MANCHESTER
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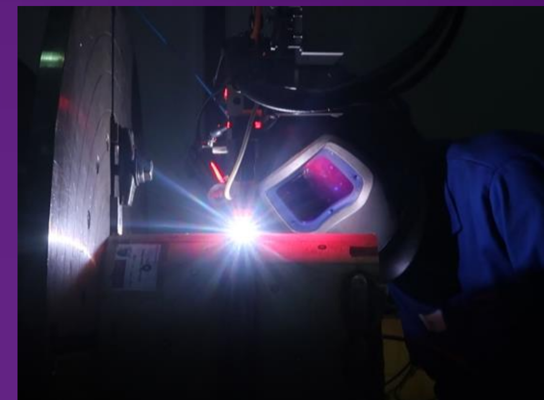
18th Oxford School on Neutron Scattering
13th September 2024



Neutrons in Engineering

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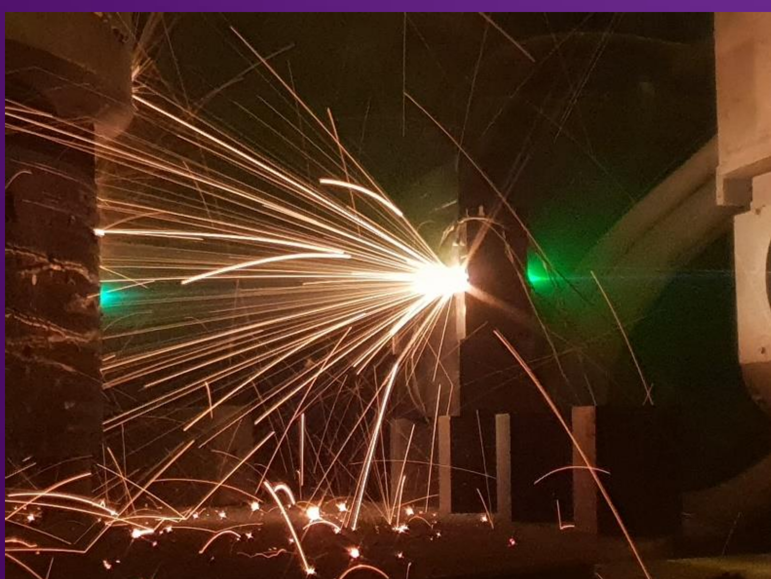


Outline

- Neutron diffraction in engineering
- Stress, strain and residual stress
- Bragg's Law
- Set-up of Engineering Instrument
- Diffraction methods available
- Residual stress measurement techniques
- Case Studies
- Conclusions on neutrons pros/cons

Introduction

Problem setting

Neutrons in Engineering

Neutron diffraction (ND) in engineering is typically used for:

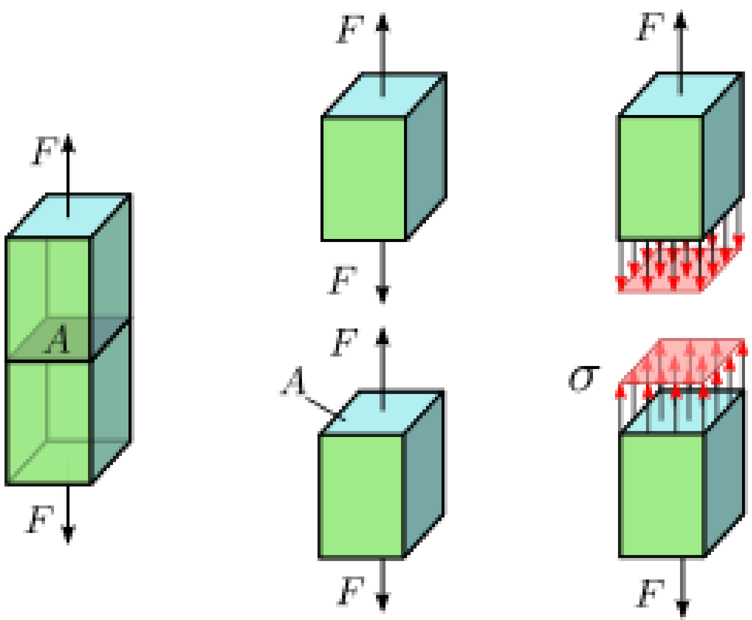
- measuring **residual stress (RS)** in materials,
- assessing **crystal structure alterations or damage** caused by exposure to severe conditions (e.g., high temperature, pressure, or radiation),
- studying **deformation heterogeneity**, such as variations in strain or stress distribution within a material.

But first…back to basic concepts and principles.

Stress, Strain

Basic Engineering Principles

What is Stress?



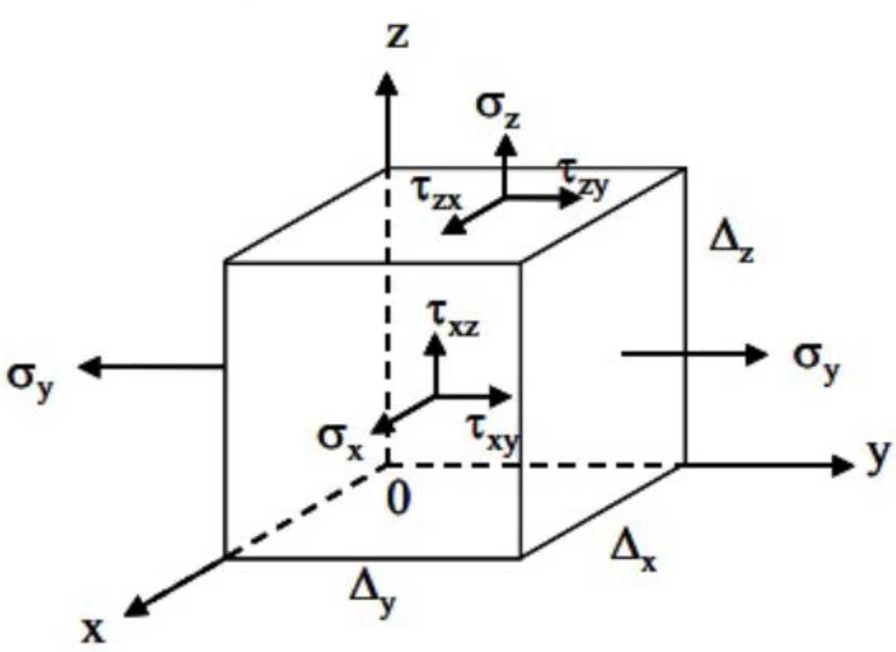
https://en.wikipedia.org/wiki/Stress_%28mechanics%29

$$\sigma = \frac{F}{A}$$

Where:

σ = stress (Pascals, Pa, or N/m²)
F = force (Newtons, N)
A = cross-sectional area over which the force is applied (in square meters, m²)

Stress Types & Components



σ_x / σ_y / σ_z :

- **Normal** stress acting in the x/y/z direction.
- Acts on plane **perpendicular** to the x/y/z direction.

Convention

✓ positive values → tensile stress

✓ negative values → compressive.

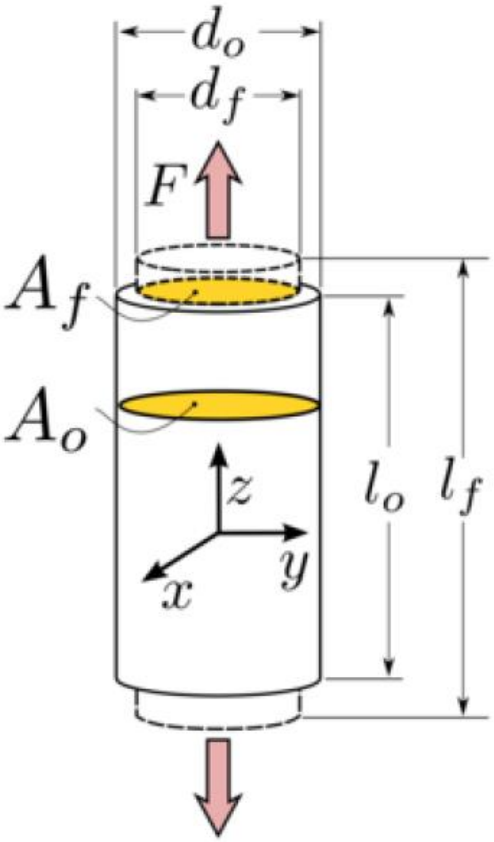
τ_{xy} / τ_{xz} / τ_{yz} :

- **shear** stress
- Acts on plane parallel to the x/y/z planes.

Can we measure Stress?

.... we can measure Strain!

What is Strain?



Engineering strain is captured by:

$$e = \frac{l_f - l_o}{l_o}$$

True strain is captured by its rate of change:

$$d\varepsilon = \frac{dl}{l}$$

$$\varepsilon = \int_{l_o}^{l} \frac{dl}{l} = \ln\left(\frac{l_f - l_o}{l_o} + 1\right)$$

$$\varepsilon = \ln(e + 1)$$

From Strain to Stress

- **Hooke's Law**
- $\boldsymbol{\sigma = E\varepsilon}$

(simple uniaxial tension/compression)

Where:
σ = stress (Pa)
E= Young's modulus or modulus of elasticity (Pa), which measures the material's stiffness
ε = strain (dimensionless)

- 3D equation (tensor form)

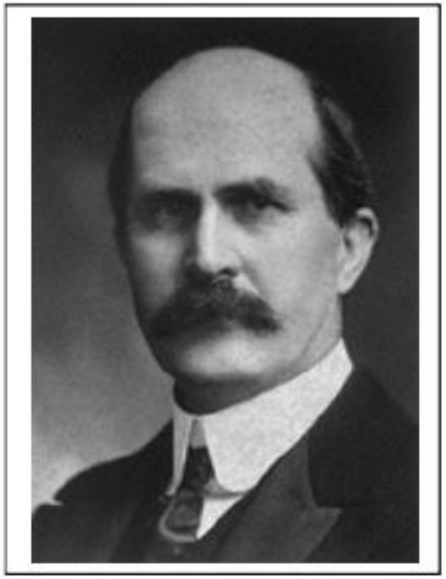
$$\begin{bmatrix} \sigma_{11} \\ \sigma_{22} \\ \sigma_{33} \\ \sigma_{23} \\ \sigma_{13} \\ \sigma_{12} \end{bmatrix} = \frac{E}{(1+\nu)(1-2\nu)} \begin{bmatrix} 1-\nu & \nu & \nu & 0 & 0 & 0 \\ \nu & 1-\nu & \nu & 0 & 0 & 0 \\ \nu & \nu & 1-\nu & 0 & 0 & 0 \\ 0 & 0 & 0 & \frac{1-2\nu}{2} & 0 & 0 \\ 0 & 0 & 0 & 0 & \frac{1-2\nu}{2} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1-2\nu}{2} \end{bmatrix} \begin{bmatrix} \varepsilon_{11} \\ \varepsilon_{22} \\ \varepsilon_{33} \\ 2\varepsilon_{23} \\ 2\varepsilon_{13} \\ 2\varepsilon_{12} \end{bmatrix}$$

Assuming isotropic material

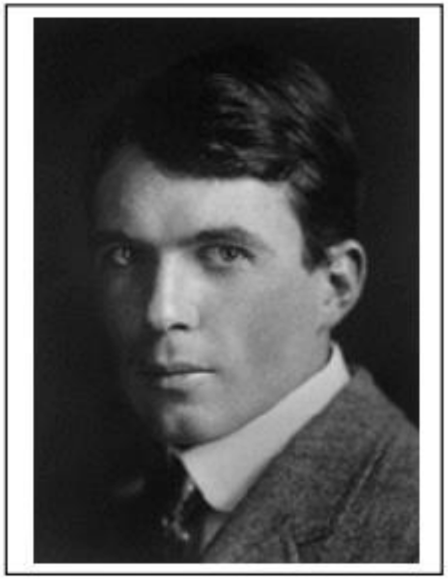
Bragg’s Law!

Diffraction Basic Principles

The Braggs



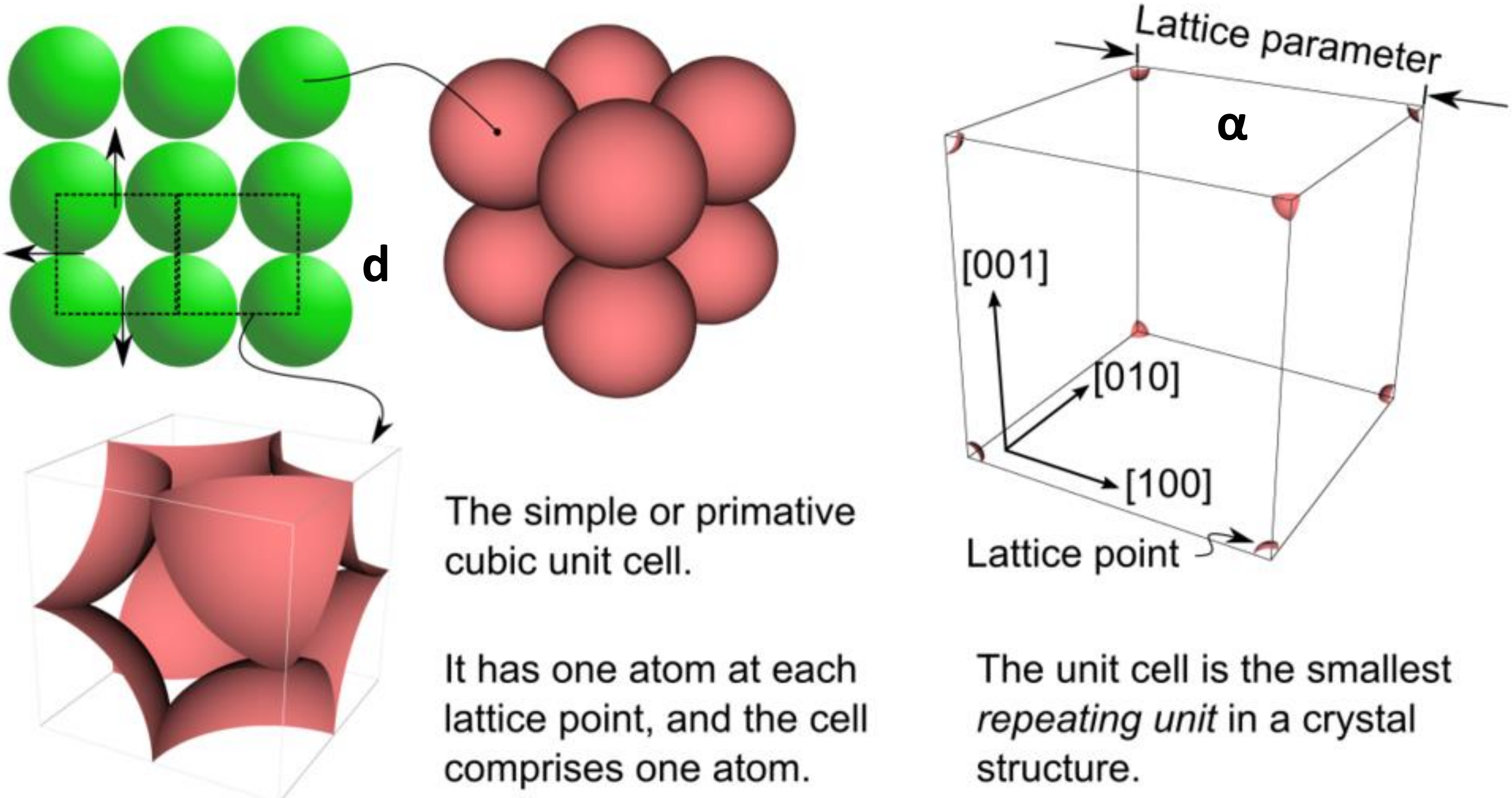
W.H. Bragg
(1862–1942)



