Advanced Practical Course from the Technisches Unversität München

Residual stress analysis with neutrons at STRESS-SPEC instrument

Introduction

This experiment is intended to familiarize you with the possibilities which neutron diffraction and namely the STRESS-SPEC diffractometer offers to residual stress analysis. Residual stresses are self-equilibrating stresses in a body that is stationary and at equilibrium with its surroundings.

Mechanical stresses in solids are connected via their elastic material properties to elastic lattice strains, i.e. to the changes of distances and relative positions of the atoms in the crystal lattice. In this respect diffraction-based techniques are powerful methods for an engineer since they are able to quantify the distances between atomic planes accurately, providing a nondestructive probe for elastic strain and thus stress characterization. In particular, the penetration depth of thermal neutron radiation into matter makes neutron diffraction an increasingly important technique in assisting engineering design and the advancement of engineering materials. Evaluation and interpretation of the results of diffractometric strain and stress analysis requires fundamental knowledge on definitions and concepts of elasticity theory, which will be illustrated in the following chapter. In context of this tutorial, however, we forgo a detailed description of scattering theory in polycrystalline materials (Bragg scattering) and refer to corresponding textbooks on solid state physics [1,2].

Basic Principles

Residual stresses arise because of shape misfits (sometimes called "eigenstrains") between the unstressed shapes of different parts, different regions, or different phases of the component. They may be categorized by cause (e.g. thermal or elastic mismatch), by the scale over which they self-equilibrate, or according to the method by which they are measured.

To illustrate and define the stress components we consider a small cube with face areas *A* within a homogeneously stressed body, Figure 1. The cube is aligned to the axes of the coordinate system X, Y, Z (Figure 1). Each face is characterized by one of the unit vectors X, Y, Z or -X, -Y, -Z. Firstly we regard the faces towards the positive directions. The surrounding material will excert forces F_i on the face *i* of the cube. The forces are proportional to the surface area A. Each of the forces F_i can be partitioned in components parallel to the three coordinate axes. The stress component σ_{ij} is then defined to be the force per area acting on the face *i* in direction *j*:

$$\sigma_{ij} m_j = \frac{\mathrm{F}_i}{\mathrm{A}}$$

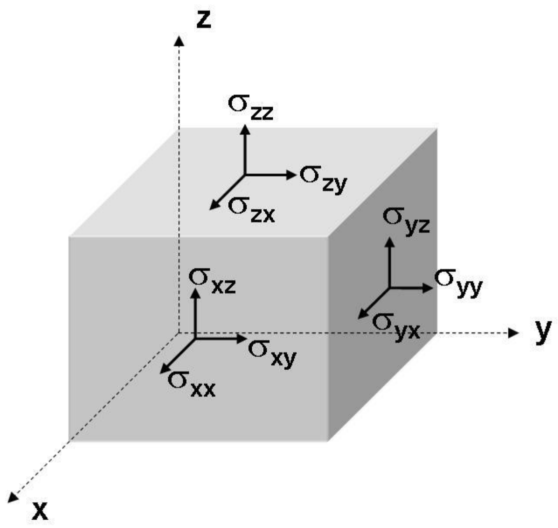


Figure 1 – Definition of residual stress tensor

For instance, σ_{xx} is acting on the X-face in x-direction, σ_{yz} acts on the Y-face in z-direction. $\sigma_{ij}\ (i = j)$ are called normal components, because the respective forces act perpendicular (normal) to a face, the $\sigma_{ij}\ (i \neq j)$ are called shear components, they act parallel to a face. Since the small cube is assumed to be homogeneously stressed, the forces on the three opposing sides are of the same amount but of opposite direction. The condition of force equilibrium of the stressed volume element account for the symmetry: $\sigma_{ij} = \sigma_{ji.}$ The stress components σ_{ij} form a symmetrical tensor of second rank with 9 components.
If a body is stressed, each point x within the body undergoes a displacement u and the material experiences a strain. In the following discussion displacement and strain are assumed to be small. For the one dimensional case (e.g. a string with the initial length L_0) the strain is defined as $\varepsilon(x) = d_u/d_x$. If ε is homogeneous, i.e. it does not depend on x, one obtains:

$$\varepsilon = \frac{(L - L_0)}{L_0} = \frac{\Delta L}{L_0}$$

In three dimensions the strain tensor describes the change of the volume of one volume element and also forms a symmetrical second rank tensor with 9 components.. Because of the symmetry conditions $\sigma_{ij} = \sigma_{ji}$ and $\varepsilon_{ij} = \varepsilon_{ji}$, only 6 of the 9 components are independent and are thus sufficient to describe the stress and strain state at a point in the material.

