

Engineering

Advanced diffraction
techniques for Residual
Stresses determination

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10th Oxford School on Neutron Scattering

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Introduction

- Residual stresses in materials
- Principles of measuring residual stresses by diffraction
- Neutron and Synchrotron X-ray diffraction
 - Properties
 - Facilities
- Case Studies / Questions
- Conclusions

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What are residual stresses

Deformation mismatch

Example: Welding



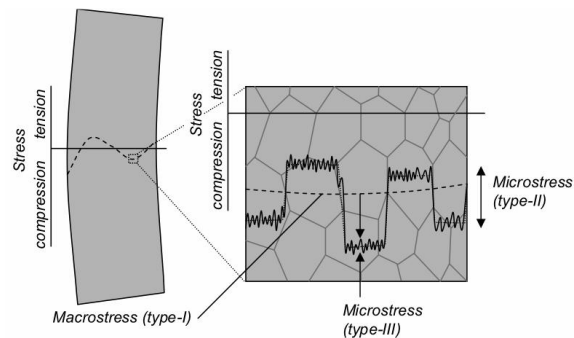
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Residual Stresses

- Internal stresses
- Caused by misfit
 - Type I
 - Type II
 - Type III

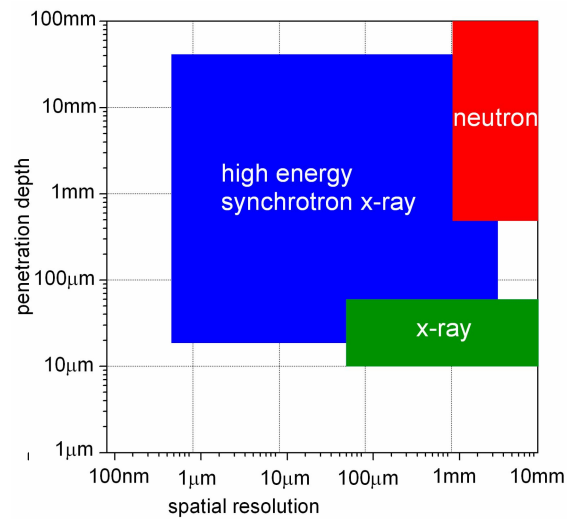
Bent bar:



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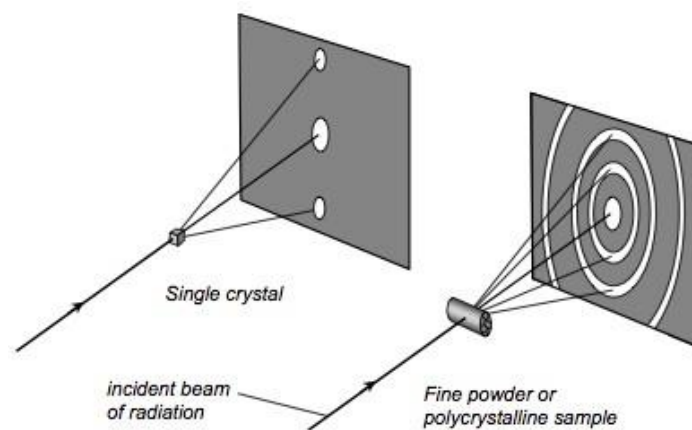
General Overview: Diffraction methods available



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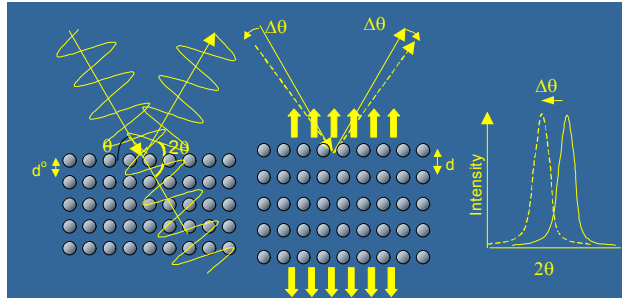
General Overview: Basic Principles: diffraction



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General Overview: Basic Principles: diffraction



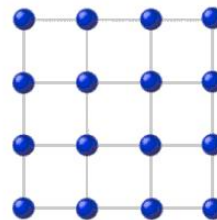
$$\lambda = 2d \sin \theta$$

- Diffraction measures elastic lattice strain as peak shifts
- Uses the poly-crystalline lattice planes as internal strain gauges

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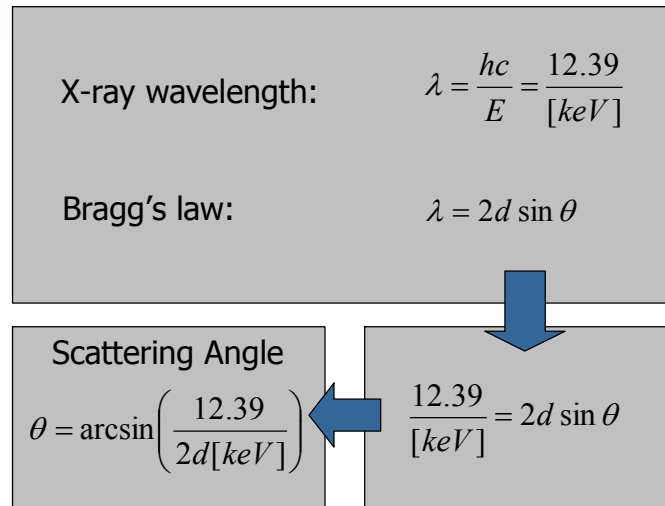
Basic Principle



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Bragg scattering angle



General Overview: Basic Principles

- Measured strains have to be converted into stresses! (Hooke's law) $\varepsilon = \frac{a - a_0}{a_0} = \frac{d - d_0}{d_0}$

- Often requires the unstrained lattice parameter a_0

e.g. isotropic triaxial
along principal
directions:

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

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

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

(Attention: not always this simple!)