W.L. Bragg
(1890–1971)

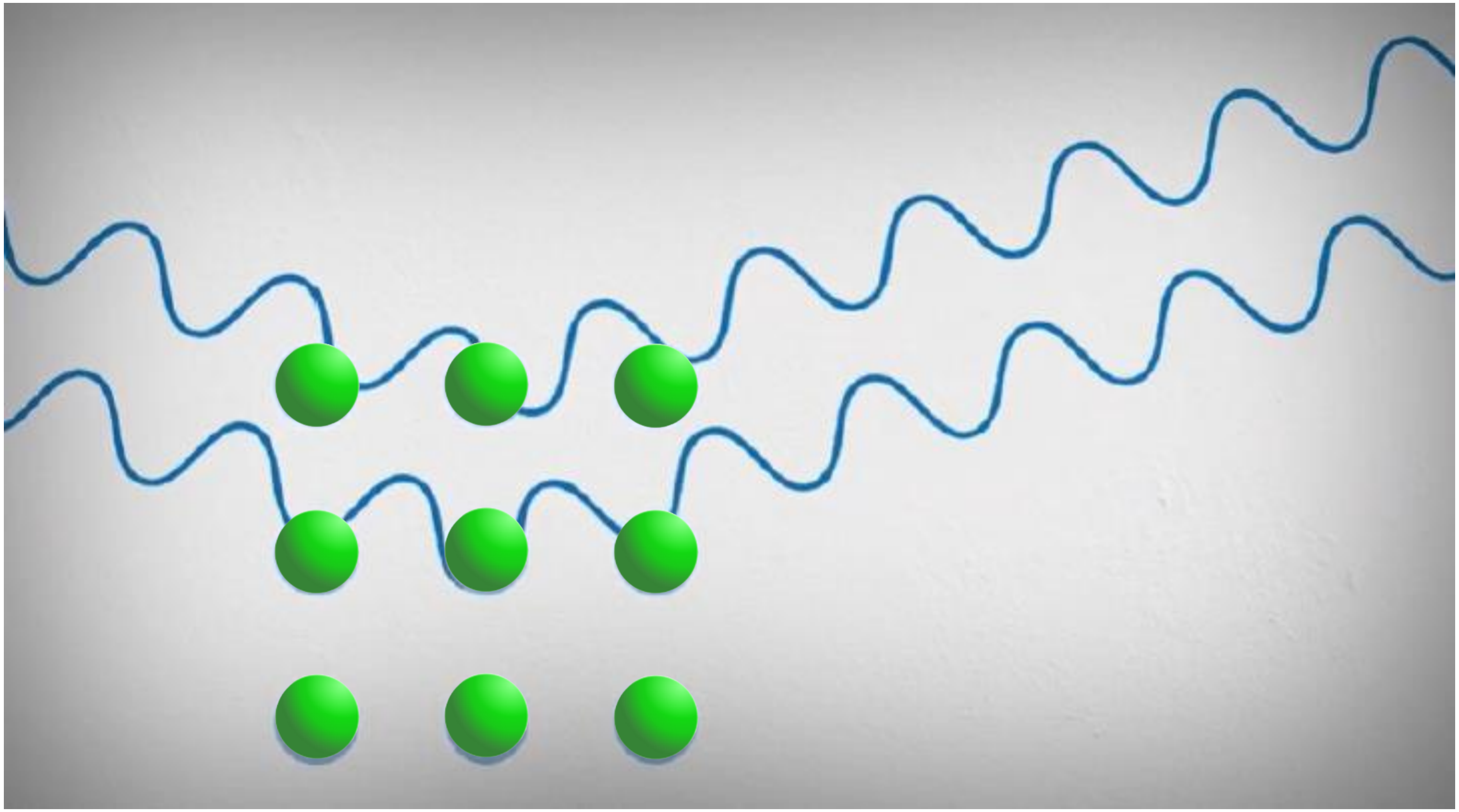
Nobel Prize in Physics (1915)

Unit cell, d spacing, lattice parameter α

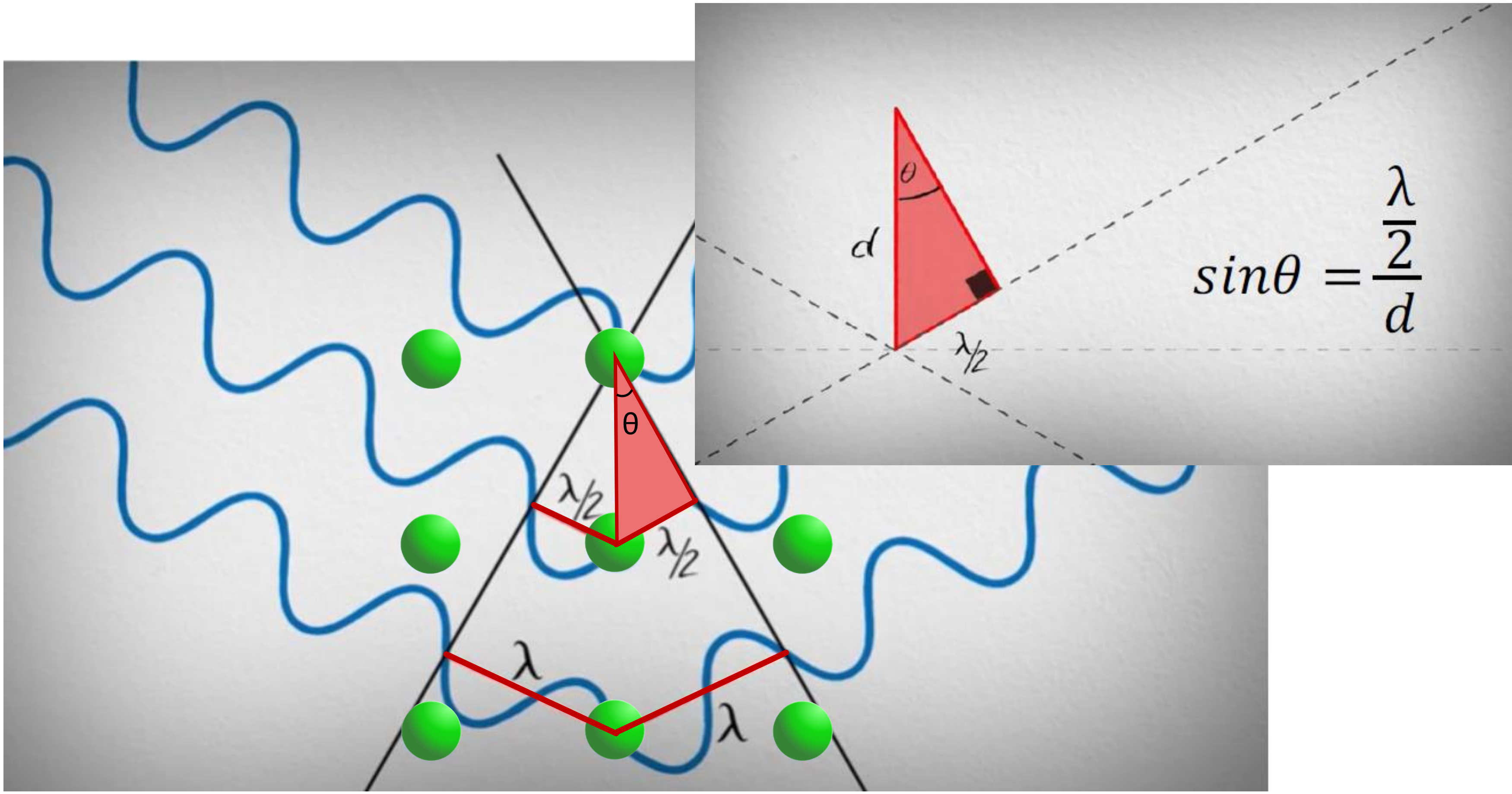


(2020, M.J. Roy) Materials 1, Teaching material, The University of Manchester

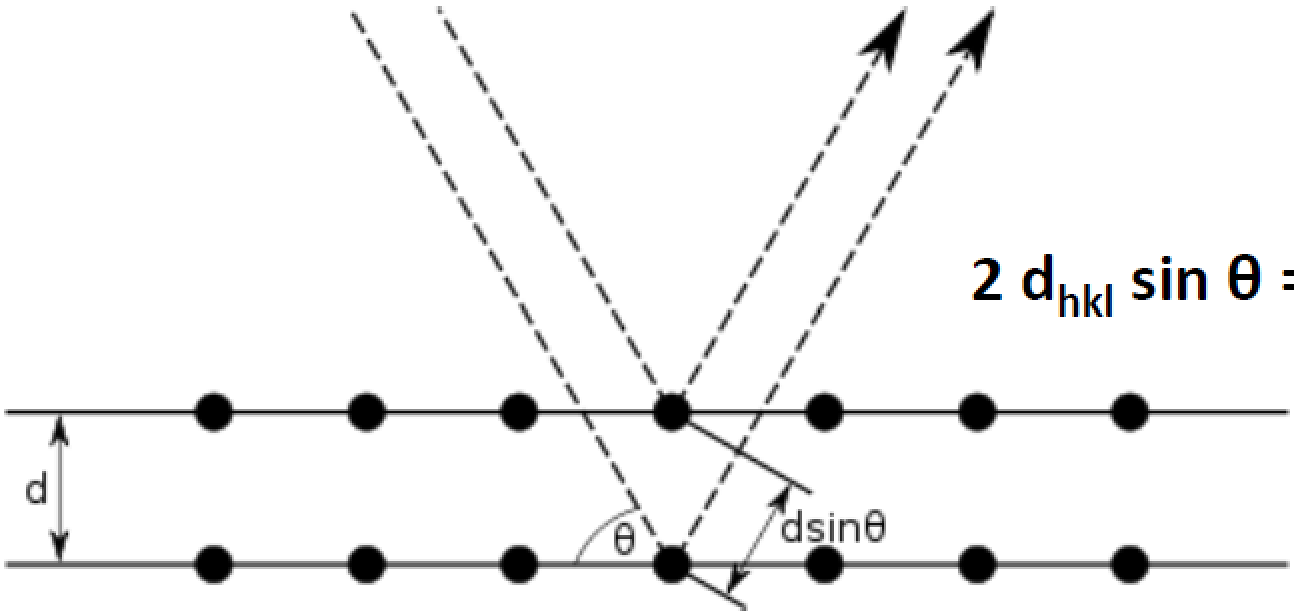
Bragg’s Law



Bragg's Law



Bragg's Law



$$2\, d_{hkl} \sin\theta = n\,\lambda$$

λ : the wavelength of the incident radiation (fixed in most diffractometers)

d_{hkl} : the distance between the (hkl) planes (geometric function of the size and shape of the unit cell)

θ : the incident angle (the angle between the planes and the incident beam)

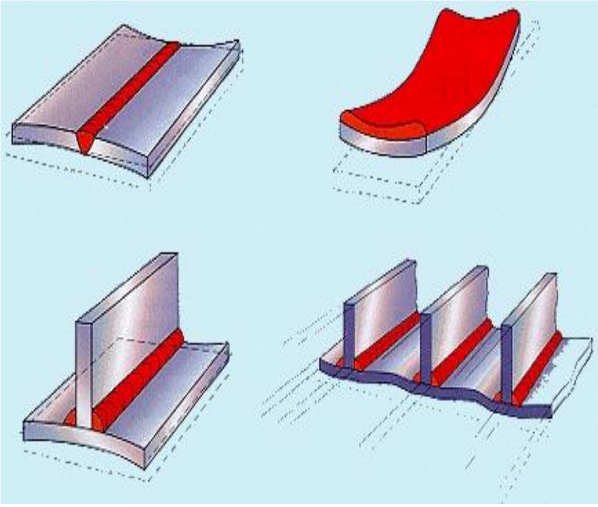
n : the diffraction order (integer)

Residual Stress

Definition

What is Residual Stress?