Elastic material properties

When a body underlies certain stresses, the strain response depends on the elastic properties of the material. The strain can be of elastic and of plastic kind.

$$\varepsilon = \varepsilon^{el.} + \varepsilon^{pl.}$$

When stresses are released, the elastic strain will vanish and the plastic part will remain. For stresses not exceeding the yield limit of the material, the strain response is only elastic. If the elastic strains are sufficiently small, they depend linearly on the applied stresses. In practise this is valid in most cases. In the one-dimensional case the Young's modulus E connects the stress and the strain (Hooke's law):

$$\sigma = E\varepsilon$$

E is the Young Modulus (usually given in [GPa])

The most general linear relation between the stress and the strain tensor is given, when each stress component depends linearly on all the 9 strain components. There are 9 equations with 9 independents.

$$\sigma_{ij} = c_{ijkl}\varepsilon_{kl}$$

This relation defines the 4th rank tensor of elastic stiffnesses, it has 3^4 = 81 components c_{ijkl}. The stress as well as the strain tensor is symmetric, and so also the elastic stiffness tensor can be written symmetrical:

$c_{ijkl} = c_{jikl} = c_{ijlk}$,

which reduces the number of independent components to 21. The number of independent components is further reduced by symmetry of the crystal lattice. This leaves in case of an elastically isotropic material with cubic symmetry only two independent components; for further details see references [2-4]. The elastic behavior of such an isotropic body is then fully described by the Young's modulus E and the Poisson's ratio ν.

Nature and origin of residual stresses

All stress states within a material which are independent of outside forces are called residual stresses. The average of the residual stresses taken over each cross section of the body has to be zero. Examples how residual stresses arise are illustrated in Figure 2. They originate from misfits between different regions. In many cases, these misfits span large distances, for example, those caused by the non-uniform plastic deformation of a bent bar. They can also arise from sharp thermal gradients, for example, those caused during welding or heat treatment operations (Figure 2). These stresses can be advantageous, as in the case of shot peening (mechanically induced) and toughening of glass (thermally induced).

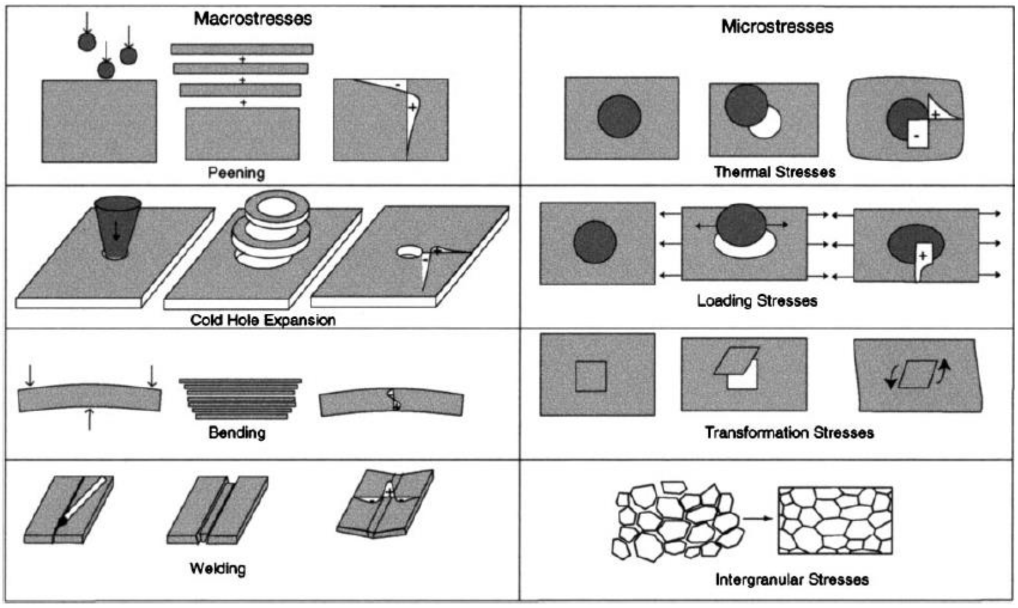


Figure 2 – Typical examples of residual stress origins: misfits between different regions of the material or between different phases within the material. Schematic illustration of macro and micro residual stresses [2]