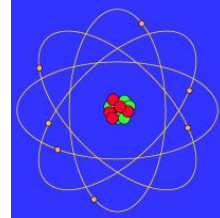
Why do we like neutrons ?

- Part of the nucleus
- Same mass as protons
- Interesting wavelength/mass relationship:

$$\lambda = \frac{h}{p} = \frac{h}{mv}$$

← Planck

← Mass * Velocity
- “Thermal” neutrons: wavelength similar to those of X-rays 0.5-5Å similar to atomic spacing in solids
- Allows cubic gauge volumes!
- Relatively divergent beam !!



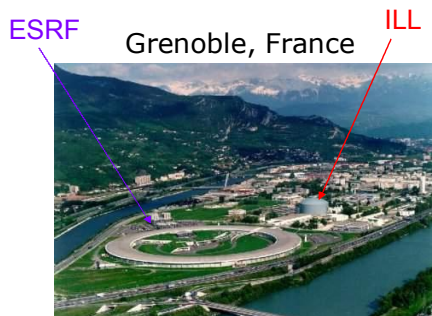
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Neutron and Synchrotron Sources

- Neutron:**
- Reactor Sources (Fission)
 - Constant wavelength/Single Peak
 - Accelerator Sources (Spallation)
 - Time-of-flight / Full Spectra / Rietveld

- Synchrotron:**
- Monochromatic λ and white beam



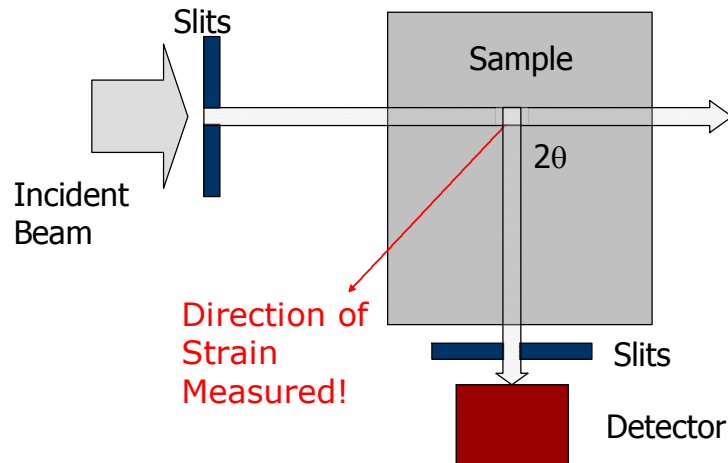
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General Overview: Strain Scanning

Diffracting Gauge Volume: The volume element defined by the incident slits and diffraction slits

Neutron Diffraction

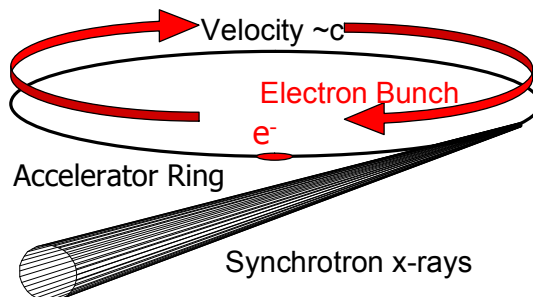


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In comparison: Synchrotrons

- Synchrotron X-rays are energy X-rays (10-300keV) produced using “synchrotron” accelerators
- Main difference is the wavelength/energy
- Penetration depends on wavelength
- Very high intensity:
 - X-ray tube $\sim 10E8$
 - Synchrotron $\sim 10E15$

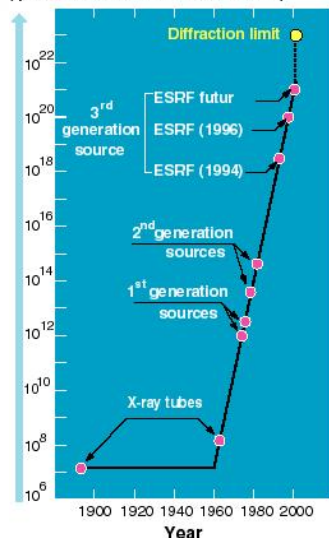


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In comparison: Synchrotrons

Brilliance of the X-ray beams
(photons / s / mm² / mrad² / 0.1% BW)



- Synchrotron sources provide very intense (million times more flux than a lab source) high energy beams
- beam is highly parallel (10¹² times more brilliance than a lab source)
- at energies of 40-80keV penetrations of many mm possible
- small micron sized beams

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How does a Synchrotron work ?

- X-rays produced by sending high energy (9GeV) electrons round a ring
 - radial acceleration causes emission of electromagnetic radiation
 - low energy radiation
 - much greater radiation if you insert devices which bend beam sharply
- X-ray beam produced by bending magnets, undulators and wigglers

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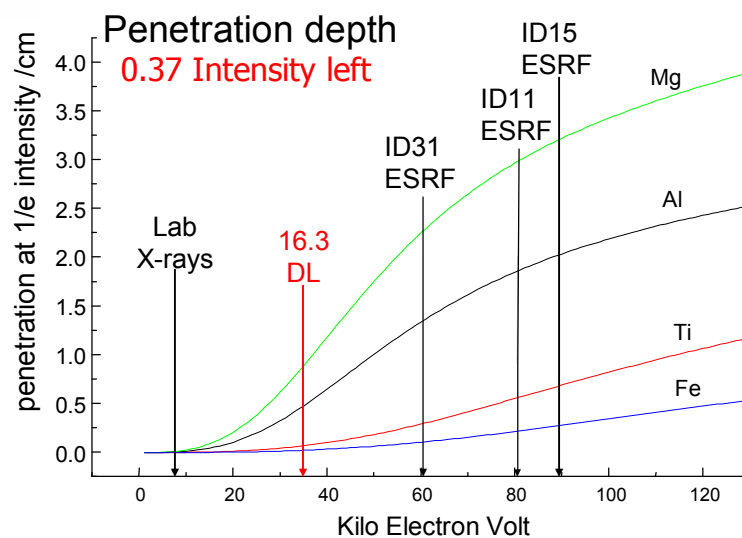
How does a Synchrotron work ?