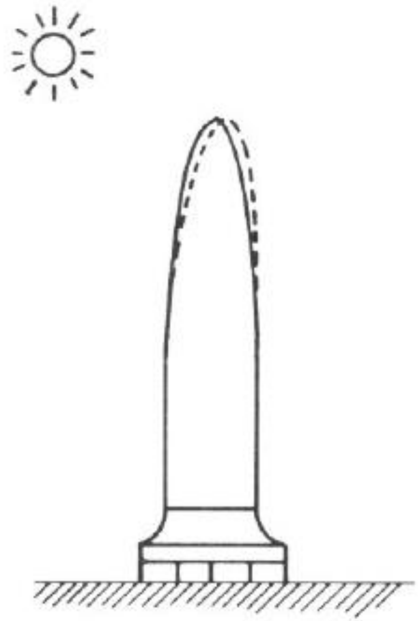
Residual stresses: Stresses that **remain** in a solid material after the original cause of the stresses has been removed.



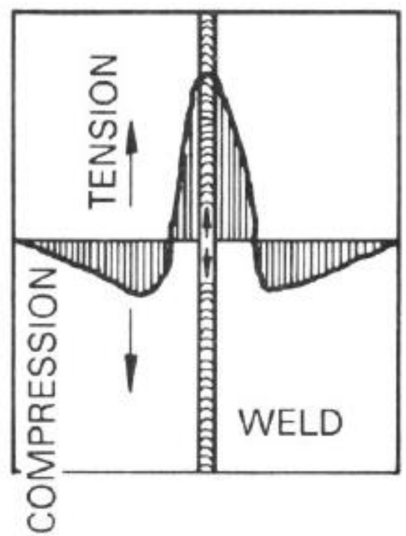
Occur through various mechanisms:

- Plastic deformations
- Temperature gradients (thermal cycle)
- Structural changes (phase transformations)

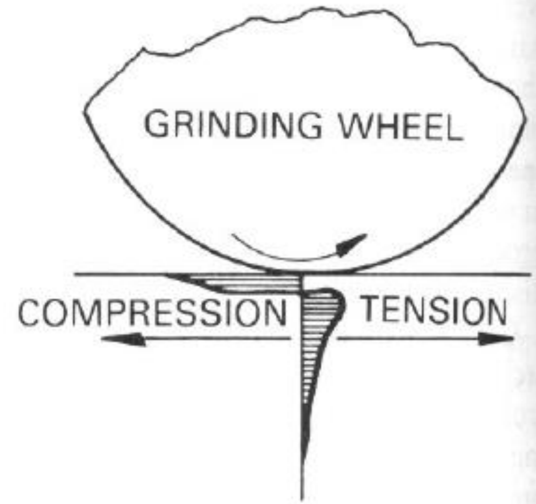
Examples of Residual Stress



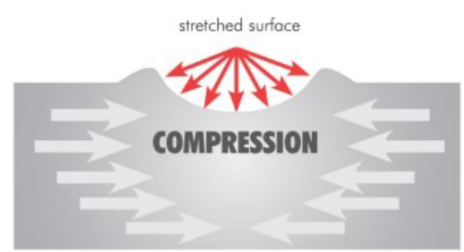
(A) Thermal Distortion in a Structure Due to Solar Heating



(B) Residual Stresses Due to Welding



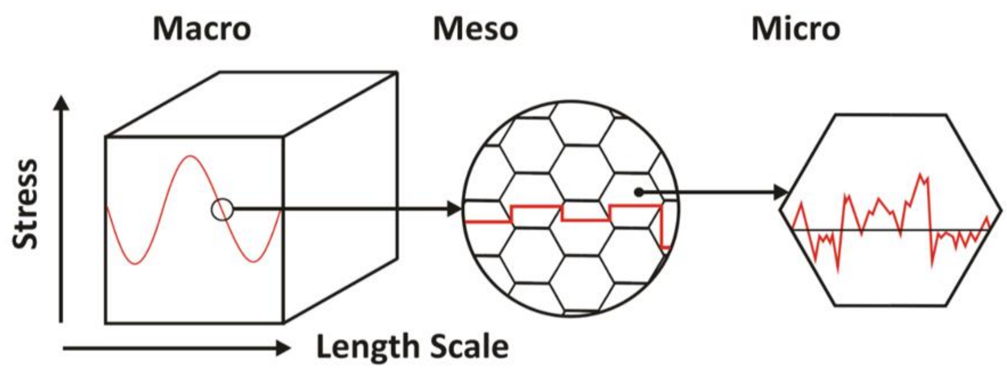
(C) Residual Stresses Due to Grinding

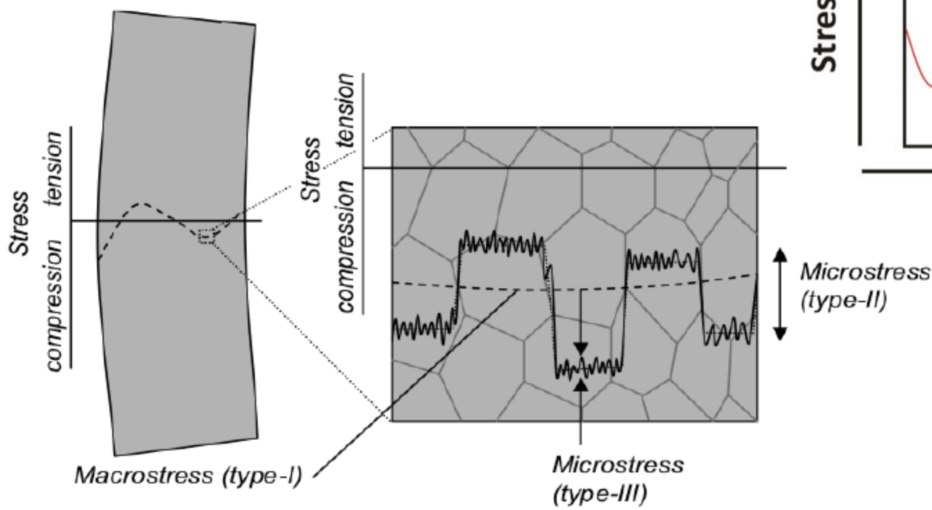


Shot peening process

Residual Stress Types

- TYPE I (long range over multiple grains, macrostresses)
- TYPE II (intergranular, vary between grains, microstresses)
- TYPE III (atomic level)





Bent Bar

Internal stresses
Caused by a misfit

Effects of Residual Stress

- **Positive Effects**: Compressive residual stress on the surface of a material can **increase** its
  - **fatigue strength,**
  - **wear resistance, and even**
  - **resistance to crack propagation.**
- **Negative Effects**: Tensile residual stress, on the other hand, can **weaken** the material, making it more
  - **susceptible to crack initiation and growth,**
  - **susceptible to corrosion, and**
  - **failure under lower loads.**

Managing residual stress is crucial in many engineering applications to ensure the durability and performance of components.

Why care about Residual Stress?

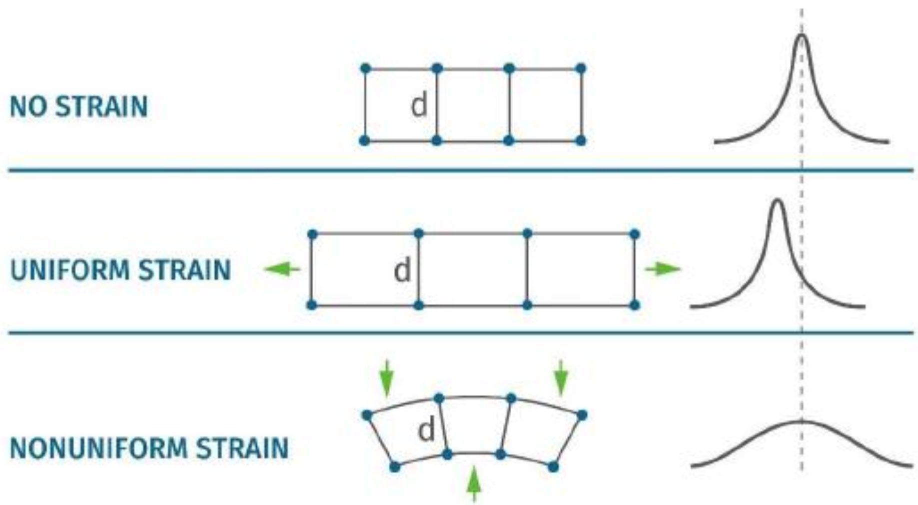
- Play a significant role in affecting the long-term structural performance.
- Can contribute to the driving force for crack growth.
- Can activate degradation mechanisms such a creep and stress-corrosion cracking, even in the absence of operating stresses.
- Cause deformation *(distortion, dimensional accuracy).*

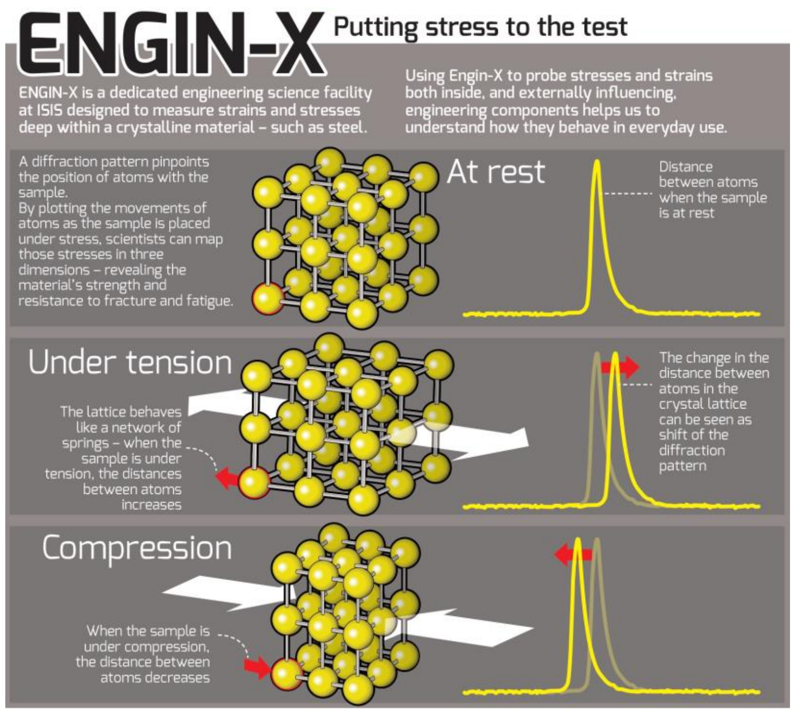
Diffraction for Measuring Residual Stress

Overview

Effect of elastic strain on diffraction signal

- When measuring residual stress, we actually measure residual STRAIN!! *(stress is strain energy density)*
- Diffraction measures elastic lattice strain as **peak shifts**
- Uses the poly-crystalline lattice planes as internal strain gauges



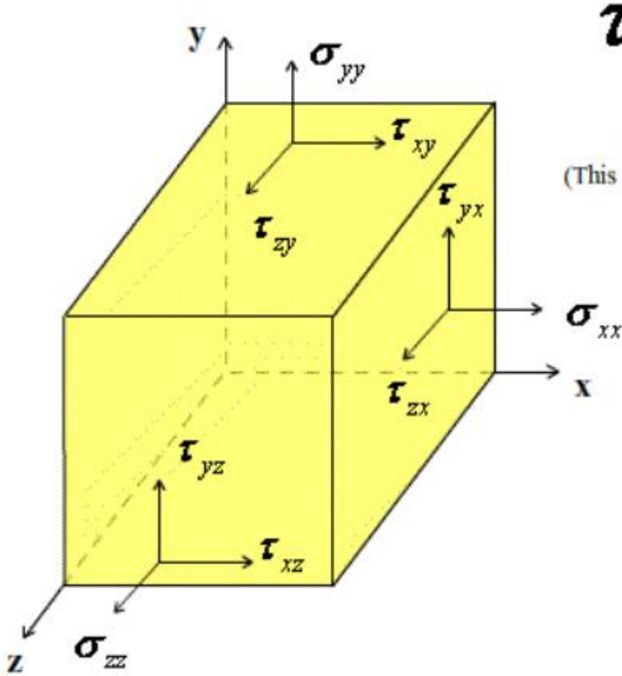


Data Analysis Workflow

Measured strains have to be converted into stresses! (Hooke's law)

$$\varepsilon = \frac{a - a_0}{a_0} = \frac{d - d_0}{d_0}$$

The 9 components of a stress tensor:



The stress acts in the x-direction

τ_{xy}

on the plane with a normal in the y direction
(This convention maybe vice versa in some books.)

$$\sigma_{ij} = \begin{pmatrix} \sigma_{xx} & \tau_{xy} & \tau_{xz} \\ \tau_{yx} & \sigma_{yy} & \tau_{yz} \\ \tau_{zx} & \tau_{zy} & \sigma_{zz} \end{pmatrix}$$

Tensor Equation: $\sigma_{ij} = C_{ijkl}\varepsilon_{kl}$

Matrix Equation: $\sigma_p = C_{pq}\varepsilon_q$

Data Analysis Workflow

- Measured strains have to be converted into stresses! (Hooke' s law)

$$\varepsilon = \frac{a - a_0}{a_0} = \frac{d - d_0}{d_0}$$

e.g. isotropic triaxial along principal directions:

$$\varepsilon_{11} = \frac{1}{E}\left[\sigma_{11} - \upsilon(\sigma_{22} + \sigma_{33})\right]$$

$$\varepsilon_{22} = \frac{1}{E}\left[\sigma_{22} - \upsilon(\sigma_{33} + \sigma_{11})\right]$$

$$\varepsilon_{33} = \frac{1}{E}\left[\sigma_{33} - \upsilon(\sigma_{11} + \sigma_{22})\right]$$

To calculate a stress direction:

$$\sigma_{11} = \frac{E}{(1+\nu)(1-2\nu)}\left[(1-\nu)\varepsilon_{11} + \nu(\varepsilon_{22} + \varepsilon_{33})\right]$$

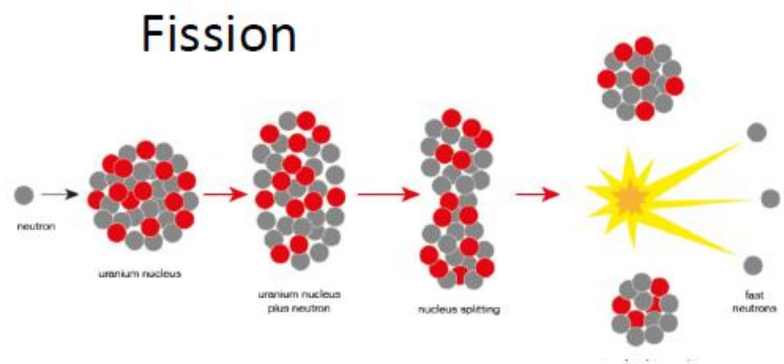
(Attention: not always this simple!)