The stresses are commonly divided into three classes, or types, by the length scales over which they vary and over which they self-equilibrate, and are labelled as type I, type II, and type III stresses. They are often categorized as macrostresses (Type I) and microstresses (types II and III). Type I stresses self-equilibrate over a length which is comparable to the macroscopic dimension of the structure or component in question. Type II stresses self-equilibrate over a length scale comparable to that of the grain structure. They arise from misfits having a characteristic length scale, comparable to the grain size of polycrystalline solids, usually a few tens of microns. Type III stresses self-equilibrate over a length scale smaller than the characteristic length scale of the microstructure; that is, the grain size or the fiber/particle spacing for composite materials. These could be stresses varying within a specific grain, such as due to grain subdivision into smaller cell structures. In the case of real materials, the actual residual stress state at a point is the superposition of stresses of type I, II and III stresses, as illustrated in Figure 3. In a diffraction experiment both type I and the average type II stresses for the particular grain set will cause a shift of the Bragg reflections, while type III stresses can be only identified due to the broadening of the interference line. As diffraction is phase specific it can be exploited for multiphase materials to provide information on the residual stress state of the individual crystalline phases separately. Figure 3 shows schematically how the overall stress state is composed in a two-phased material.

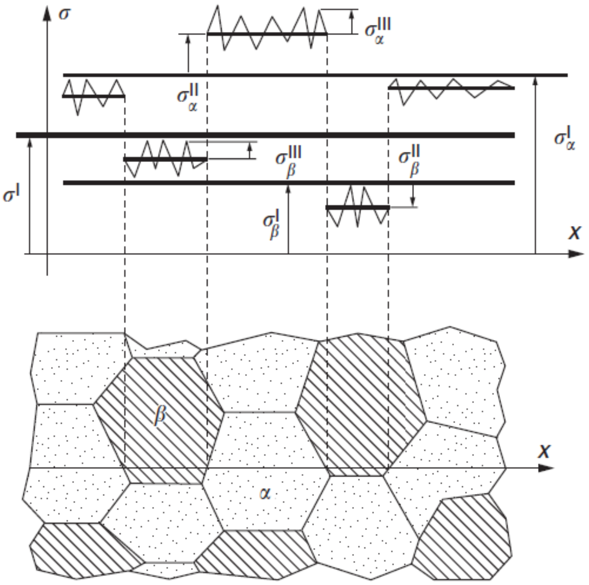


Figure 3 – Schematic representation of the 3 types of residuals stresses in a two-phase material (phases α and β) [5].

Basic equation for residual stress analysis by diffraction

The Bragg equation is, in its simplicity, the essential basis for diffraction-based techniques for strain and hence residual stress characterization.
Changes in inter-planar spacing d_{hkl} of atoms, Figure 4(a), can be used with the Bragg equation to measure elastic strain ε through the knowledge of the incident wavelength λ and the change in the Bragg scattering angle $\Delta\theta$.

$$\lambda = 2d_{hkl}\sin\theta_{hkl}$$

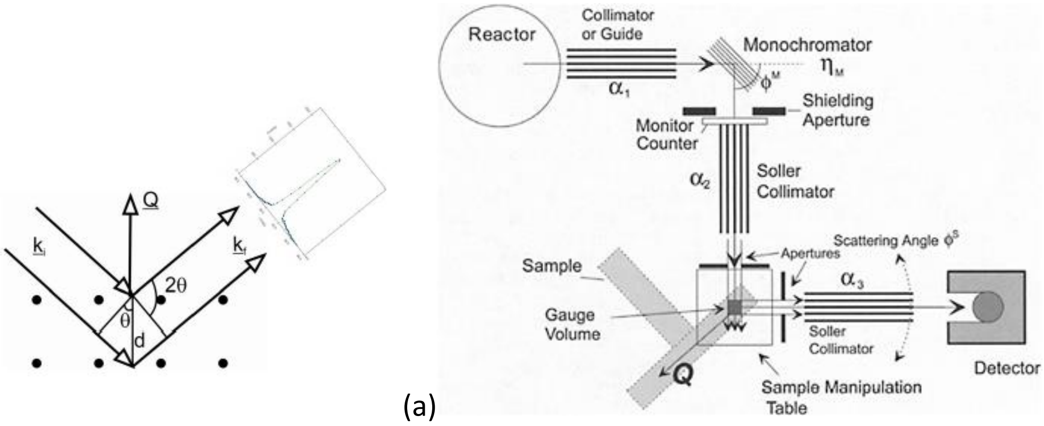
giving

$$\varepsilon_{hkl} = \frac{\Delta d_{hkl}}{d_{hkl}} = -\cot\theta_{hkl}\Delta\theta_{hkl}$$

For calculating strain it is necessary to have an accurate measure of the stress free spacing d_0,. The strain results can then be converted into stress using a suitable value of the stiffness.