- Bending magnets create a wide spectrum of X-ray radiation
- Wigglers are more intense because bend beam many times
- Undulators bend the beam such that radiation interferes to create very high fluxes of certain wavelengths (determined by spacing and number of magnets)
- Highly parallel beam is produced

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Synchrotron Diffraction: Penetration depth (monochromatic beam)



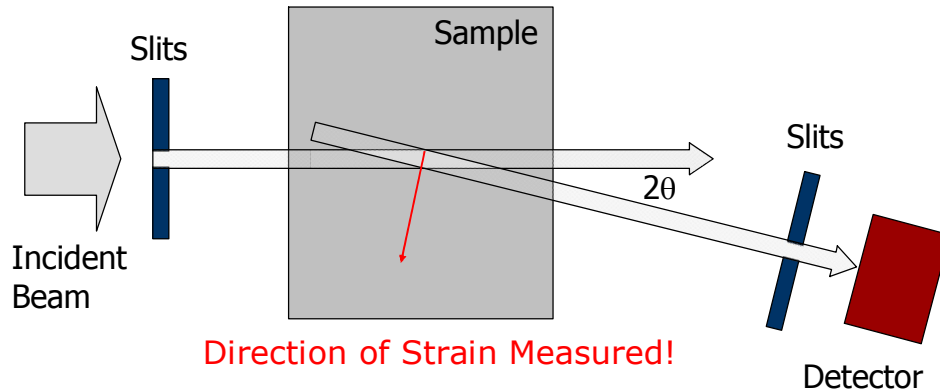
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General Overview: Strain Scanning

Diffracting Gauge Volume: The volume element defined by the incident slits and diffraction slits

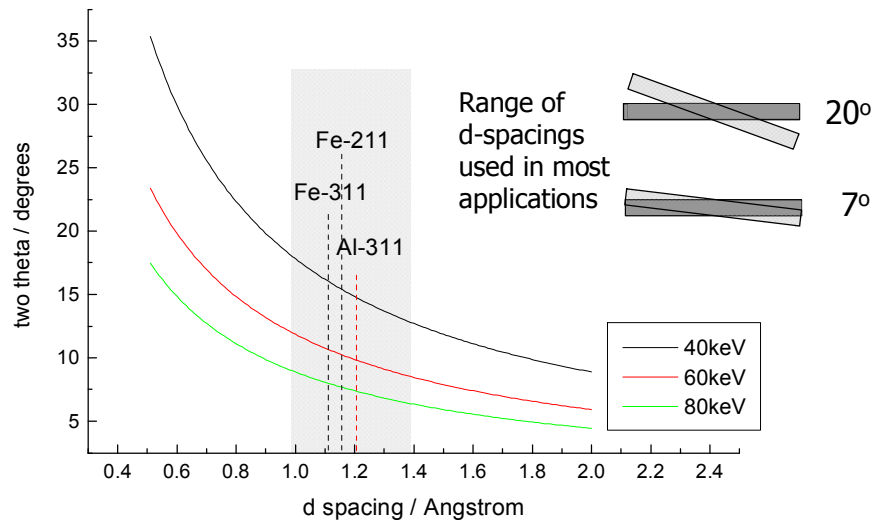
High Energy Synchrotron X-ray Diffraction



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Synchrotrons: Scattering Angle 2θ



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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

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Near surface measurements

Neither peak shift (strain) nor measurement location is correct near a surface!



- Partial filling of sampling gauge gives a peak shift - need to correct peak shift
- Translator records centre of gauge which is rarely the centre of gravity of diffracting region
 - need to correct gauge position

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Neutron Properties

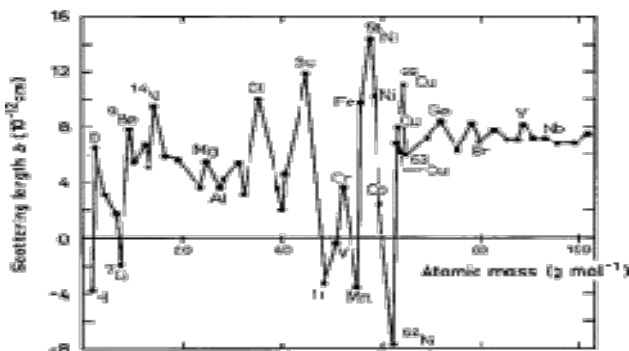
- Neutrons are scattered by atomic nuclei (electrons and X-rays which are scattered by the electron cloud).
- Since the scattering is nuclear process, scattering amplitude varies greatly for different isotopes of same element and in a unpredictable manner from element to element. X-ray and electron scattering increase monotonically with atomic number

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Neutron Properties

- Random Scattering length
- Penetration depth independent of energy/wavelength
- Electrically neutral
- Great penetration
- Low flux/intensity

