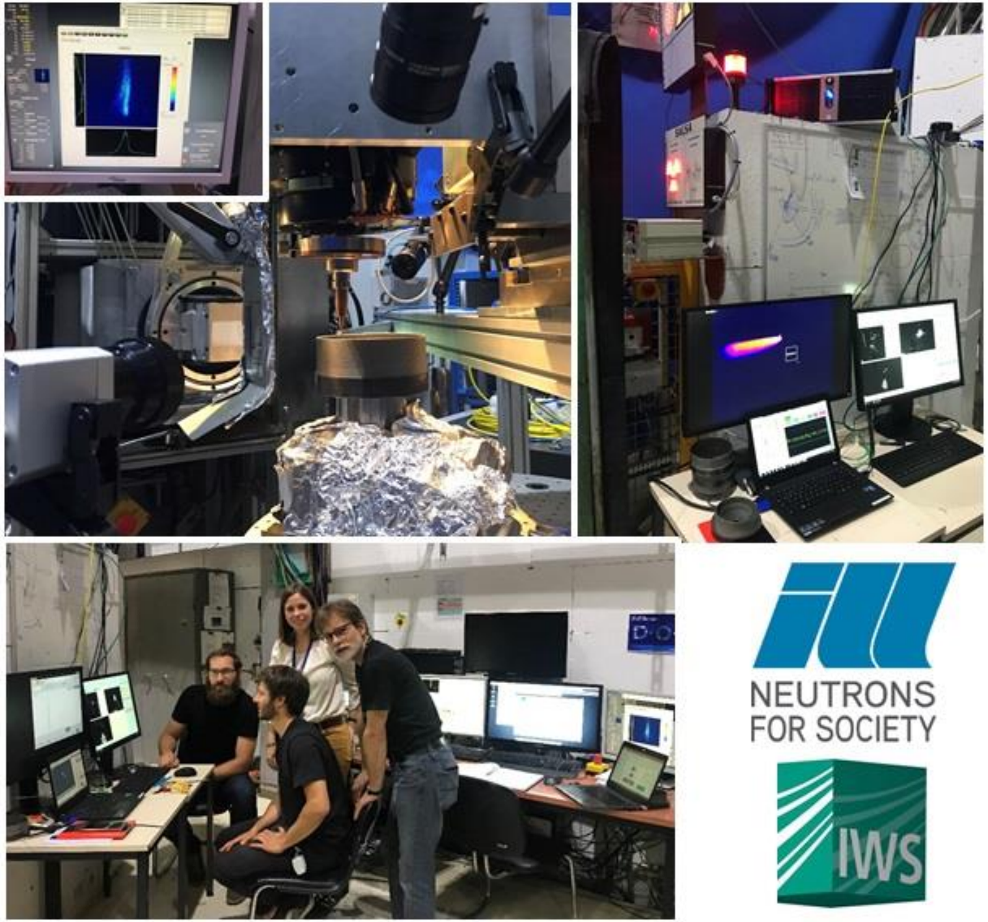
H. Coules, T. Pirling (2018)

University of BRISTOL



C.E. Seow, H. Coules (2018)

In-situ strain monitoring during continuous metal printing

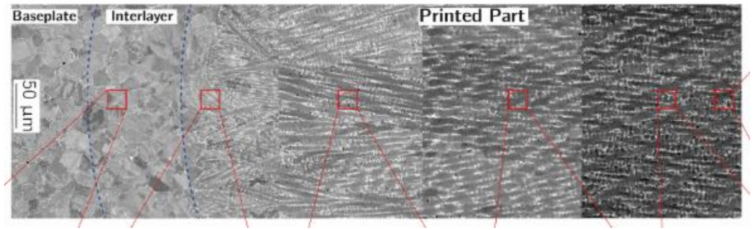


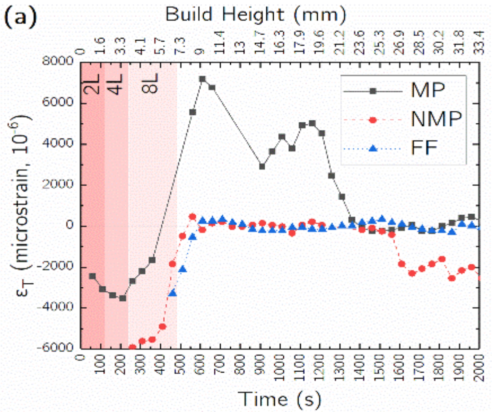
INCONEL 718

Laser Direct Metal Deposition (wire)