A typically neutron diffractometer for strain measurements, in this case at steady state reactor, is shown in Figure 4 (b). A beam of polychromatic neutrons emanating from the neutron source impinges on the monochromator which reflects a monochromatic beam according to Bragg's law. A small fraction of the beam hitting the sample is diffracted. As the incident beam is monochromatic, the diffracted beam is confined to a well-defined direction, again given by Bragg's law. Finally, the diffracted beam is recorded by a neutron detector. Collimators placed before and after the monochromator and after the sample define the angular spreads of the neutron beam in the horizontal and in the vertical plane, respectively. To achieve the necessary spatial resolution for strain mapping the relatively wide beam generated by a typical neutron monochromator is narrowed down by a slit, a few

millimeters wide, placed just before the sample. Similarly, the diffracted beam is confined by another slit placed just after the sample. These two slits define the gauge volume which is normally much smaller than the sample itself. Strain mapping over the entire sample is then done by moving the sample stepwise with respect to the gauge volume.
The direction in which the lattice strain ε_{hkl} is measured is parallel to the scattering vector Q. In order to determine the strain in different directions in the sample, the sample must be rotated accurately about the center of the gauge volume so that each required direction is parallel to Q.



(a) (b)

Figure 4 - (a) Illustration of diffraction by a lattice plane. (b) Schematic representation of a strain instrument on a steady state neutron source [2].

This requires very careful alignment and centering of the sample, and is a major challenge to the instrument designer and to the experimentalist.

Figure 5 shows the general case of a strain measurement of a plate sample where the strain is measured for crystallographic planes {hkl} which are perpendicular oriented to the direction (φ,ψ) within the diffractometer coordinate system. Using the generalised form of the Hooke's law and the so called diffraction elastic constants (taking into account the elastic anisotropy of the crystal) the strain and stress component can be related:

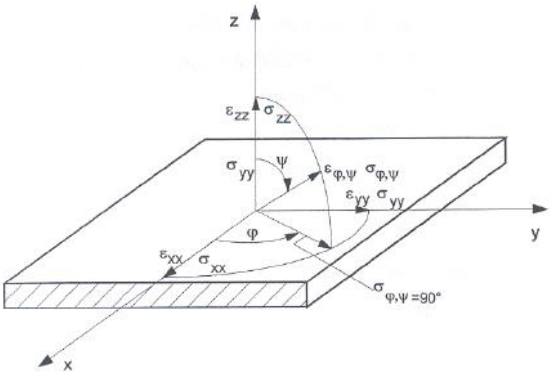


Figure 5 - Coordinate system for residual stress measurements.

$$\varepsilon_{\varphi,\psi}^{hkl} = \frac{1}{2}S_2^{hkl}\left[\left(\sigma_{xx}\cos^2\varphi + \sigma_{xy}\sin 2\varphi + \sigma_{yy}\sin^2\varphi - \sigma_{zz}\right)\sin^2\psi\right] + \sigma_{zz}\cos^2\varphi + \frac{1}{2}S_2^{hkl}\sigma_{zz} + S_1^{hkl}\left(\sigma_{xx} + \sigma_{yy} + \sigma_{zz}\right) + \frac{1}{2}S_2^{hkl}\left[\left(\sigma_{xz}\cos\varphi + \sigma_{yz}\sin\varphi\right)\sin 2\psi\right]$$

with

$$S_1^{hkl} = \frac{\mathrm{\gamma}^{hkl}}{\mathrm{E}^{hkl}}$$

and

$$\frac{1}{2}S_2^{hkl} = \frac{\mathrm{\gamma}^{hkl} + 1}{\mathrm{E}^{hkl}}$$

In many cases symmetry considerations enable assumptions about the principal stress and strain directions to be made, and then the elaborate and time-consuming process of measuring the full strain tensor based on six or more measurements is avoided. In this case, only the three orthogonal principal strain components need to be determined.

$$\sigma_{xx} = \frac{\mathrm{E}^{hkl}}{(1 + \mathrm{\gamma}^{hkl})}\left[\varepsilon_{xx} + \frac{\mathrm{\gamma}^{hkl}}{1 - 2\mathrm{\gamma}^{hkl}}(\varepsilon_{xx} + \varepsilon_{yy} + \varepsilon_{zz})\right]$$