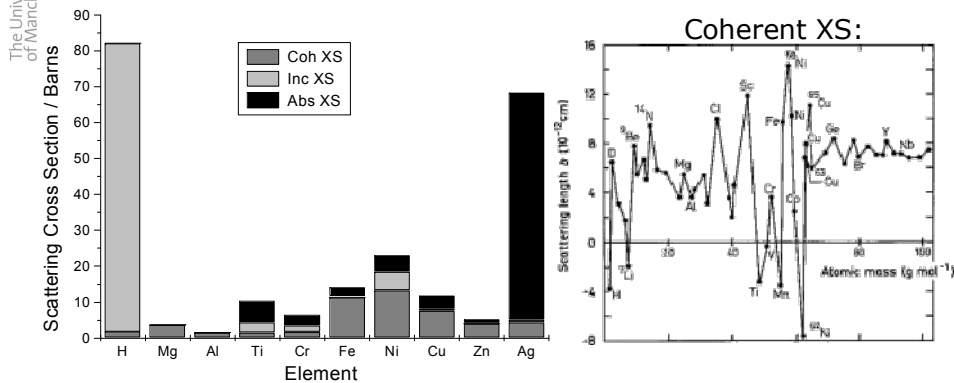


Economic Depth	Al	Steel	Cu	Ti	Ni	SiC
mm	250	37	40	27	24	200

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Neutron Scattering



$$I_{\text{trans}}(t, \lambda) = I_{\text{inc}}(\lambda) e^{-\Sigma^* t}$$

Coherent XS ~ Signal
Incoherent XS ~ Background
Absorption XS ~ 1/Intensity

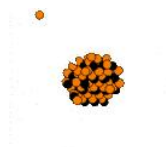
Penetration depth ~
1/Sum of Scatt XS

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Research Reactors

- Fission in Reactor Core
 - Moderated neutrons
 - Monochromators in guide
- **"Constant Wavelength"**
- Many Facilities in Europe:
 - **ILL**, Saclay (Fr), FRM-2 (G), Petten (NL),
 - ...
 - Generally low flux except ILL (and FRM-2)



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Reactor Sources: Measurement of Strain

- Intensity as Function of diffraction angle $\Theta/2\Theta$

$$\lambda = 2d \sin \theta$$

fixed



Strain: $\varepsilon = -\cot(\theta_0) (\theta - \theta_0)$

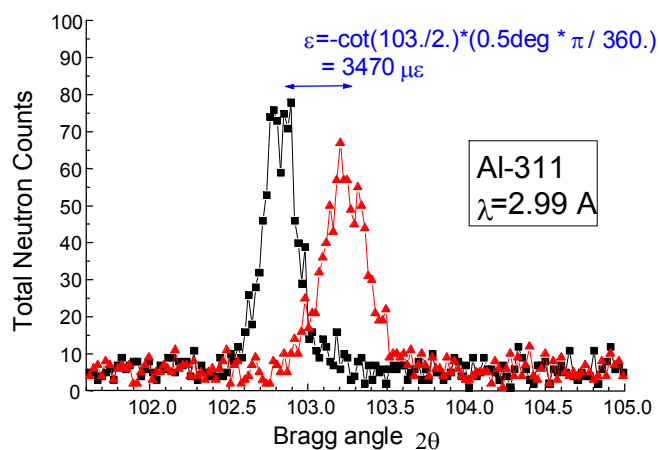
↑
In units of *rad*

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Single Wavelength at Reactor

Single-wavelength instrument: D1A at the ILL
New instrument at ILL: SALSA

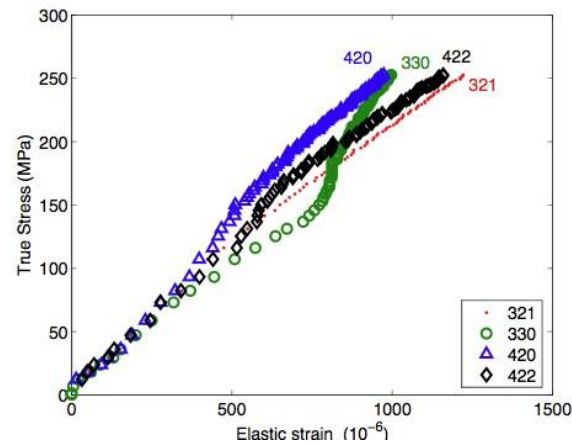


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Which peak gives us the pure macrostress response ?

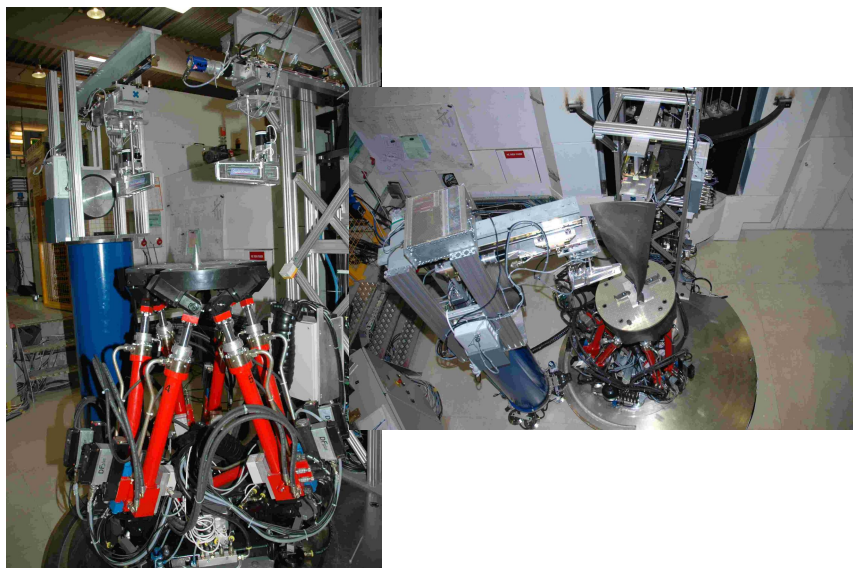
In-situ Loading on a neutron diffraction beam line



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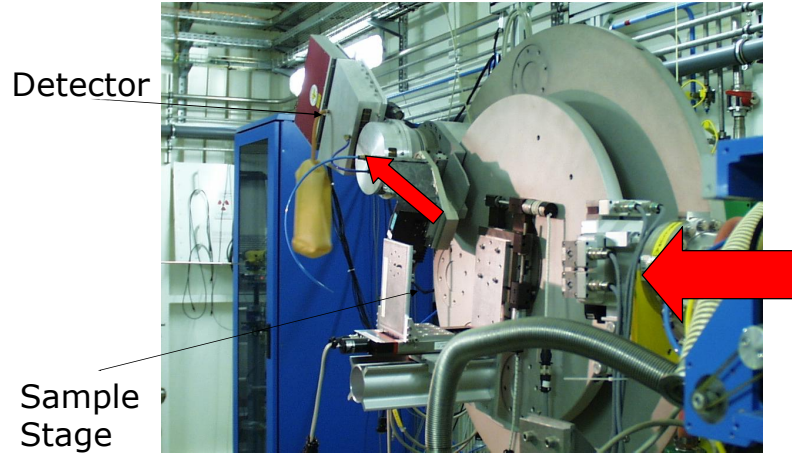
SALSA, ILL