Types of neutron sources

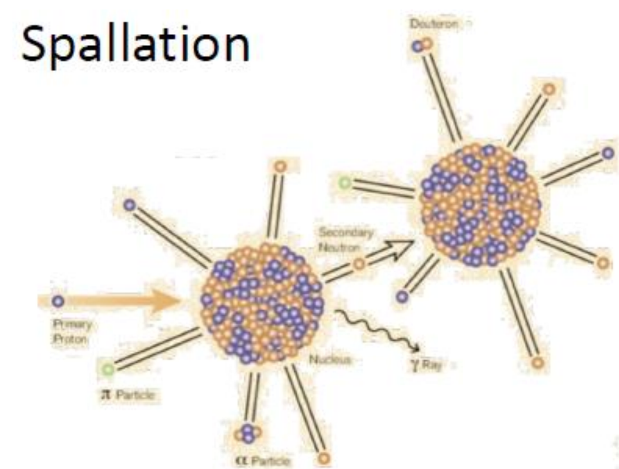
Overview, set-up, typical examples

Neutron Sources

- **Reactor-based (steady state/continuous)**
  - Slow neutron capture of 235 U
  - Constant wavelength/Single Peak
  - Fission source



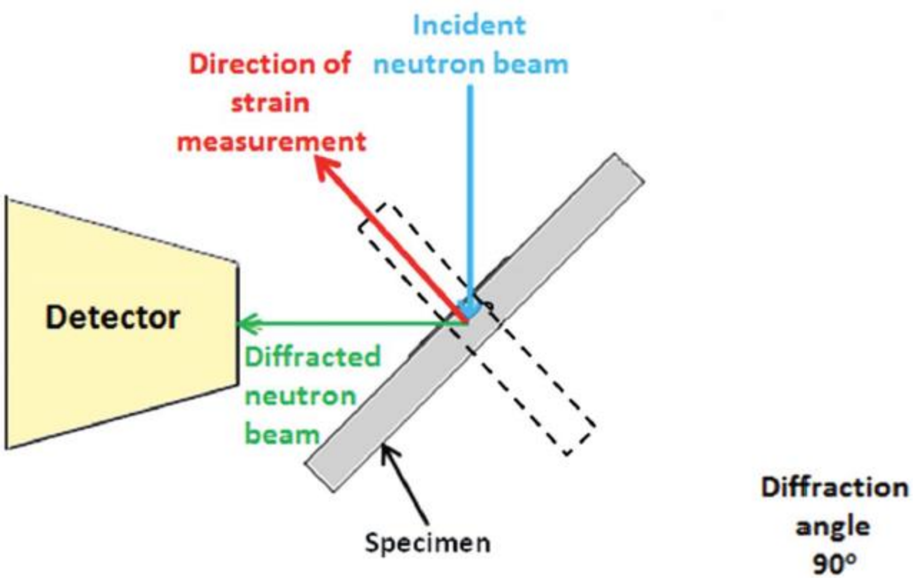
- **Short-pulse spallation (pulsed)**
  - Nuclei bombarded with high energy particles
  - Accelerator Sources
  - Time-of-flight (ToF) / Full Spectra / Riet



Monochromatic vs ToF

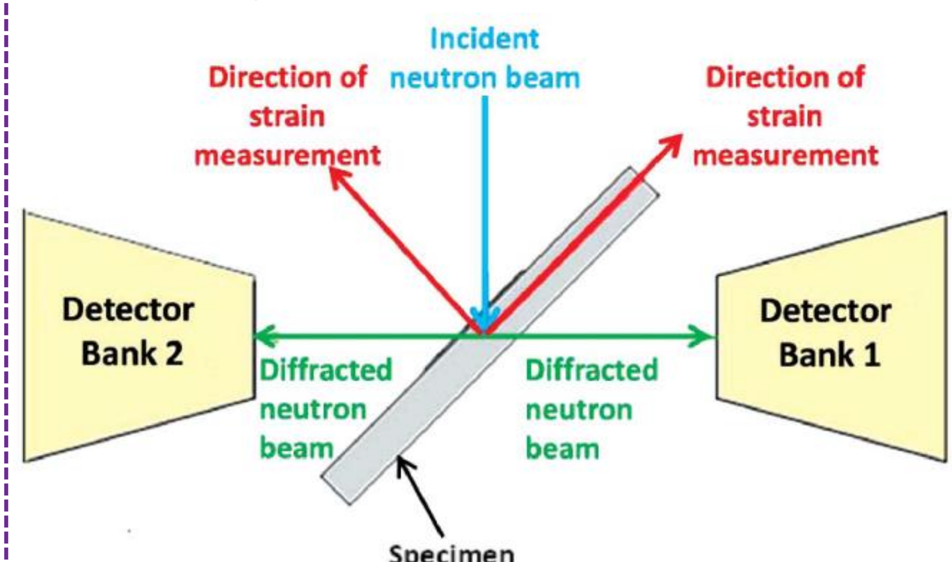
Monochromatic (CW)

- Fix wavelength and scan detector angle
- Multiple 2θ required to cover Q(d) spacing range
- Q(d) spacing limit 4π/λ (2π/d)
- Instrumental count rate factors: Source power, monochromator reflectivity, detector coverage and efficiency, etc

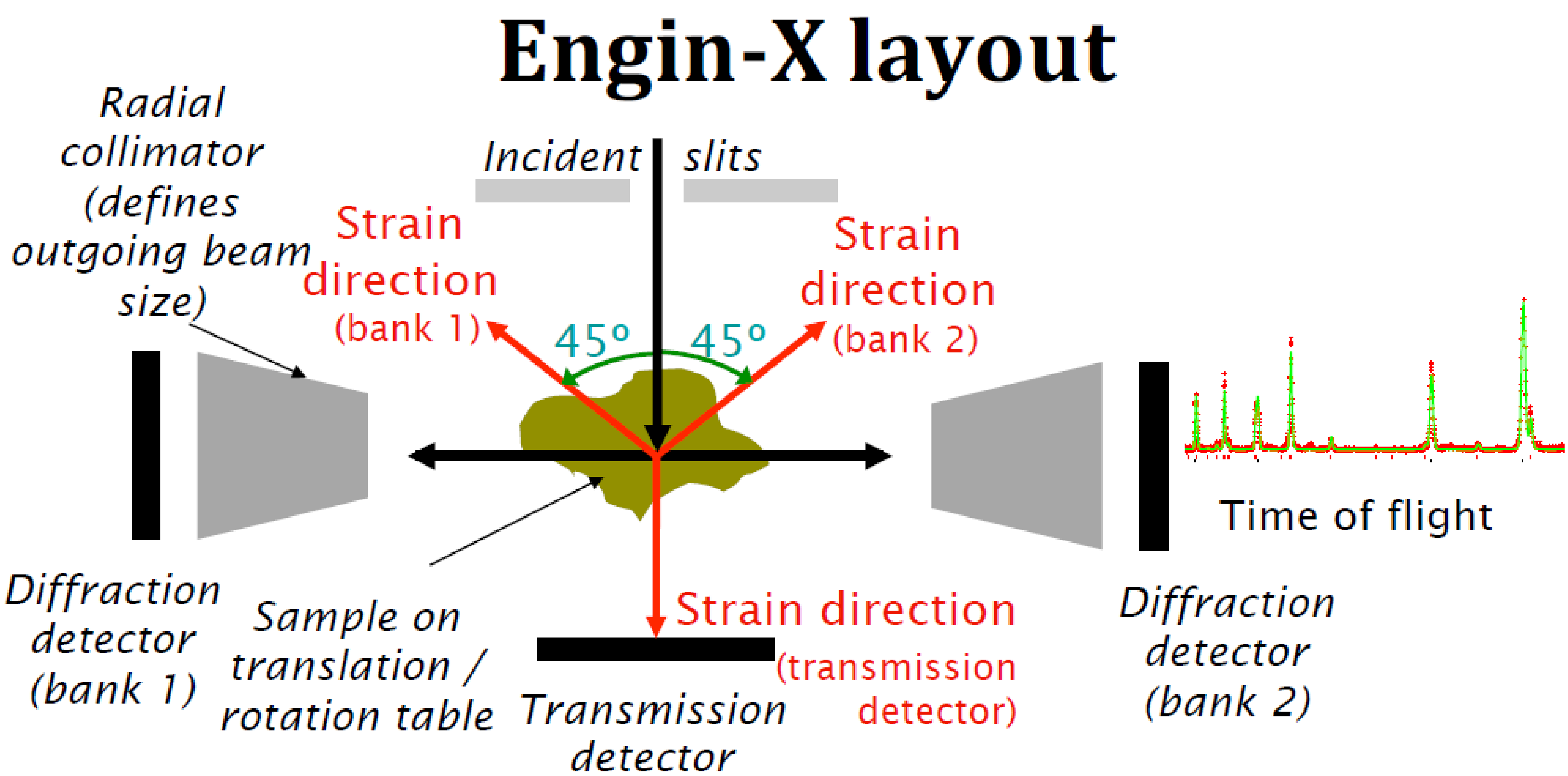


Time of Flight (ToF)

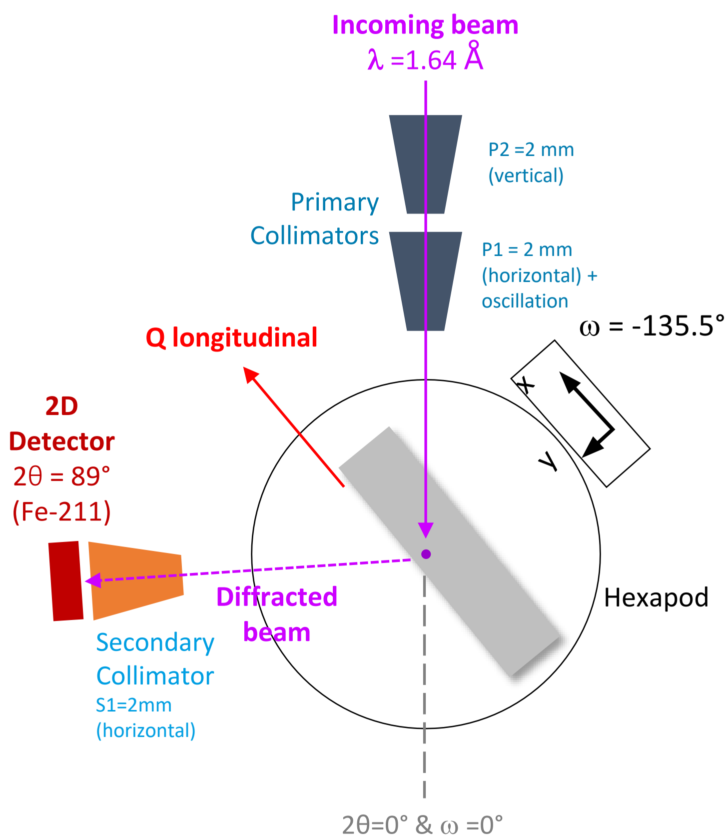
- Fix detector angle and scan wavelength
- Single 2θ covers range of Q(d) space
- Q(d range) determined by λmax, λmin and θ
- Instrumental count rate factors: Source power, moderator performance, beam transport efficiency, detector coverage and efficiency, etc

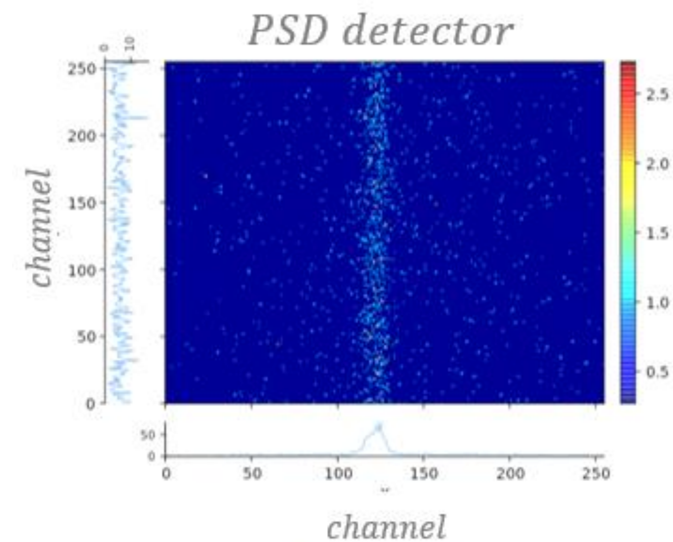


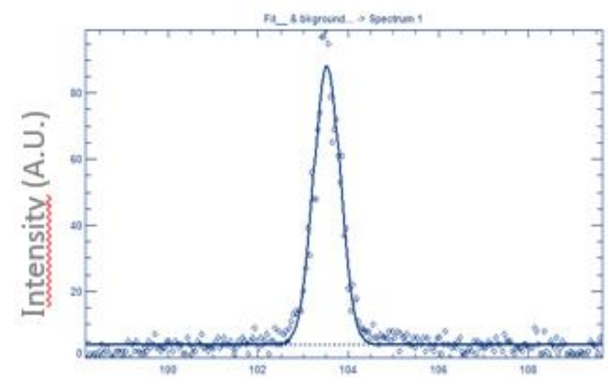
Set-up of Engineering Instrument: Spallation/ToF



Set-up of Engineering Instrument: Reactor-based







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Neutron Beam Instruments

https://nucleus.iaea.org/sites/accelerators/Pages/Interactive-Map-of-NB-Instruments.aspx