Penetration depth

Thermal neutrons have the advantage over X-rays that for wavelengths comparable to the atomic spacing, their penetration into engineering materials is typically many centimeters instead of a few tens of µm. The true absorption of thermal neutrons by matter is due to nuclear reactions: for most elements the absorption is weak (in particular for C, O, Al, Fe, Zr, Nb, Pb). In some cases, however, the absorption by nuclear reactions is very high (e.g. B, Cd, Gd). In Figure 6 the material thickness of different materials necessary to attenuate the neutron beam by 50% (λ= 1.8 Å), is shown.

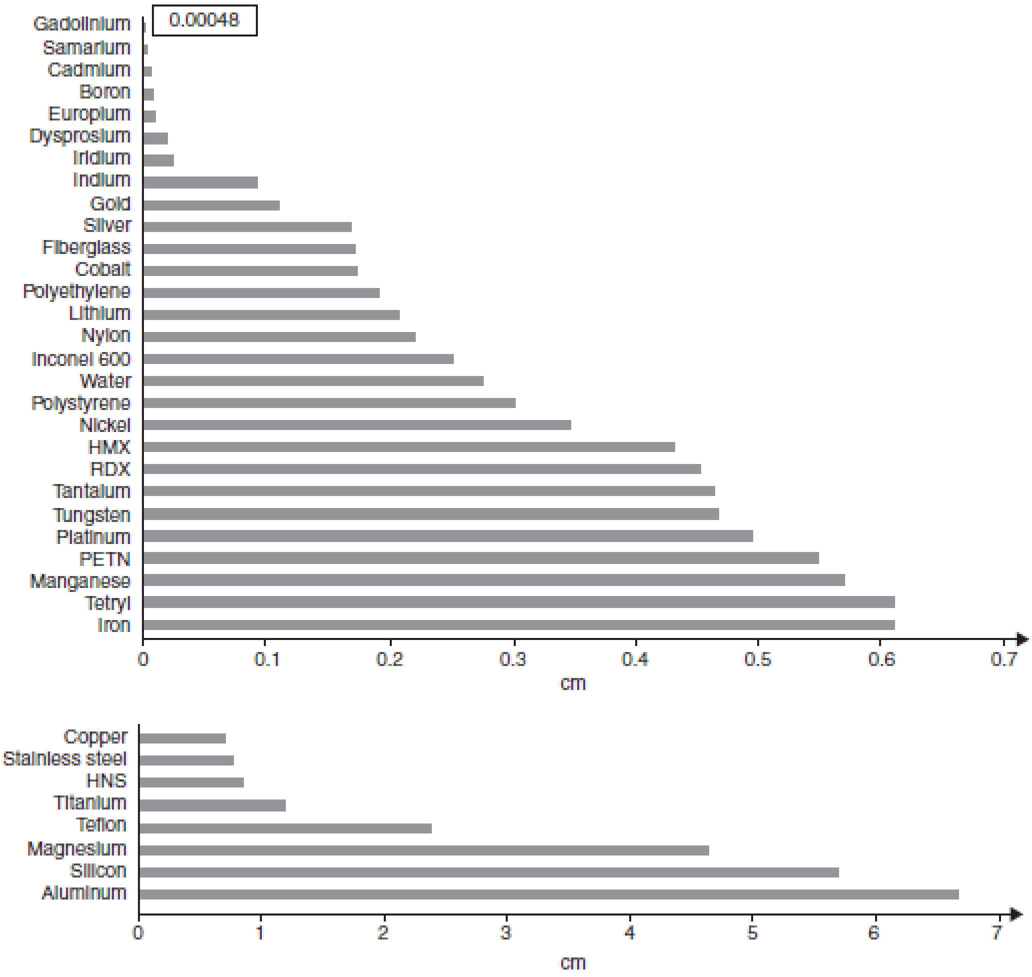


Figure 6 - Material thickness required to attenuate the beam by 50% [5].