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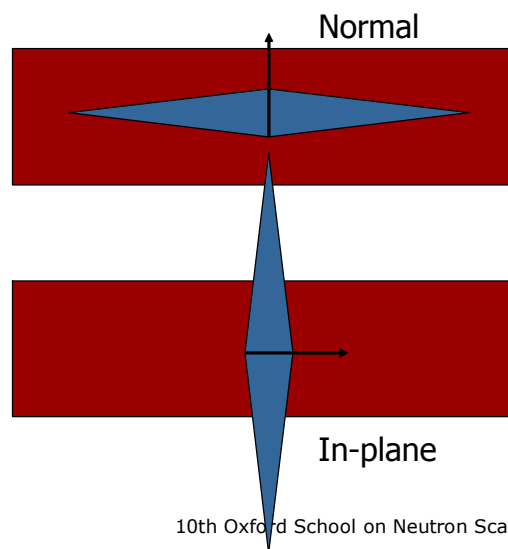
Typical Diffractometer at Synchrotron (here ID31 at the ESRF)



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Diamond gauge volume



Two orthogonal
strain components
sample different
material volume

**Analysers crystal
for partly filled
Gauge volume
necessary!**

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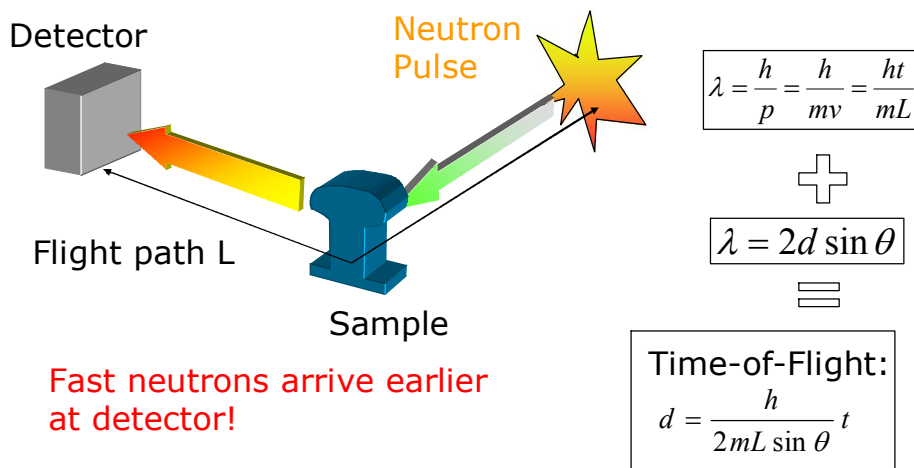
Time of flight method

- Sharp pulse leaves source
- High energy neutrons (short λ) travel faster and arrive first, low energy (long λ) last

$$\lambda = \frac{h}{mv} = \frac{h}{mL/t}$$
 where L is the path length and t time of flight
- a single stationary detector records whole diffraction spectrum as a function of time of flight
- neutrons travel at $\sim 100\text{m/s}$ (speed of sound)

$$\lambda = 2d \sin \theta \quad \text{with } \theta \text{ fixed} \quad \text{i.e. } \lambda \text{ proportional to } d$$

Spallation Sources: Time of Flight



Spallation Sources

- Pulsed/Continuous Spallation Sources

- "Whole-Spectrum (TOF)"

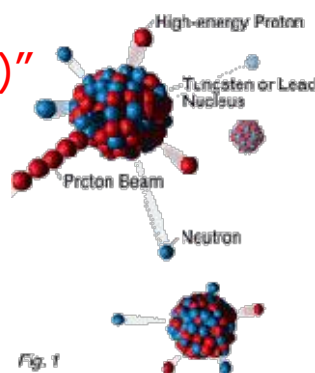
- Facilities in Europe:

- Pulsed: ISIS (UK)
- POLDI (CH)

- More information

$$\lambda = 2d \sin \theta$$

fixed



Spallation Sources: Measurement of Strain

Strain: $\varepsilon = \frac{a - a_0}{a_0} = \frac{\lambda - \lambda_0}{\lambda_0} = \frac{t - t_0}{t_0}$

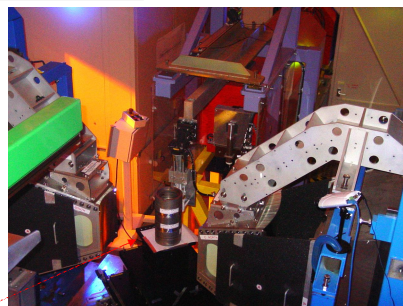
Cubic gauge volume !



Time-of-Flight:

$$\lambda = \frac{h}{2mL \sin \theta} t$$

Fixed



ENGIN-X at ISIS

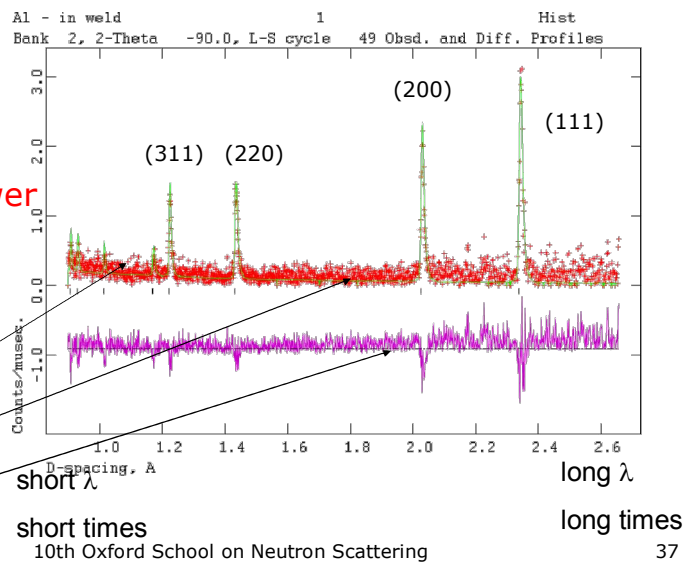
Example of TOF: Rietveld Refinement

TOF: More
Information!