Total Instruments:
372

Total Countries:
26

Neutron Strain Scanners

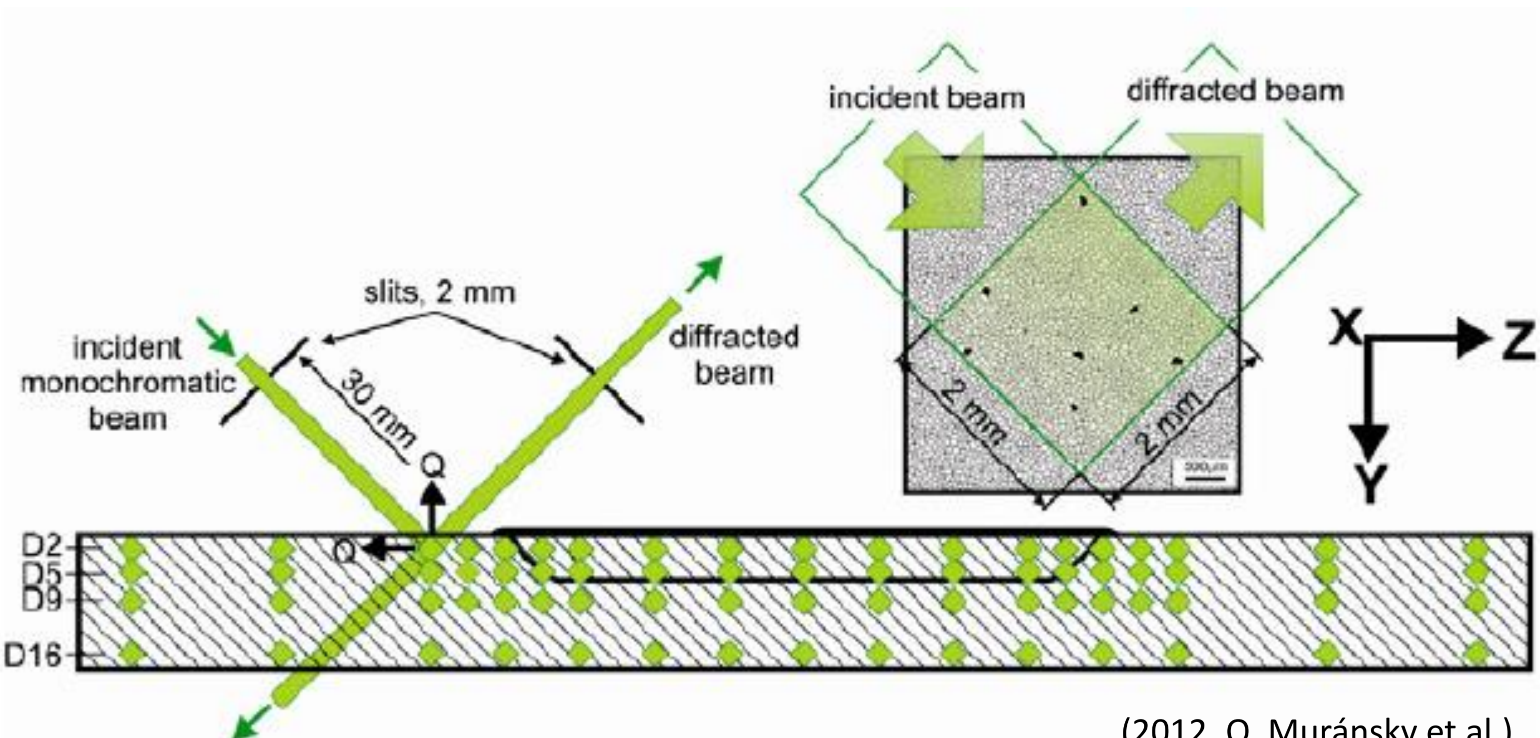
https://nucleus.iaea.org/sites/accelerators/Pages/Interactive-Map-of-NB-Instruments.aspx

> Neutron Scattering > Diffractometer > Strain Scanner

Facility Name	Instrument type	Instrument Name
China Advanced Rese..	Strain Scanner	END
China Mianyang Rese..	Strain Scanner	RSND
HANARO reactor	Strain Scanner	RSI
HFIR reactor	Strain Scanner	HB-2B NRSF2
IBR-2 M Pulsed React..	Strain Scanner	EPSILON-MDS FSD FSS
ILL Grenoble (HFR)	Strain scanner	SALSA
ISIS Spallation Source	Strain Scanner	ENGIN-X
J-PARC	Strain Scanner	BL19 - TAKUMI
JRR-3 reactor	Strain Scanner	T2-1 RESA-1
NBSR reactor	Strain Scanner	DARTS
OPAL reactor	Strain Scanner	KOWARI
PARR-1 reactor	Strain Scanner	2
Reactor IR-8	Strain Scanner	HC-3 STRESS
Research Reactor LV..	Strain scanner	HK4 SPN-100 HK9 TKSN-400
RSG-GAS Batan	Strain Scanner	DN1
SAFARI-1 Research R..	Strain Scanner	MPISI
SINQ Spallation Source	Strain Scanner	POLDI
Spallation Neutron S..	Strain Scanner	BL-7 VULCAN

The gauge-volume

ND set-up & gauge volume



(2012, O. Muránsky et al.)
doi:10.1016/j.ijsolstr.2011.07.006

The schematic drawing of the neutron diffraction geometry, the positions of measuring points on the D plane (along the weld centerline, see Fig. 2(a)), and the gauge volume depicting a random number of grains satisfying the diffraction condition (black grains

General Overview: Diffracting Gauge Volume

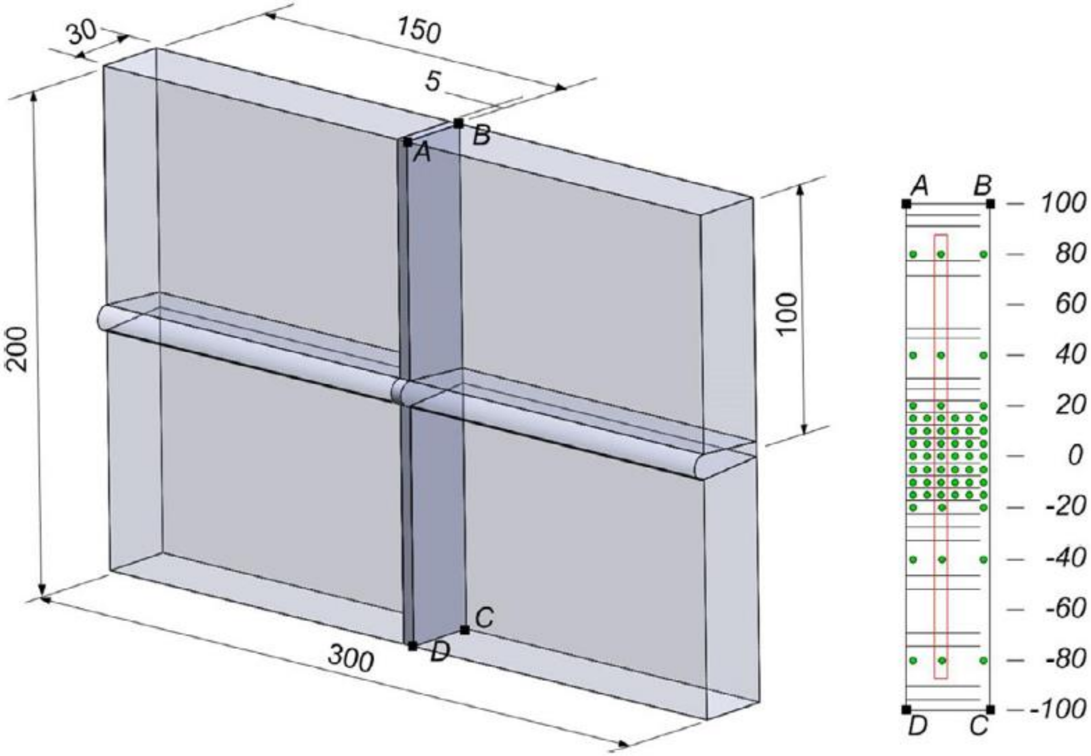
Volume element of the material in which the recorded scattering takes place

- Results in averaged d-spacing (powder diffraction - many grains)
- Defines the minimum spatial resolution of the method (around $1mm^3$ minimum gauge volume when using neutron diffraction)
- and type of residual stress resolved (macro-stress or type-I usually. Type-II for two phase materials).
- Use the largest possible gauge volume for your specific issue in order to minimise counting time

The d0 specimen

d0 samples

- **Comb**
- Pins
- Slice



d0 samples

- Comb
- **Pins**
- Slice

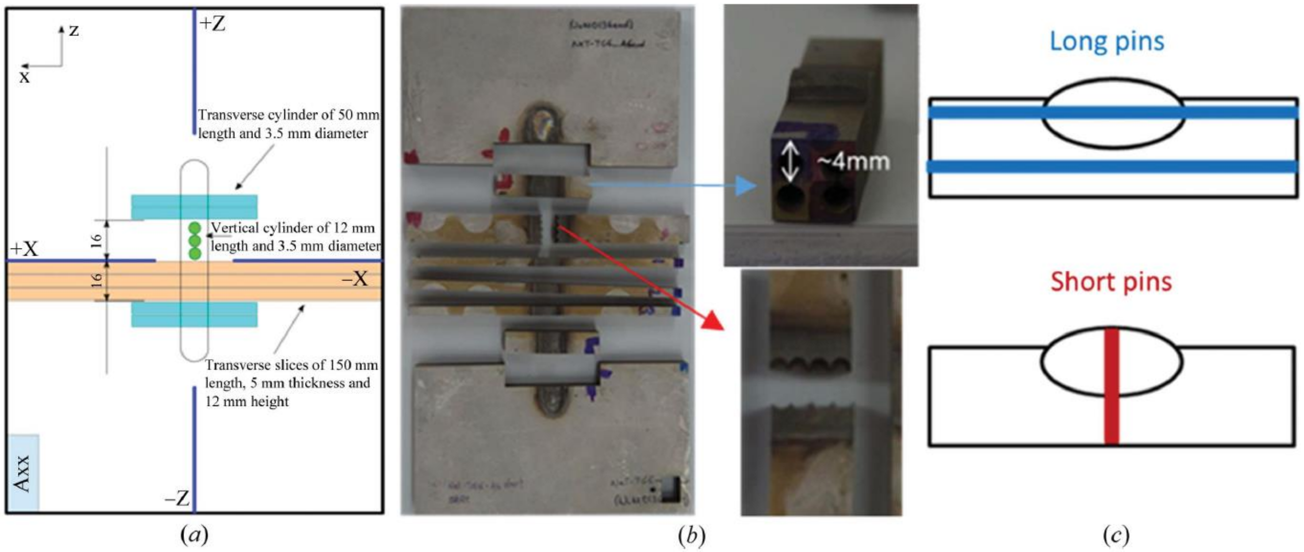
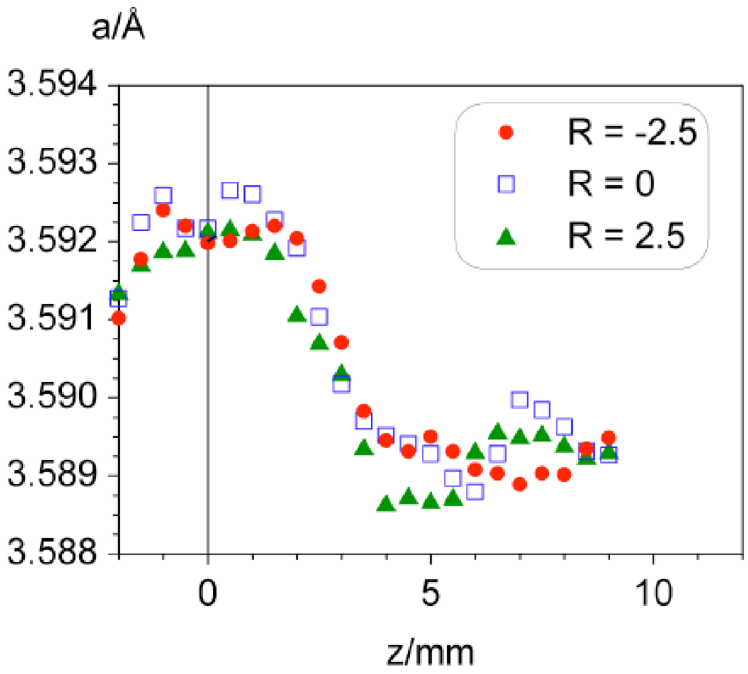


Figure 4
(*a*) The cutting plan for extraction of reference specimens from the A6, TG6 specimen (Ohms *et al.*, 2015). (*b*) The A6 specimen after extraction of the stress-free pins and slices. (*c*) The exact locations of the extracted pins used as stress-free reference samples.