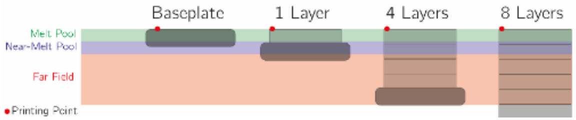
Publication 2020 Cabez et al. XX

In-situ strain monitoring during continuous metal printing
Processing considerations





$= \varepsilon_{(hkl),TH} + \varepsilon_{(hkl),M} + \varepsilon_{(hkl),S}$



- IR cameras: Stable thermal gradient is reached in the printed part at 4 mm from the melt pool after **8 layers**.
- SEM analysis: the microstructure stabilises after **4 layers**
- SALSA monitoring:
  - ❑ ***melt pool*** region stabilized after **29 layers**
  - ❑ ***near melt pool*** and ***far-field***, after **11 layers**

=> Only neutron diffraction disclosed the stable regimes and offsets of the Laser DMD process in Inconel 718

Strain Monitoring During Laser Metal Deposition of Inconel 718 by Neutron Diffraction, S Cabeza, B Özcan, J Cormier, T Pirling, S Polenz, F Marquardt, TC Hansen, E López, A Vilalta-Clemente, C Leyens, Superalloys 2020, 1033-1045

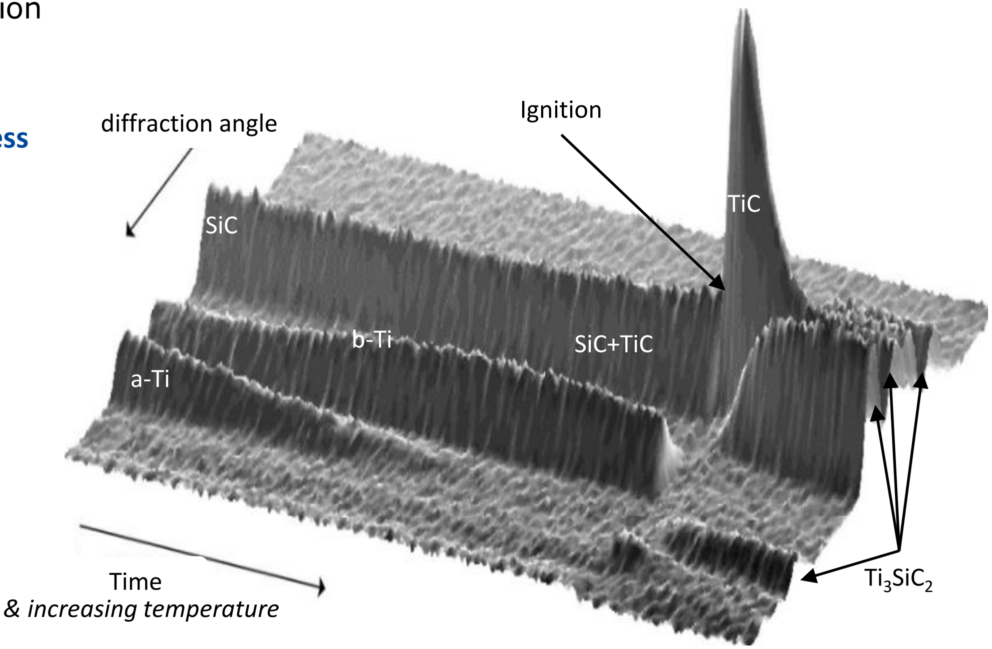
Powder diffraction: to reveal phases, location of atoms in lattice

=> Structural evolutions and phase transformations of materials under varying environmental conditions

- Various sample environments available (furnace, cryostat, magnet, pressure cell, magnetic levitation, etc.)
- Spatially resolved phase analysis, texture determination