Rietveld: Fewer
Parameters!

$$d_{hkl} = \frac{a_0}{\sqrt{h^2 + k^2 + l^2}}$$

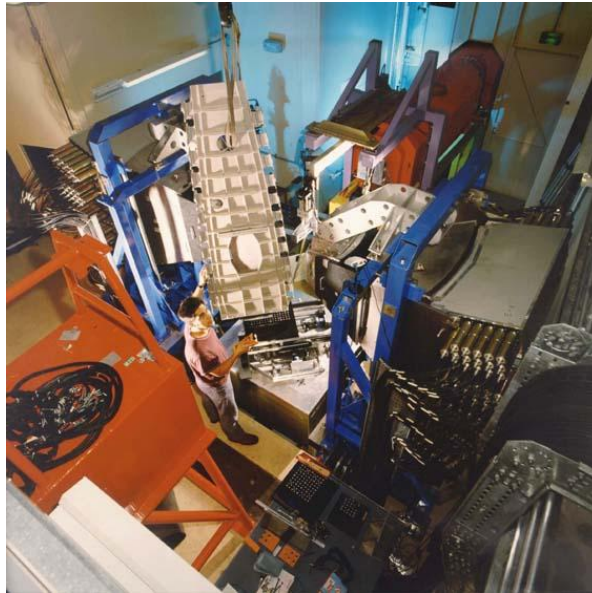
Data
Fitting
Difference



ENGINE-X, ISIS

QuickTime™ and a
Microsoft Video 1 decompressor
are needed to see this picture.

ENGIN-X, ISIS

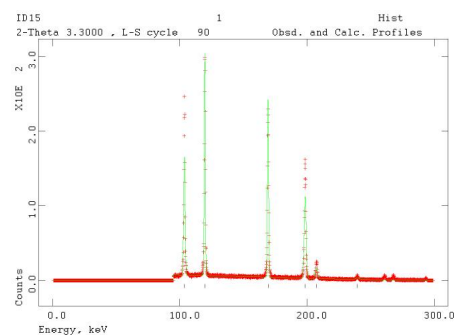


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Energy Dispersive Synchrotron Diffraction

- Larger range of wavelengths available
- Energy/Strain Resolution up to $10E-5$
- Higher penetration depth
- More elongated GV



$$\lambda = \frac{hc}{E} = \frac{12.39}{[keV]}$$



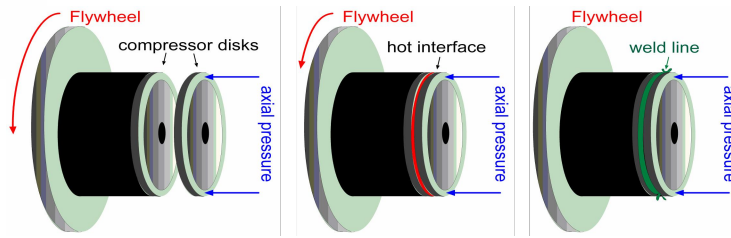
$$\varepsilon = \frac{E_0}{E} - 1$$

Strain

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Case Study: Inertia Friction Welding

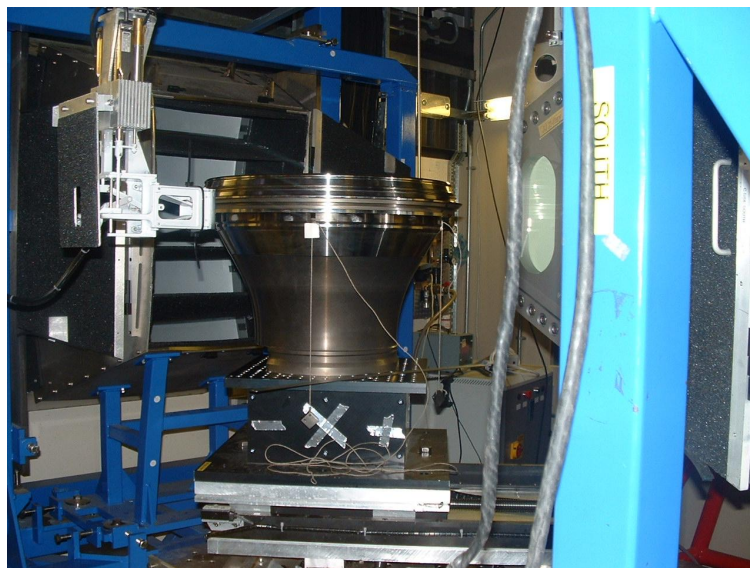


Solid state joining of compressor, turbine discs and shafts

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Case Study: Inertia Friction Welding

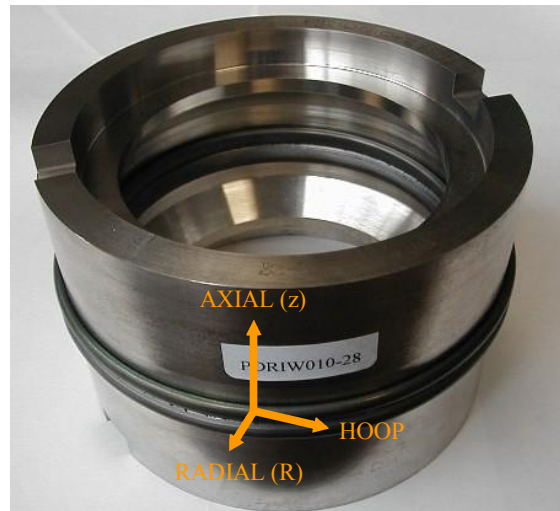


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How would you measure such a sample ?