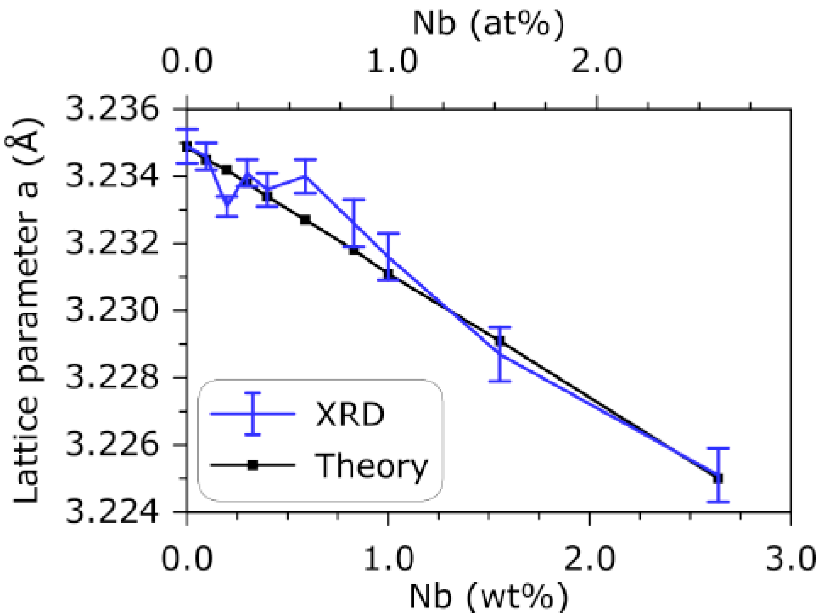
d0 variation

Accurate strain analysis relies on accurate determination of d_0

$$\varepsilon = \frac{a - a_0}{a_0} = \frac{d - d_0}{d_0}$$



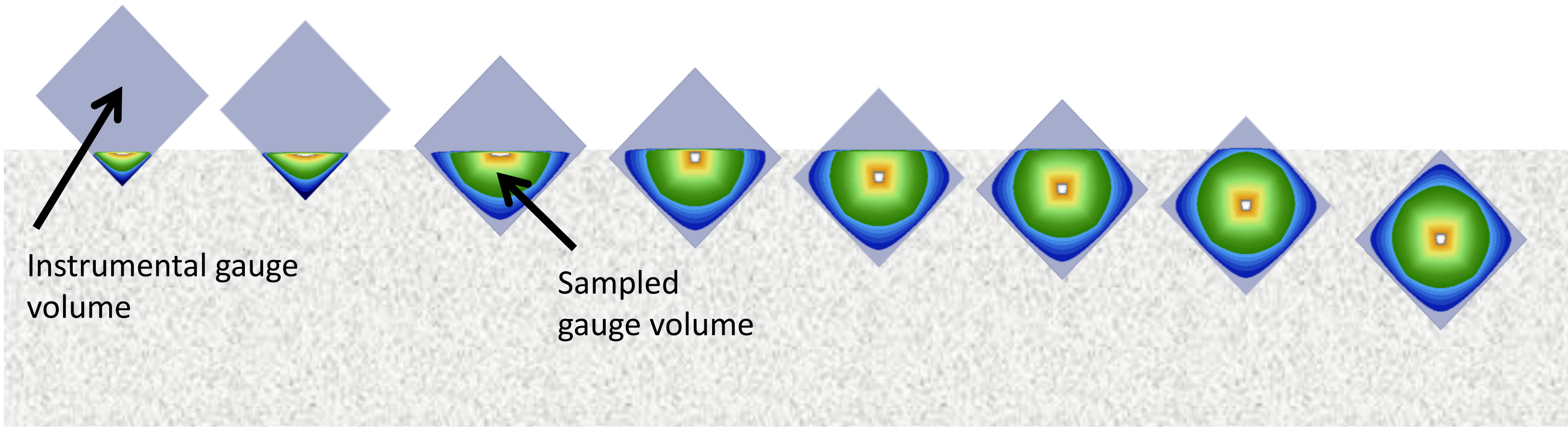
Example of d_0 variation across a tubular Nickel weld



The Vegard Law
Example: Nb in Zr

Near surface

Near surface measurements



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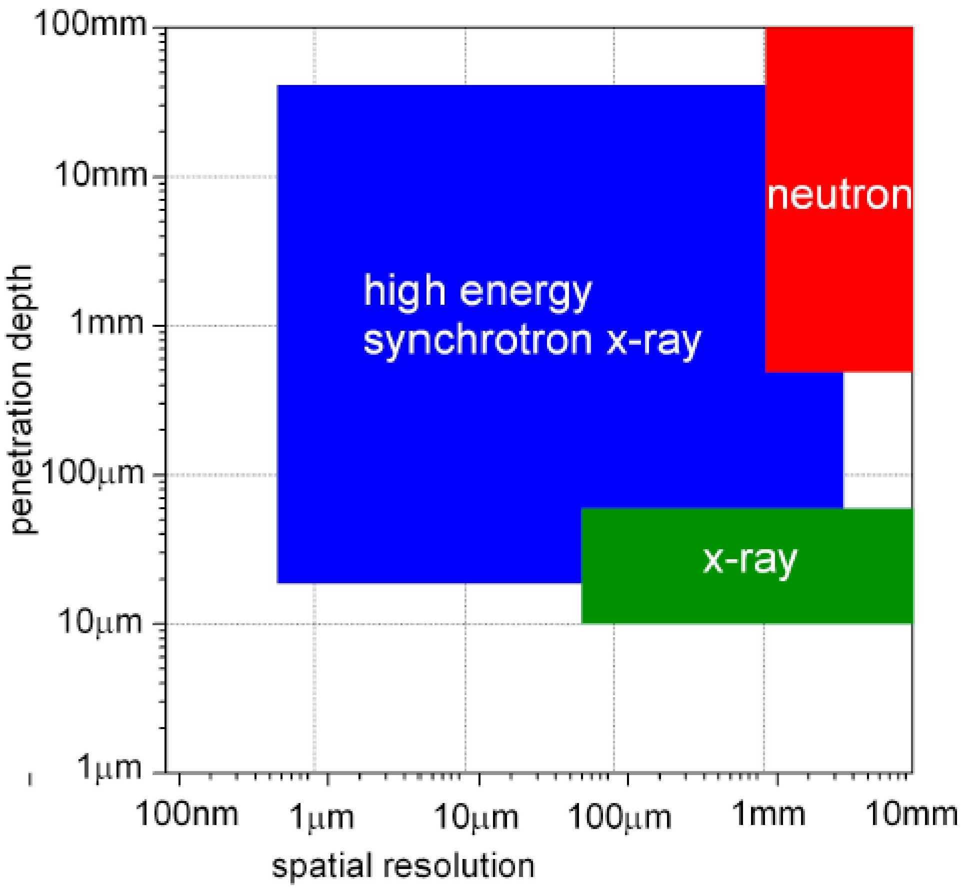
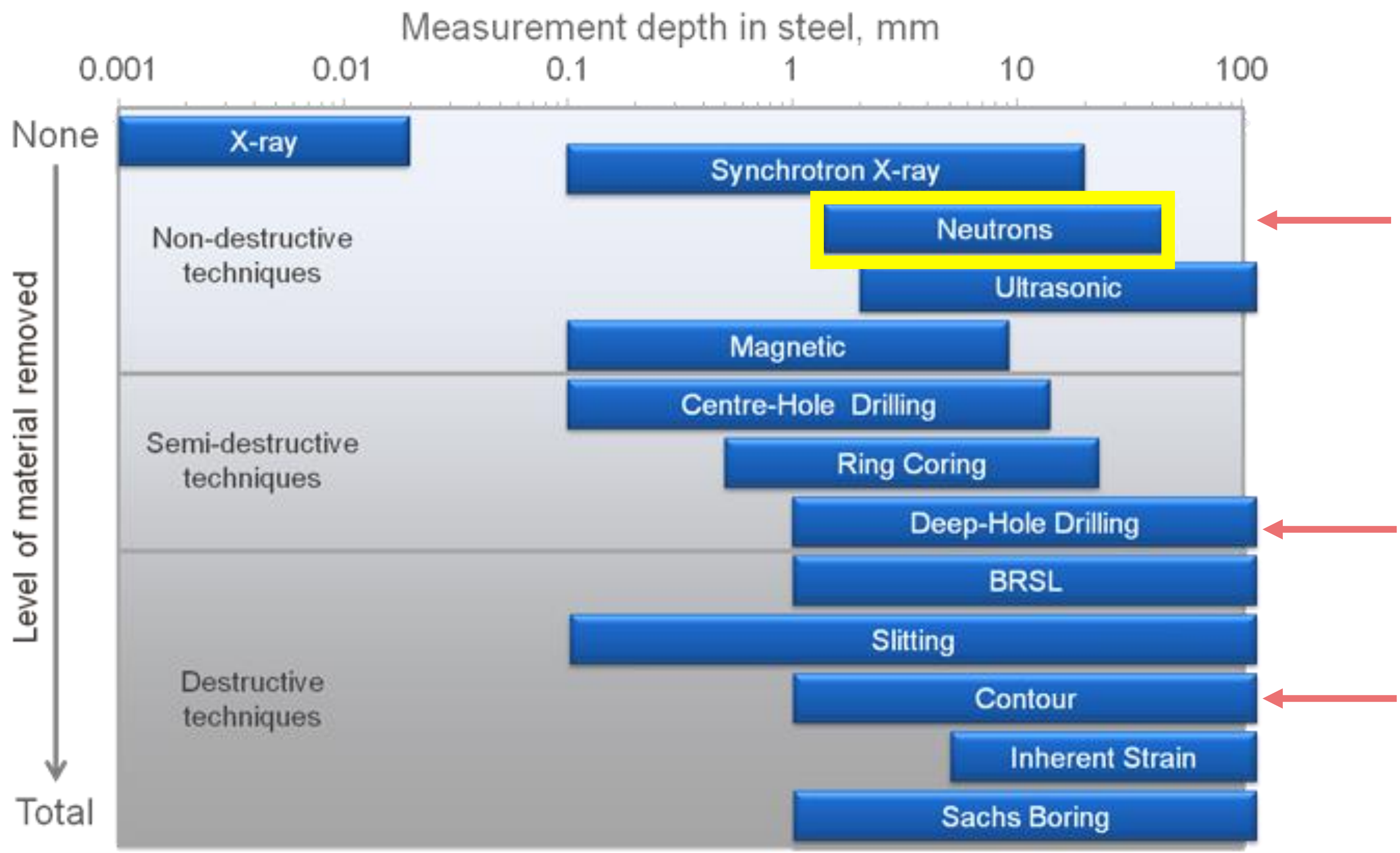
Neutrons vs Other measurement techniques

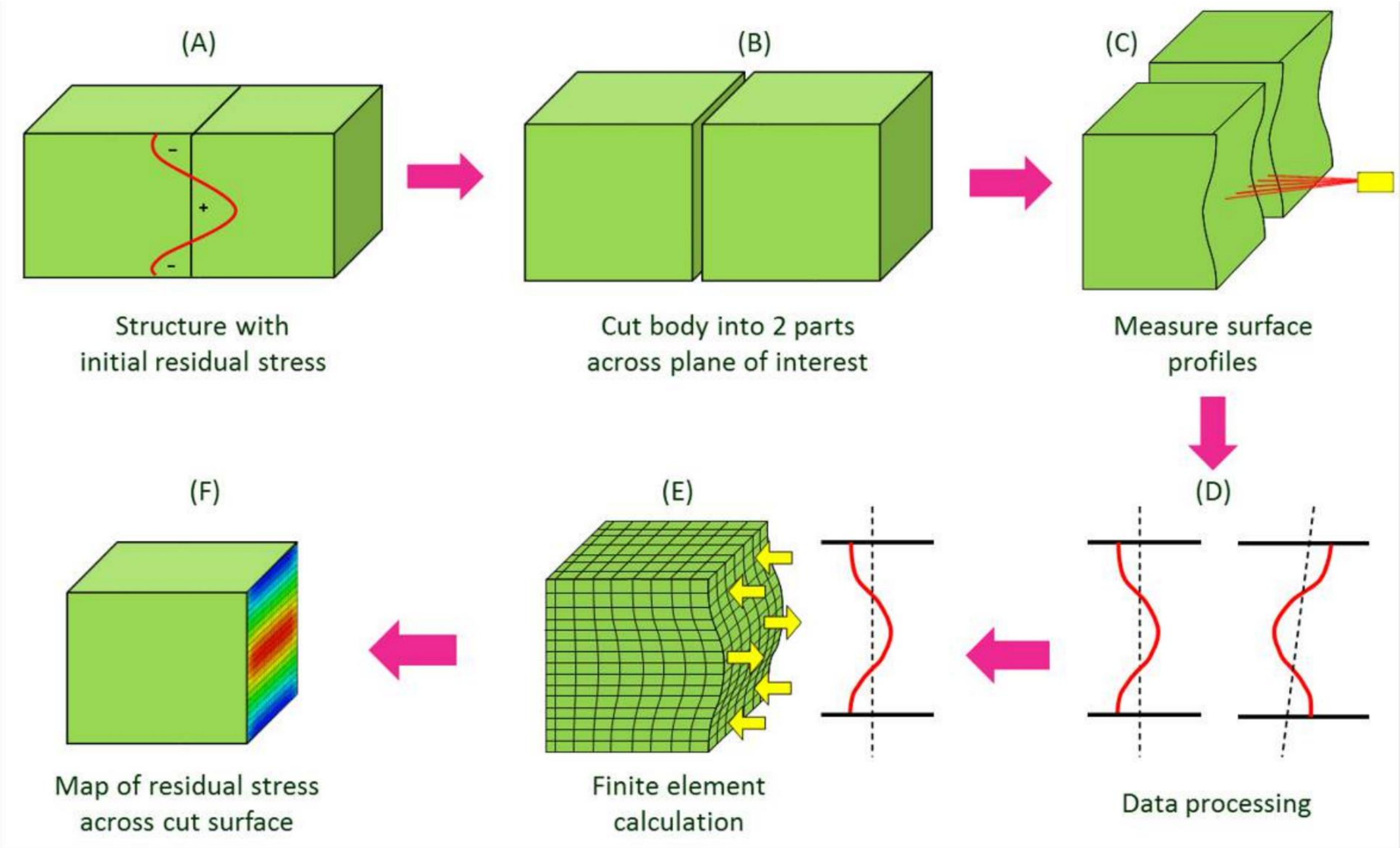
ND vs other diffraction methods



ND vs Other Residual stress measurement techniques



The Contour Method

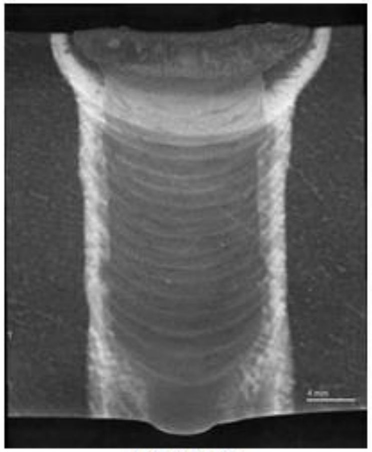


Case Studies

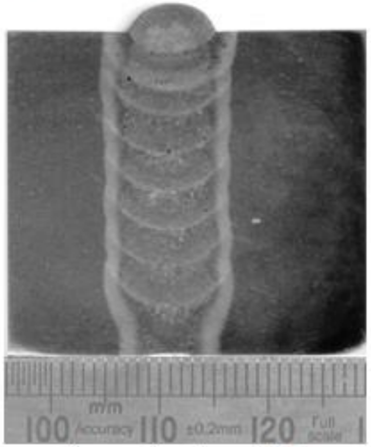
Challenges, considerations and implications