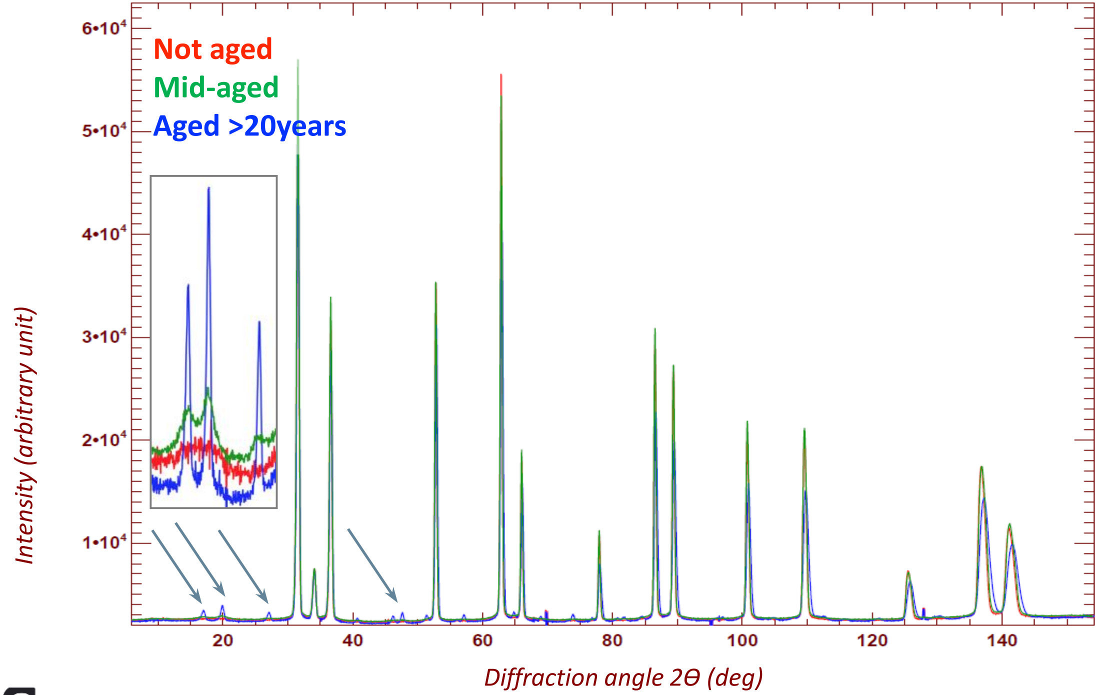
Investigation of alloy formation during a synthesis process

In situ neutron diffraction of the high-temperature ceramic material Ti3SiC2 was performed. Powder diffraction patterns were taken every 0.9 seconds while the precursors were heated from 850°C to 1050°C. Riley at al. 2006



Neutron diffraction
=> Structure of aged Ni-Cr based alloys

- **Motivation**: understanding alloy ageing
- Neutron diffraction in complement of several other techniques



SINE2020 project receives funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 654000.

Neutron Radiography and tomography

specific contrast schemes

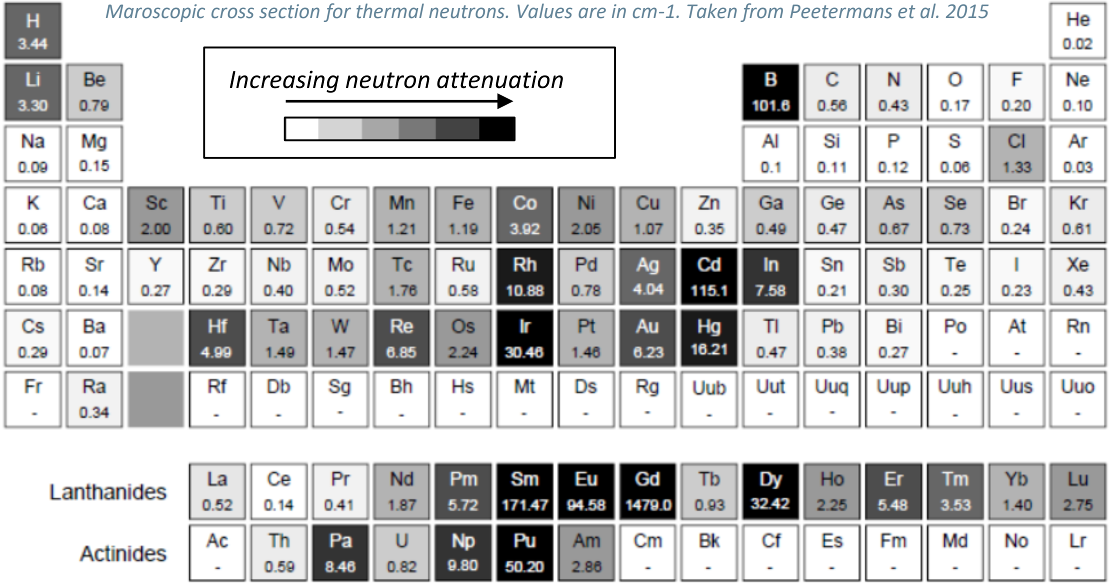
Radiography and tomography @ ILL: NEXT

UNIVERSITÉ Grenoble Alpes

- Main features:
  - **Simultaneous x-ray and neutron tomography**
  - Spatial resolution: from 4 µm
  - Max field of view: 16x16 cm²

- Examples of use cases: highly x-ray absorbent devices, quantitative imaging of fluid flows, residual water in metalic pieces, boron distribution.