143mm diameter test inertia friction welds

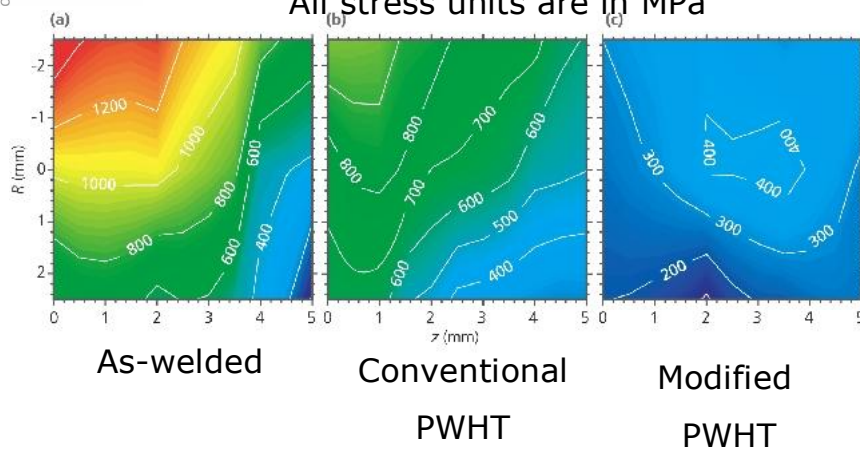


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Hoop stresses in IFW'd nickel-base superalloy

All stress units are in MPa



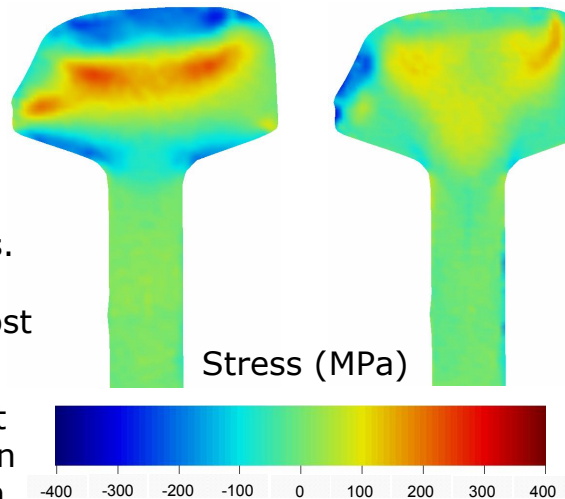
Residual stress measurements were used to develop
a new PWHT

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Railway Rails

- Slices were cut from the rail to measure the horizontal and vertical stresses. Longitudinal stresses were lost
- Measurements were carried out by using neutron and synchrotron x-ray diffraction



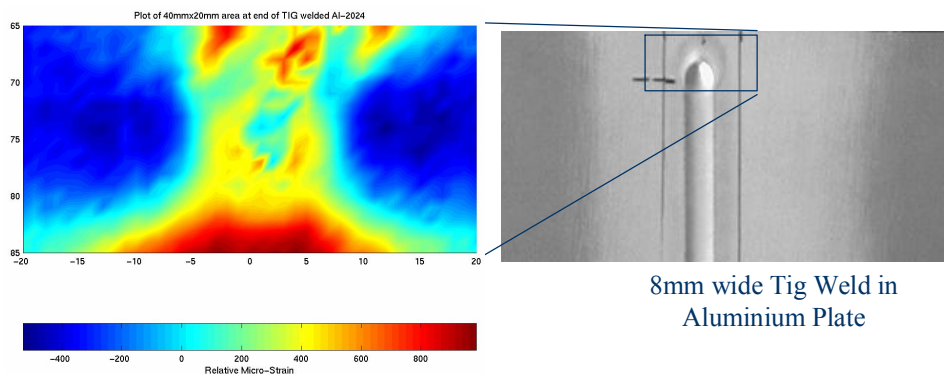
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Case Study: Strain Mapping of a TIG weld

2D Map of Residual Strain about the End of a TIG Weld at 100 μ m Resolution

This map include 20,00 measurements and took 8 hours to acquire

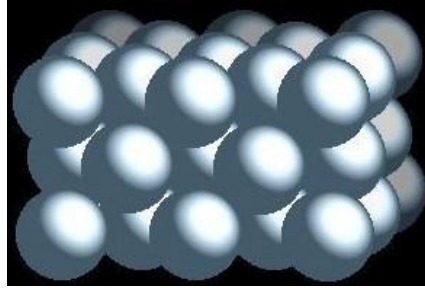


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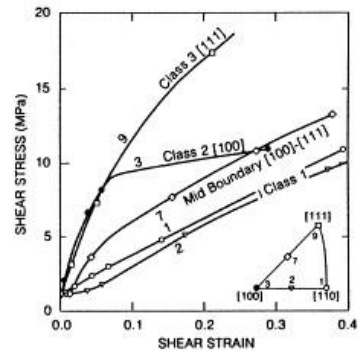
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From Engineering to Physical Metallurgy

Single Crystal Anisotropy



Al, fcc

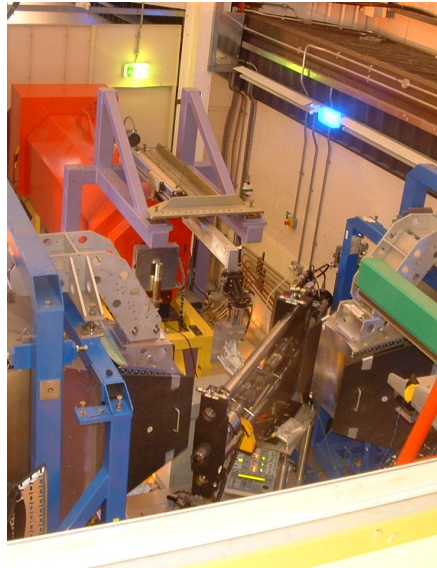


Single Crystal deformation

Deformation heterogeneity

- Polycrystalline deformation is heterogeneous
- Single crystal elastic and plastic anisotropy
- Grain incompatibility during deformation results in intergranular stresses