Case study: Cross process comparison – NNUMAN welds

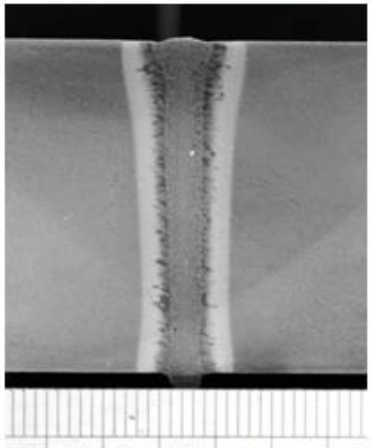
NG-SAW
(18 passes, 2 passes per layer)



NG-GTAW
(25 passes, one weaved pass per layer)



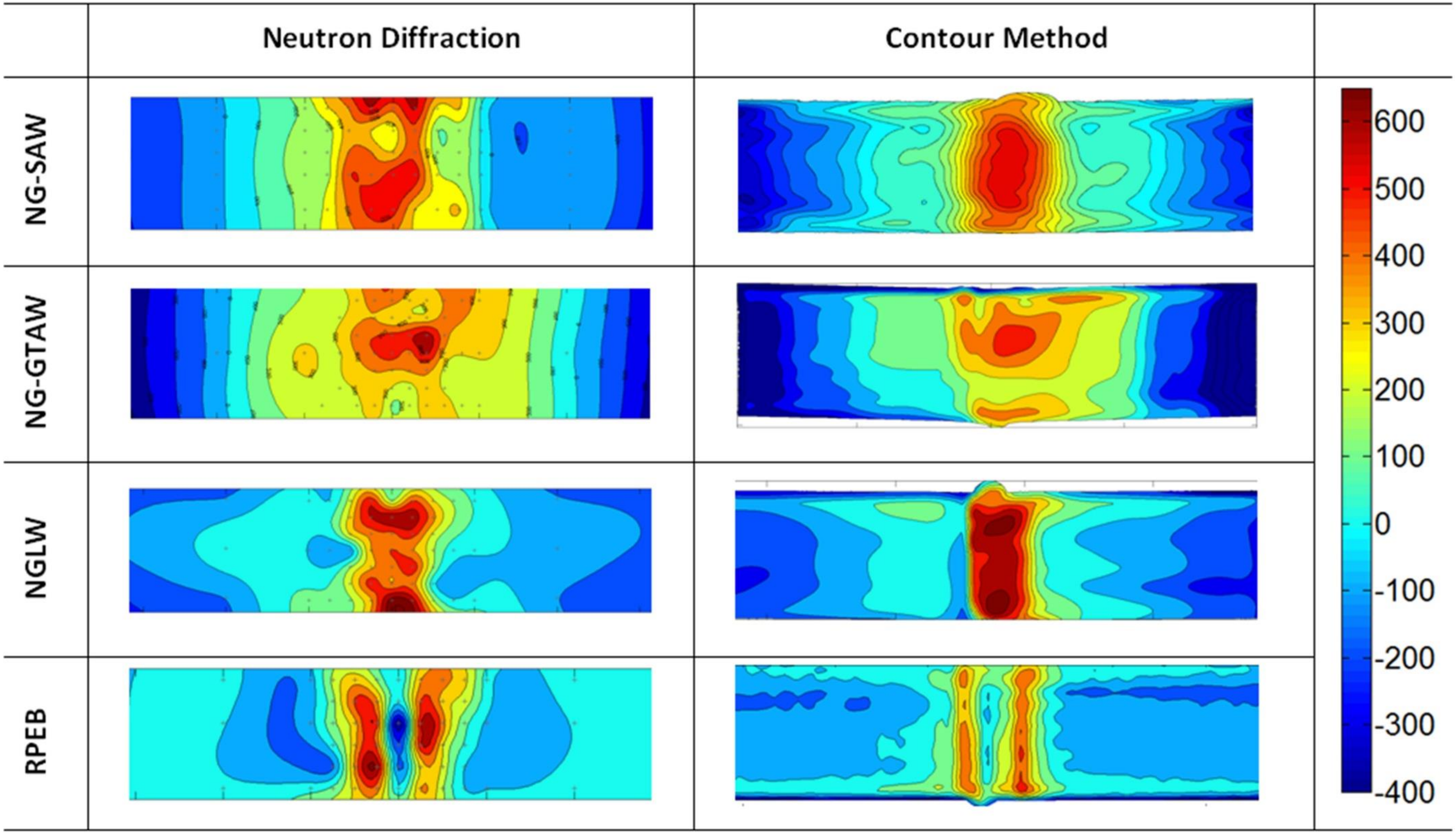
Narrow-Gap Laser Weld
(autogenous root pass, 8 filling passes)



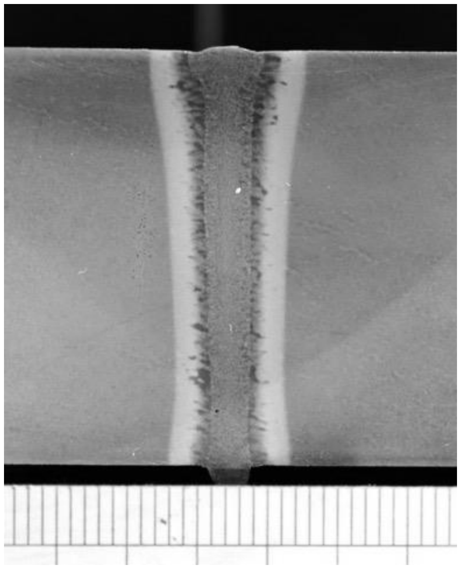
RPEB weld
(single autogenous pass)

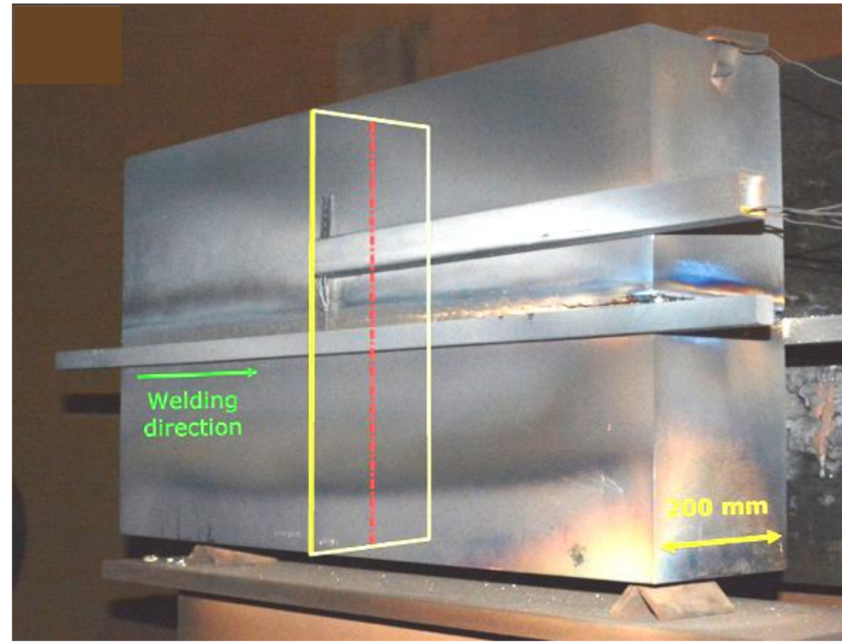
(2018, J. Balakrishnan et al.)
doi:10.1016/j.ijpvp.2018.03.004

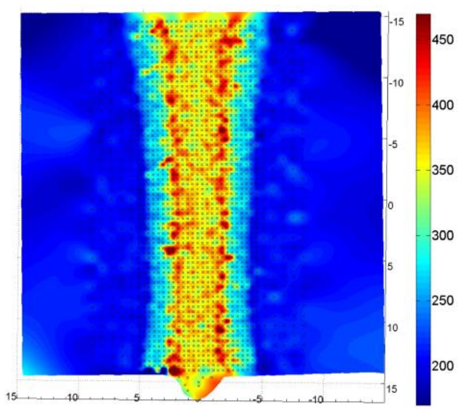
ND *vs* CM

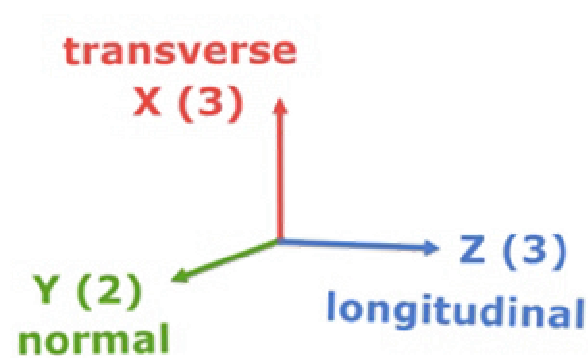

Neutron Diffraction
Contour Method
NG-SAW
NG-GTAW
NGLW
RPEB
600
500
400
300
200
100
0
-100
-200
-300
-400

Case study: EB welds





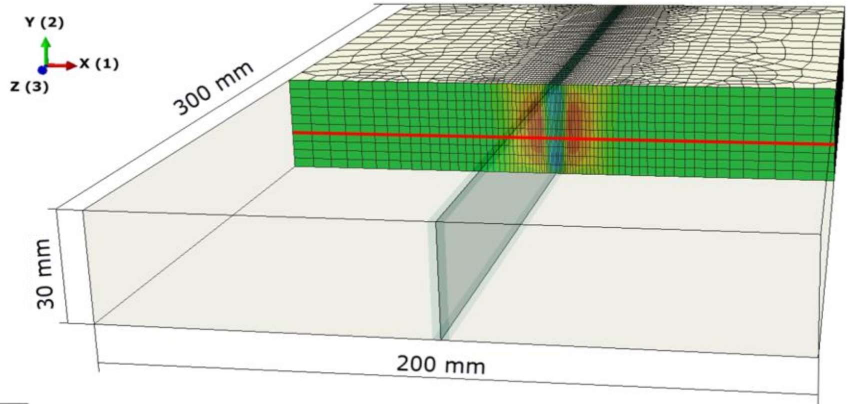


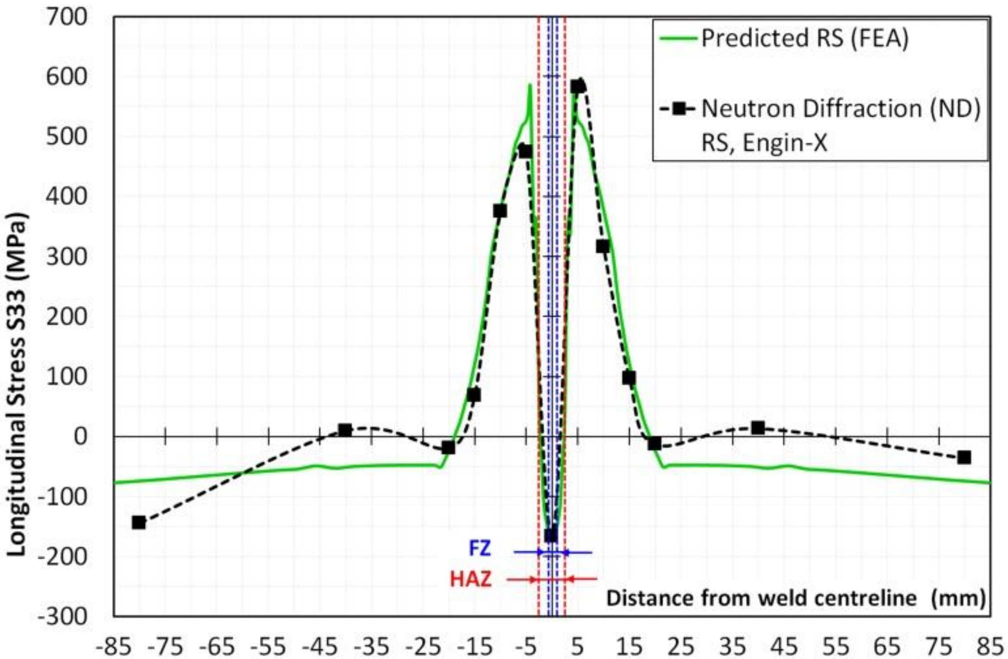


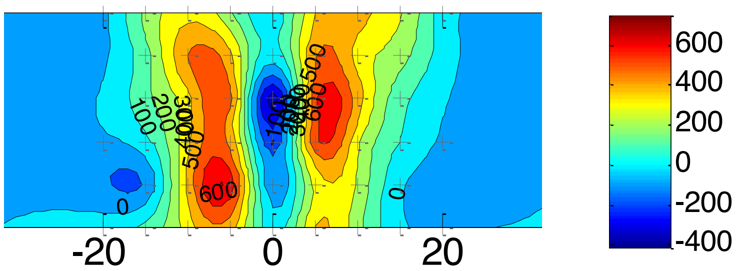
https://doi.org/10.1016/j.matdes.2021.109924
https://doi.org/10.1016/j.ijpvp.2019.03.035
https://doi.org/10.1016/j.nucengdes.2017.03.040