A polymer joint is missing: this is easily visible through the metallic structure. Picture: LLB-CEA



Maroscopic cross section for thermal neutrons. Values are in cm-1. Taken from Peetermans et al. 2015

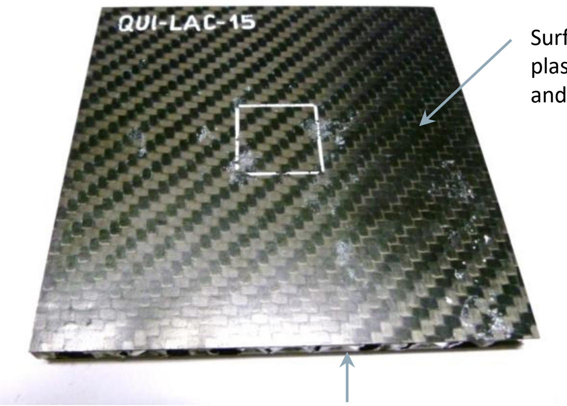
Neutron imaging: control of gluing integrity

- Aircraft composite materials

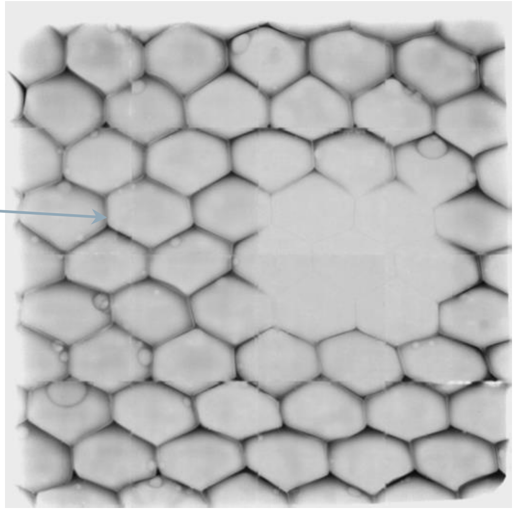
  Issue: detection of glue (low atomic number) within the metallic matrix (principally very complicated for X-rays)