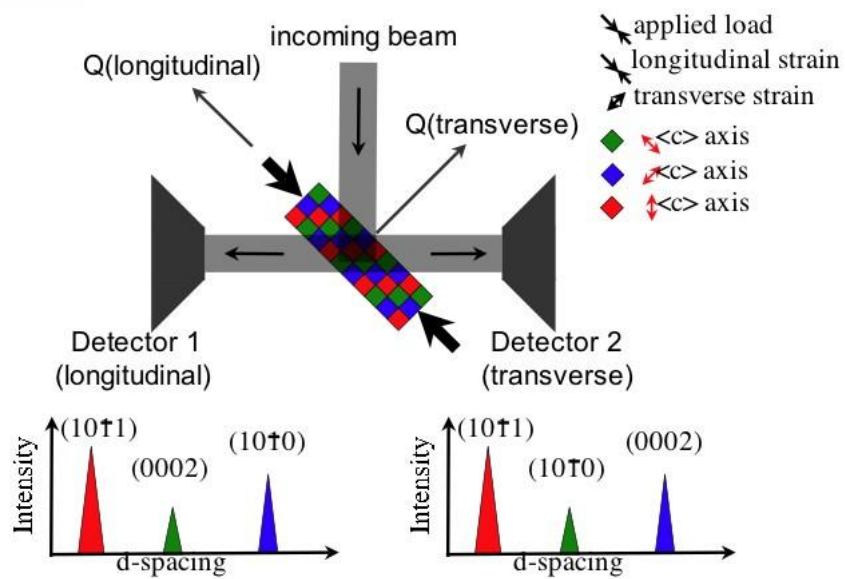
In-situ loading experiments



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Principle of in-situ loading



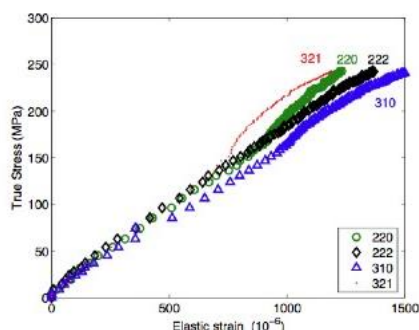
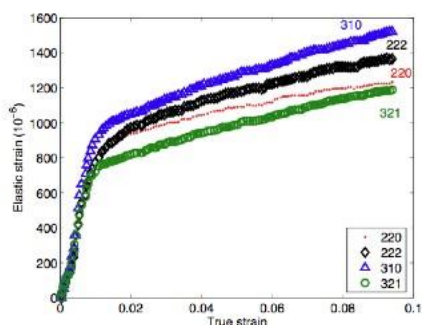
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In-situ straining

Elastic strain/elastic + plastic strain

Stress/elastic strain



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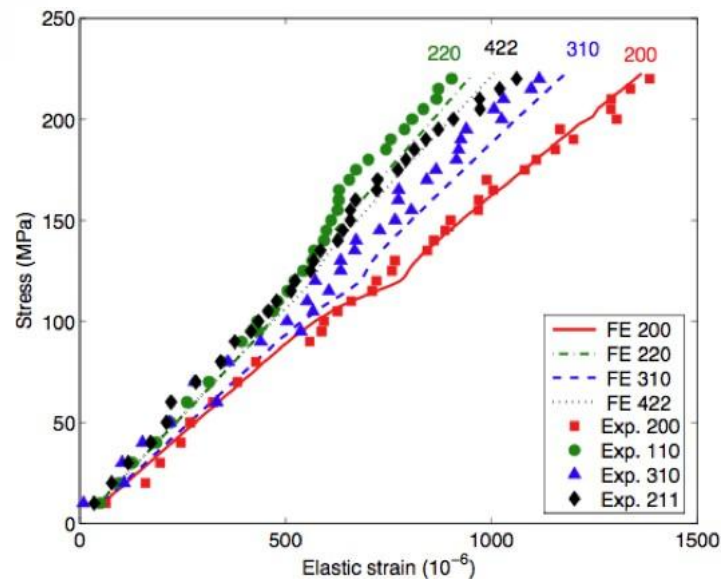
Modelling deformation

- Micromechanics
 - Dislocations, particles, grain boundaries (grain size), interstitial atoms
- Continuum mechanics:
 - Stresses and strains
 - Intergranular stresses
- Polycrystal plasticity
 - Mean field methods, i.e. every grain has the same matrix
 - Finite element methods
 - Each grain has a characteristic neighbourhood
 - Predict maximum and minimum stresses ?

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Plasticity Modelling (CPFEM)



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Attempted General Guidelines: Neutrons

Neutrons:

- Non-destructive, full stress analysis because of cubic Gauge Volume (think three directions)
- Good penetration depth due to neutrality
- Big bulky sample with low stress gradients
- Reasonable spatial resolution independent of atomic number
- Steels, aluminium, nickel, copper zinc or related
- Sample in harsh environment: furnace, cryo. etc.
- Phase analysis with Rietveld analysis

Not-so good: near surface or thin materials, titanium, boron cadmium, fast, high-spatial resolution, high instrumental resolution, hydrogenous materials

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Attempted General Guidelines: Synchrotrons

Synchrotrons:

- Non-destructive, fast strain mapping, mostly single peak
 - Light alloys (small atomic number)
 - High spatial resolution aluminium-titanium (think microns)
 - High instrumental resolution (small peak width)
 - Near surface measurement because of analyser crystal
 - Bulk materials / larger atomic number with energy-dispersive method
 - Polymers
- Not so good at: Steels and higher, big bulky samples, harsh environments, diamond shaped GV