Case study: EB welds

- Challenge: short characteristic length of residual stress distribution

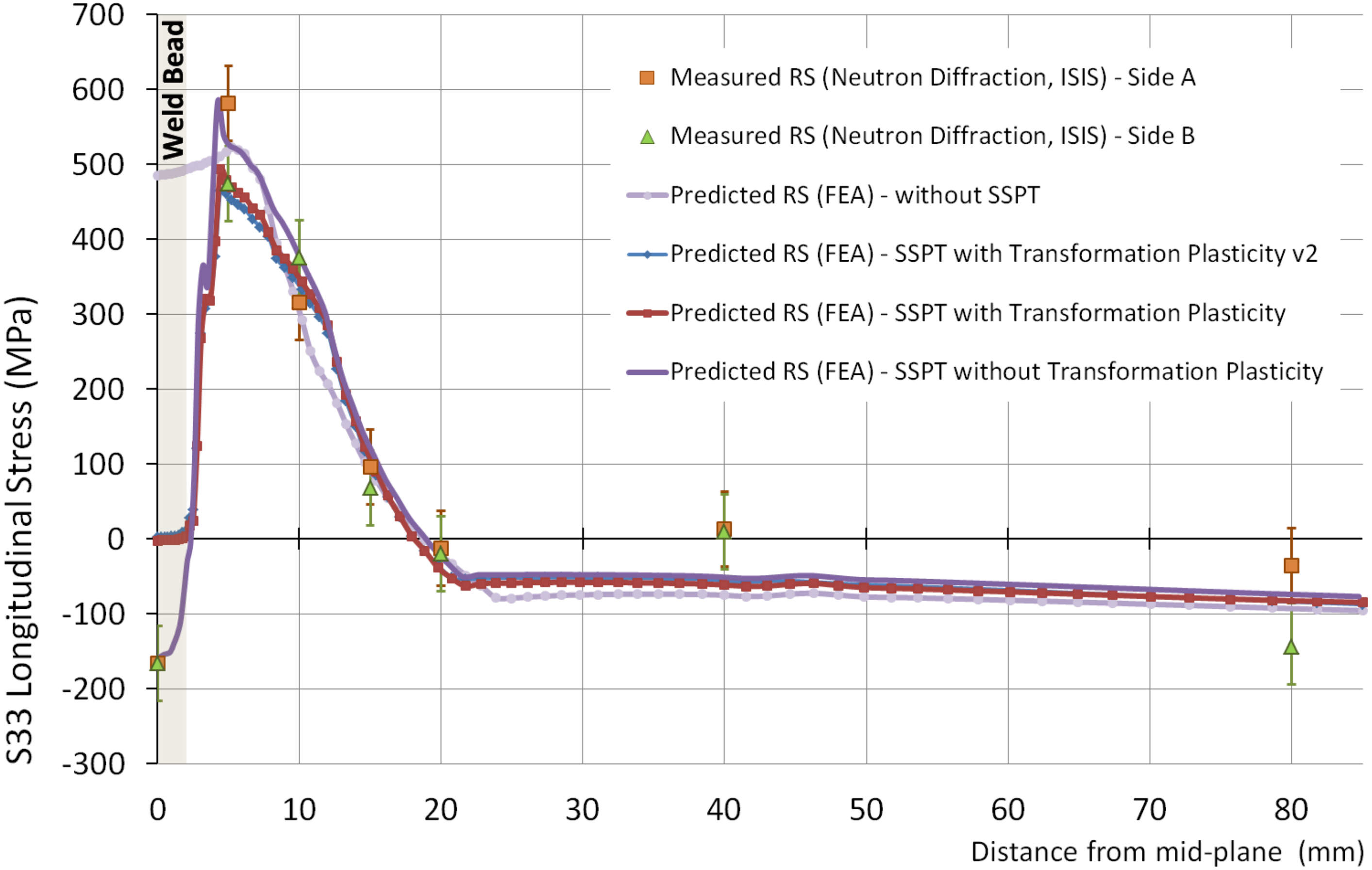






Case study: EB welds

- ND vs FE weld modelling predictions



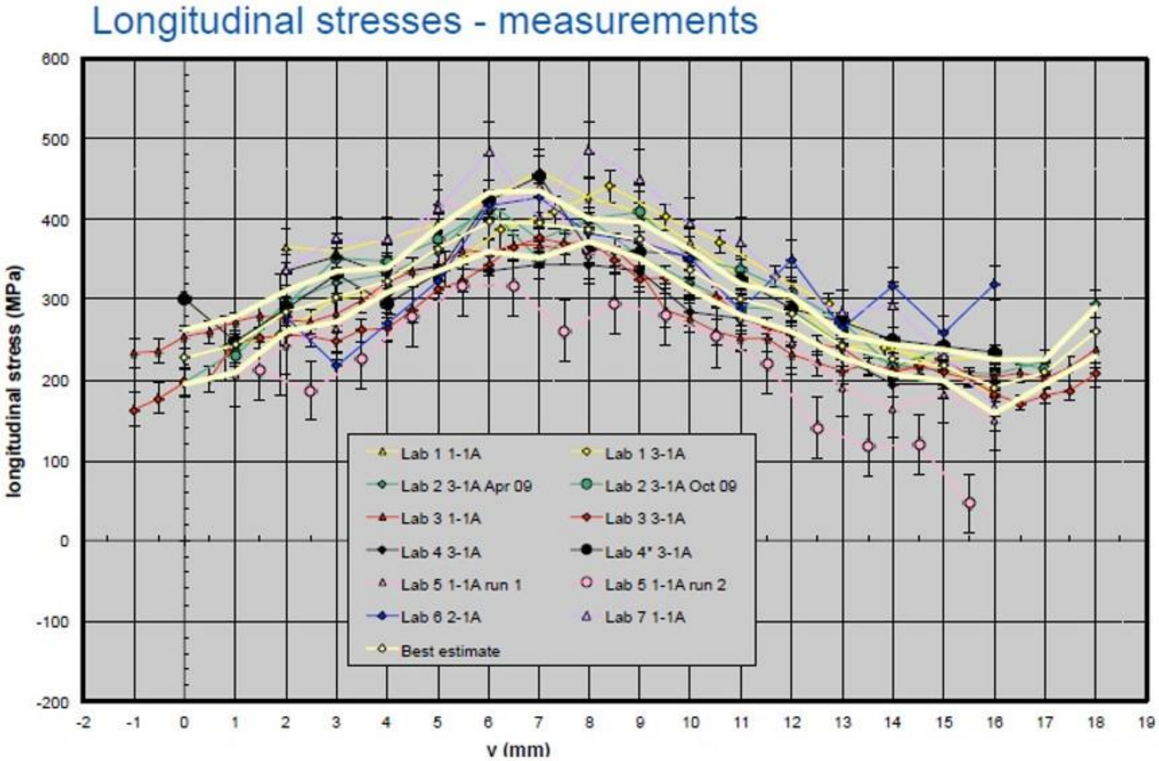
Case study: the NeT network

Network on Neutron Techniques Standarization for Structural Integrity

- Mission of the Network: to develop experimental and numerical techniques and standards for the reliable characterization of residual stresses in structural welds.
- https://www.net-network.eu
- ISO Draft International Standard 21432:2018

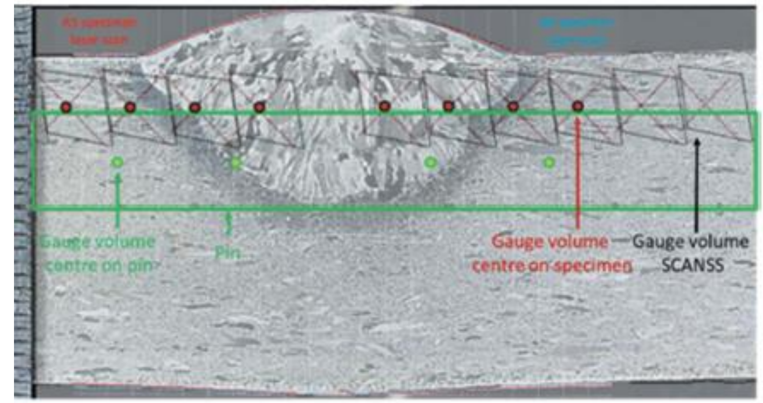
Case study: NeT-TG4

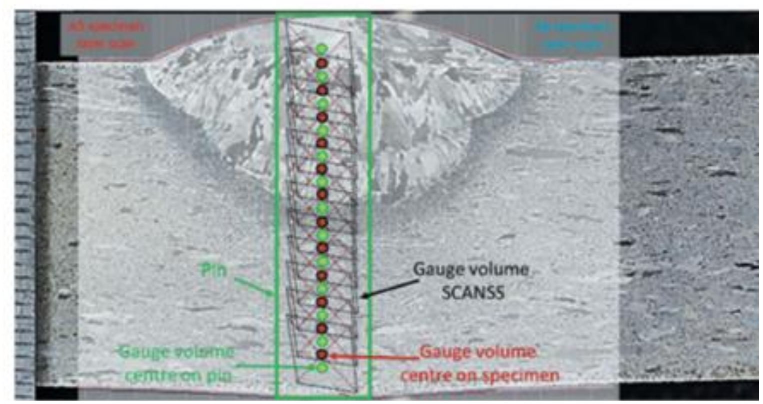
- Repeatability and standardization

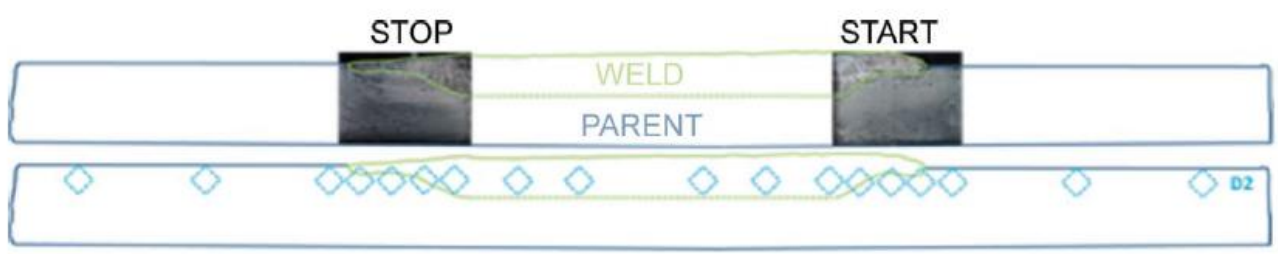


Case study: NeT-TG6

- Dissimilar metals
  - Alloy 600 parent plate
  - Alloy 82 filler wire
- Implication
  - **Interfaces**: how to properly process the data? *[Considering that the gauge volume includes both]*
  - **Misalignments** get critical



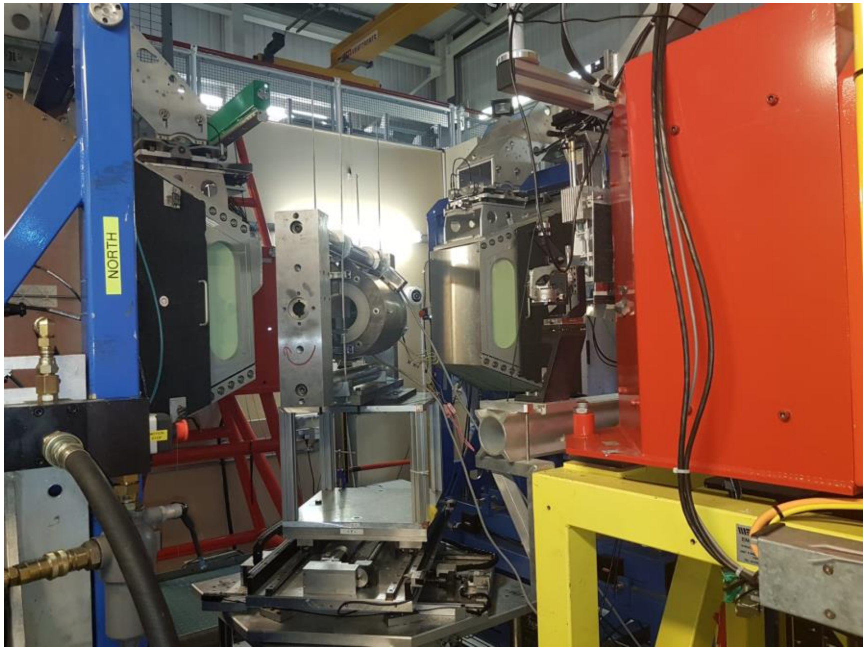




(2020, V. Akrivos et al.)
doi:10.1107/S1600576720009140

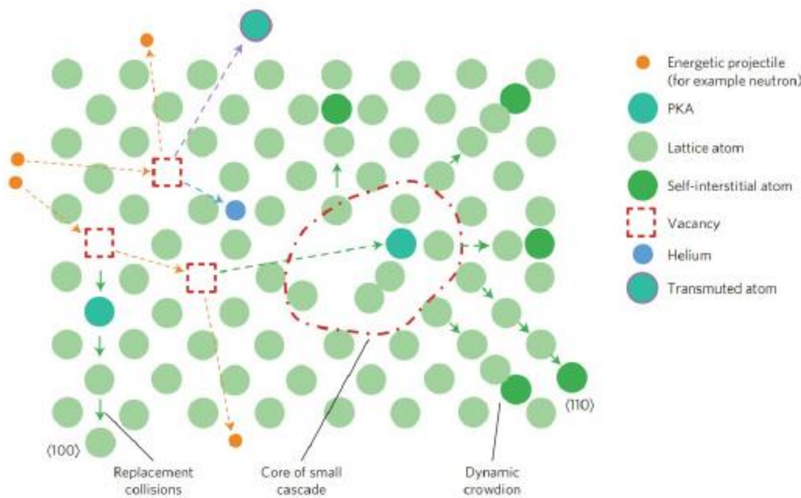
Case study: in-situ PWHT

- In-situ post-weld heat treatment
- Considerations: count time vs evolution of phenomenon



Case study: Irradiation damage

- Lattice defects introduced by irradiation
  - dislocations
  - crystallographic changes
  - changes to secondary phases in the material (amorphisation, dissolution, change in type of particles)



- All these changes can be assessed by diffraction techniques!

Concluding remarks

Neutrons:

- Non-destructive, full stress analysis
- Good penetration depth due to neutrality
- Big bulky sample with low stress gradients
- Variety of materials can be measured (Steels, aluminium, nickel, copper zinc or related)

Not-so good (handle with care): near surface or thin materials, texture (titanium, boron cadmium), high-spatial resolution/steep gradients, high instrumental resolution

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