  => **Defective area clearly detected**

Aircraft composite material



***Neutron** radiography*

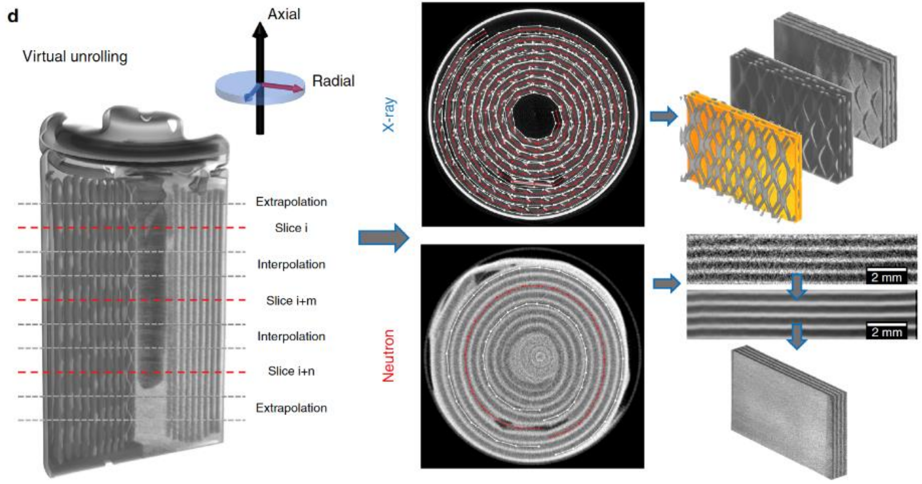


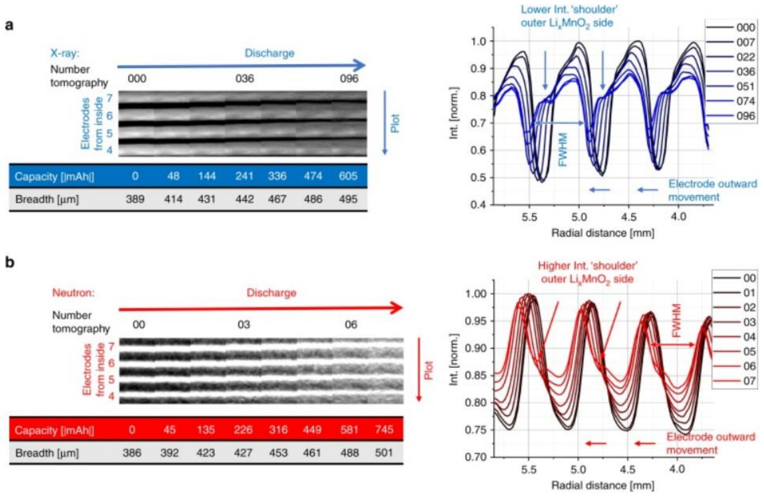
Source: ILL

4D imaging of lithium-batteries using correlative neutron and X-ray tomography

⇒**virtual unrolling technique:** temporally and spatially resolved tracking of lithium intercalation and electrode degradation

Ziesche RF, Arlt T, Finegan DP, et al. 4D imaging of lithium-batteries using correlative neutron and X-ray tomography with a virtual unrolling technique. *Nat Commun*. 2020;11(1):777. Published 2020 Feb 7. doi:10.1038/s41467-019-13943-3





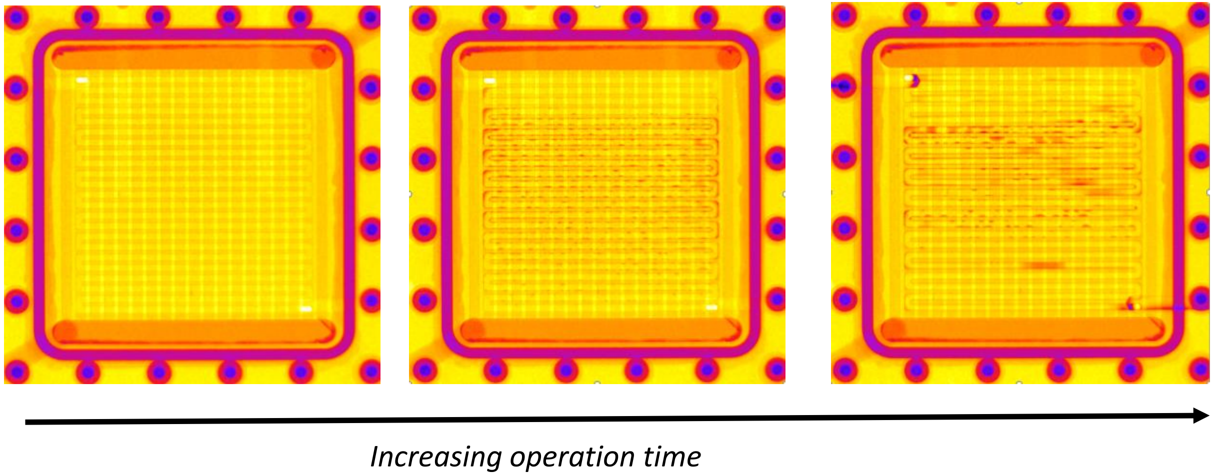
X-rays

neutrons

NEUTRONS FOR SOCIETY

Real-time neutron imaging: following water path

- Water path and quantification within running a fuel cell (CEA-LITEN, ILL, UGA)



Neutron imaging of operando proton exchange membrane fuel cell with novel membrane, Lee et al., 2021, J. Power Sources, https://doi.org/10.1016/j.jpowsour.2021.229836
Quantitative multi-scale operando diagnosis of water localization inside a fuel cell, Morin et al., 2017, J. Electrochem. 10.1149/2.1401614jes

Neutron radiography: an example
Water flow in geomaterials

UNIVERSITÉ Grenoble Alpes

Real-time imaging

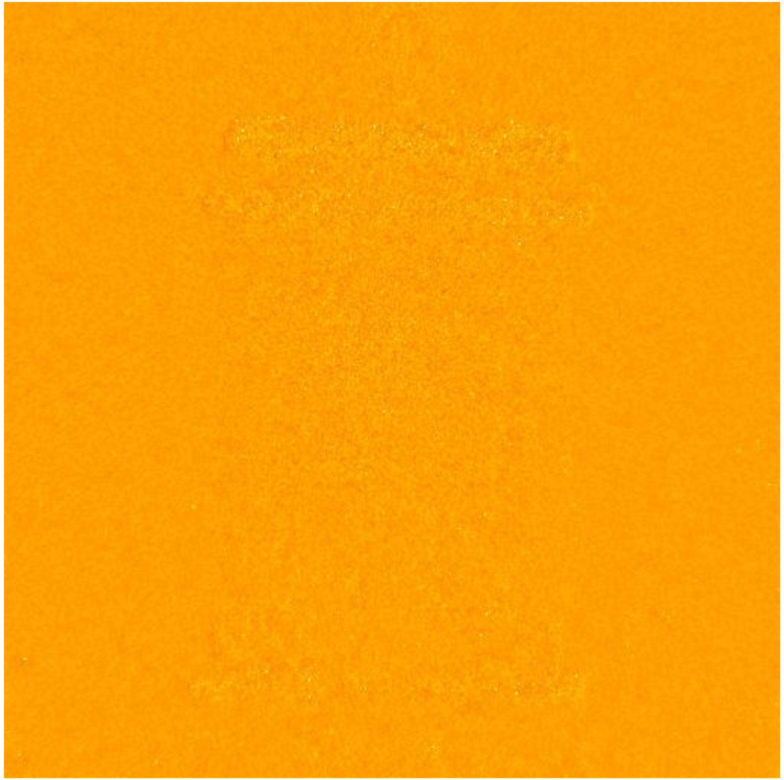
A. Tengattini, Uni. Grenoble Alpes

=>tracking of water pathways: infiltration, drying, etc.

Water within the rock is easily visible: red, violet then black indicate an increasing water content.

Rock sample: 4cm in diameter, 7 cm in height

1 image taken every 100 ms



Small-Angle Neutron Scattering SANS

statistical information on features in the range 1-900nm

Reflectometry

investigation of layers in the range 1-600 nm

Small angle Neutron scattering
Silica-Rubber Composites

- Neutrons => polymer
- X-rays => silicium

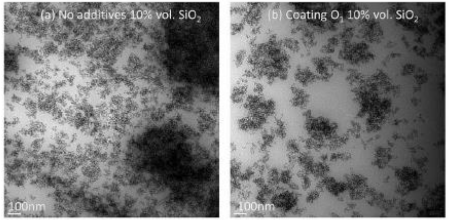
- Several findings…

Under stretching, the nanoparticles acts as an additional cross-linked junction

Bouty et al, 2016, Interplay between polymer chain conformation and nanoparticle assembly in model industrial silica/rubber nanocomposites

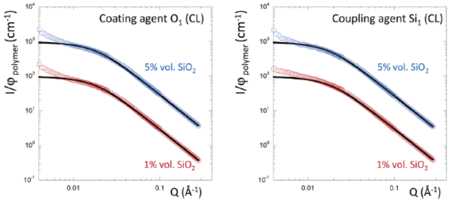
Direct pictures of the microstructure

TEM



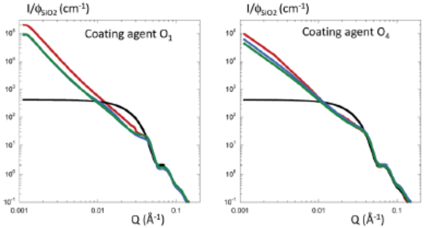
Neutrons are mainly sensitive to the polymer, statistical info under operational conditions

SANS



X-rays are mainly sensitive to Silicium, statistical info under operational conditions

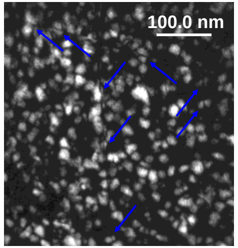
SAXS

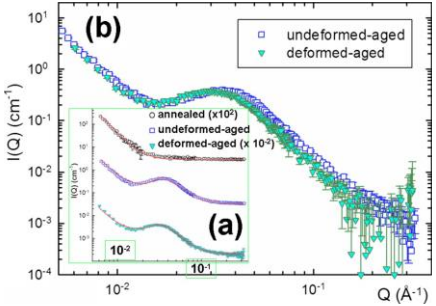


NEUTRONS FOR SOCIETY

SANS: Nanoprecipitates in alloys

SANS can probe the **nanostructures of precipitates** and nucleation in metal.

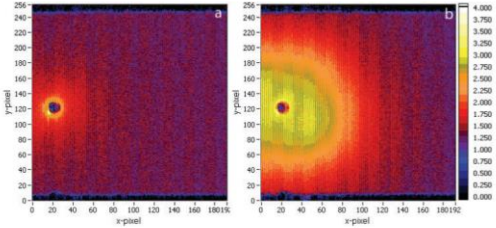


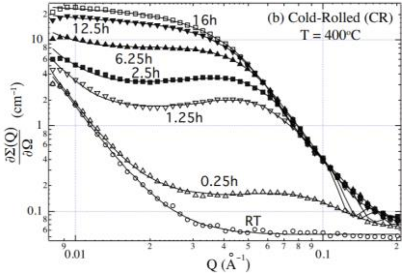


Ni based nanoprecipitates ~ ellipsoids

E. W. Huang et al. Applied Physics Letter 93, 161904(2008)

In-situ SANS can obtain the **quantitative information of growth rate of nanoprecipitates**

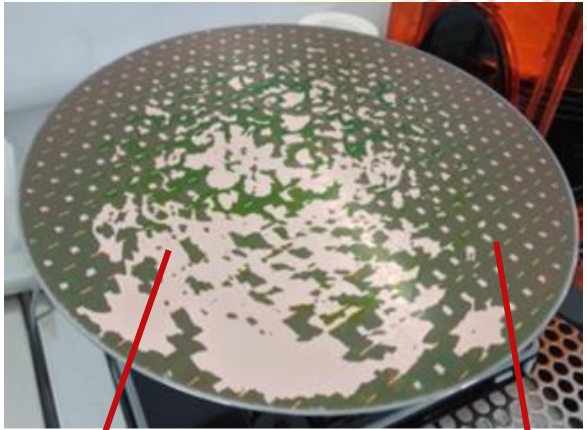




Precipitate evolution in Ti-5Al-5Mo-5V-3Cr-0.3Fe ~disk shape

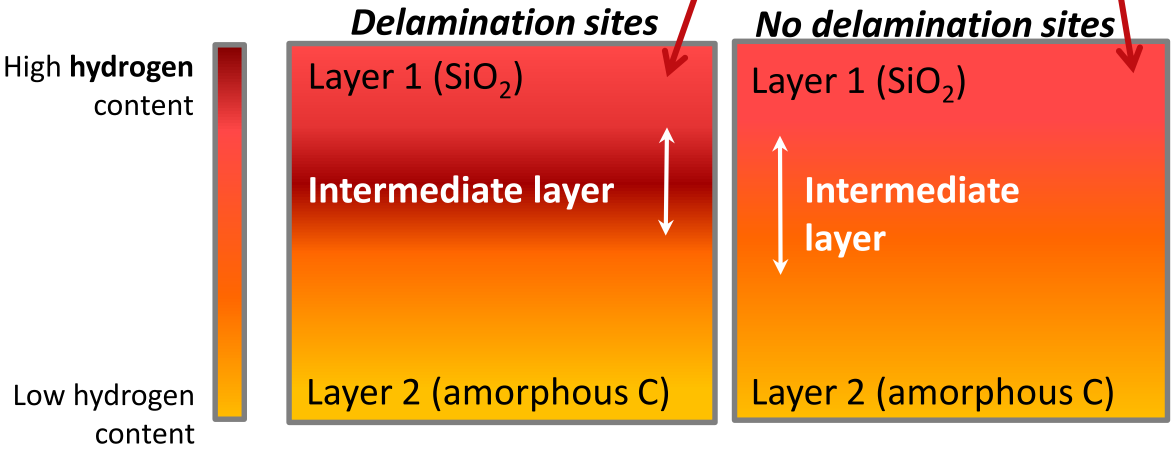
James Coakley et al. Journal of Alloys and Compounds 623, 146(2015)

Neutron Reflectometry: Investigation of interfaces on silicon wafers



- Wafers exhibiting delamination have been investigated using neutron reflectometry.

=> The wafer that undergoes delamination has a high H-content within a thinner intermediate layer.



Segura et al. 2015, Hydrogen accumulation as the origin of delamination at the a-carbon/SiO2 interface, *Journal of Applied Physics*

Irradiation with neutrons (and more)

Low energy neutrons (TENIS)

Main Applications

- Single-event effects (SEE) testing
- Qualification of HiRel components for ground and aeronautical applications

14MeV energy neutrons (GENESIS)

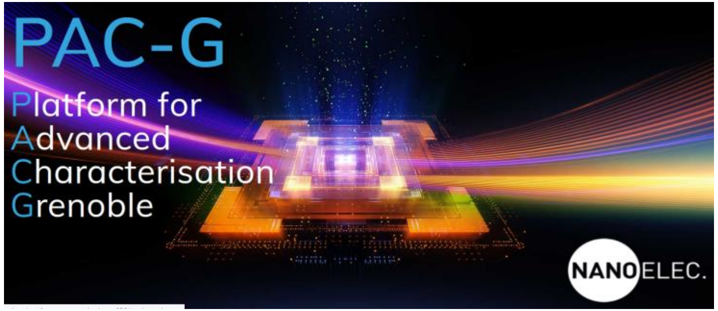
Main Applications

- Ramdon fault injection on large systems
- Single-event effects (SEE) testing for microelectronics
- Pre-qualification of components
- Debug and preparation for high energy protons testing

Pulsed synchrotron X-rays focused beam

Main Applications

- Spatially localised fault injection on microchips
- Single-event effects (SEE) testing for microelectronics
- Pre-assessment of the sensitivity of a microchip
- Debug and preparation for heavy ion testing



Irradiation of electronic components at ILL

- **T**hermal and **E**pi-thermal **N**eutron **I**rradiation**S** (**TENIS**)
  - Beam with a fission spectrum
    - Thermal neutrons (E<0,625 eV): 88%
    - Epithermal neutrons (0,625<E<1MeV): 12%
    - Fast neutrons (E>1MeV): 0,585%
  - $Flux_{th+Eth}$: 2,4 $n.cm^{-2}.s^{-1}$ estimated by Au foils activation at the sample position for a reactor power of 55MW
  - Adjustable beam size from 1x1 mm^2 to 50 x 50 mm^2

=> Ideal for single-event testing of COTS components and devices from borated processes

Neutron beam spectrum (computed)

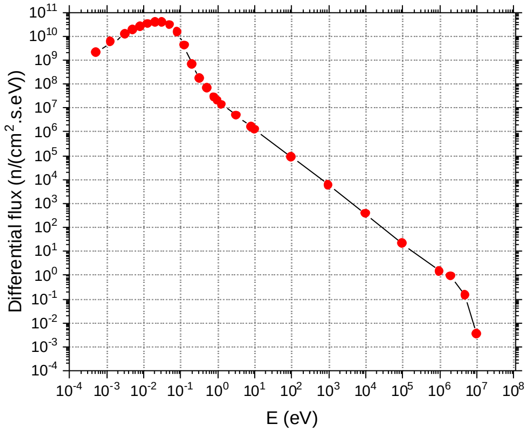
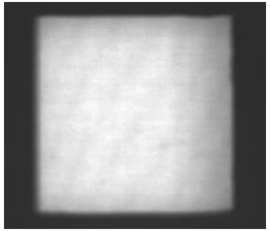


Image of the beam



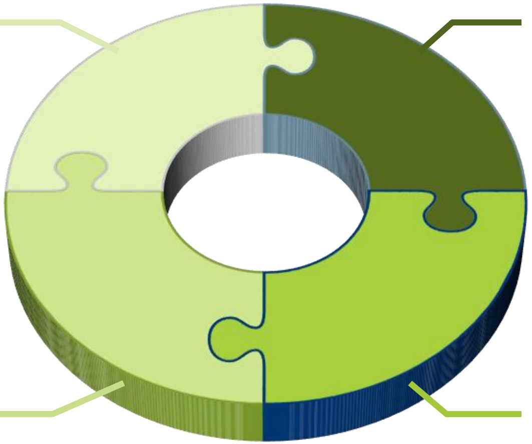
  - A calulated flux of gamma coming from the reactor, **compatible with Total Iionizing Dose testing**
    - $\Phi\gamma = 7.5 \times 10^8\ \gamma/cm^2/s$
    - Dose Rate = 18 Gy/h (1,8 KRad/h)
    - Maximum dose rate reachable using Cd: 115 Gy/h (11,5 kRad/h)

How to work with the ILL and the ESRF?

Public Beam Time

Results must be published, free of charge

- Universities with Industry
- Industry on its own
- Innovation-led long term projets



Client Services

- Rapid access
- Full IP rights to client
- Paid-for services

Technology Transfer

- Licencing technologies
- In-house manufacturing
- Consultancy

Collaboration and Grants

- Industry sponsored staff (PhD, post-doc, trainees...)
- Horizon Europe, French PIA, IRT, UK CASE, etc.

Thanks for your attention!

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