The linear absorption coefficient μ is dependent on the wavelength and obeys the exponential law:

$$I = I_0 \exp(-\mu . x)$$

x is the neutron path length within the sample and μ the absorption coefficient of the material. The absorption coefficient can be calculated by:

$$\mu = \sum_{ges} \rho$$

where

ρ is the atom density [Atom/cm^3]

Σ_{ges} is the total absorption cross section [barn]

Σ_{ges} is constituted by

$$\sum_{ges} = \sigma_{inc} + \sigma_{abs}(\lambda) + \sigma_{coh}$$

$\sigma_{\text{coh}} = \pi r(2R)^2 = 4\ \pi b^2$

where

σ_{inc} is the incoherent cross section [barn = 100 fm^2]

$\sigma_{\text{abs}}(\lambda)$ is the absorption cross section for a specific wavelength [barn]

σ_{coh} is the coherent cross section [barn]

R = b = neutron scattering length [fm]

Neutron scattering length b is defined by the radius of the effective cross section

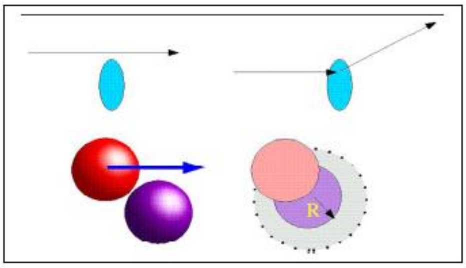
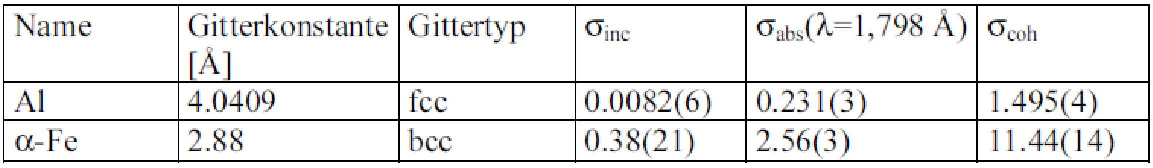


Tabelle 1



Name	Gitterkonstante [Å]	Gittertyp	σ_{inc}	$\sigma_{\text{abs}}(\lambda=1{,}798\ \text{Å})$	σ_{coh}
Al	4.0409	fcc	0.0082(6)	0.231(3)	1.495(4)
α-Fe	2.88	bcc	0.38(21)	2.56(3)	11.44(14)

Question:

1. Before starting the experiment, please determine the absorption coefficients of aluminum (Al) and iron (Fe) using the data of table 1.

2. Inform yourself about the definition of a cubic crystal system, lattice type and Miller indices. With this you should be able to calculate the optimal wavelength for which the diffraction angle of the Al (311) and Fe (211) lattice planes will be found at around 90°.

Experimental setup

The materials science diffractometer STRESS-SPEC is located at the thermal beam port SR-3 of the FRM II and can easily be configured either for texture analysis or strain measurements. The different instrument components are shown schematically in Figure 7:

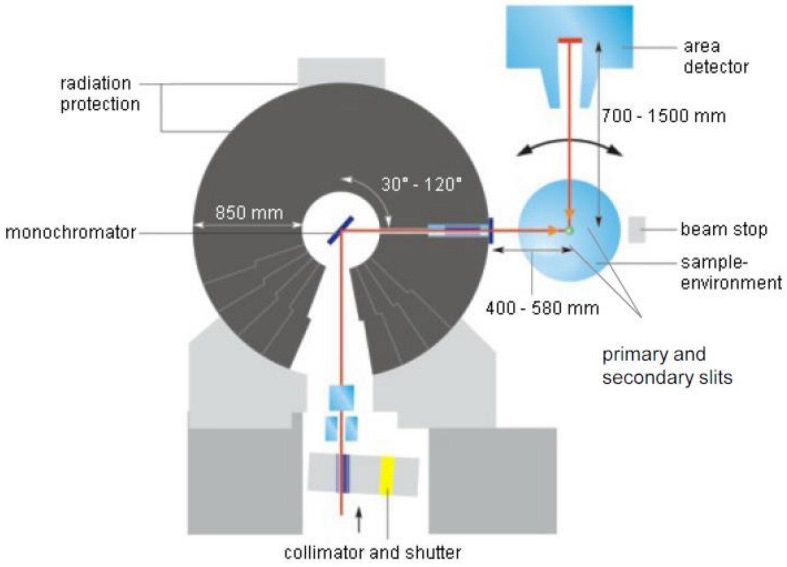


Figure 7 - Schematic representation of the material science diffractometer STRESS-SPEC

In the beam tube SR-3 there is a collimator drum which enables us to use the neutron beam with the natural collimation of the beam tube (ca. 60’), or collimatiors of 25’ and 15’ when reduction of the beam divergence is necessary. The monochromator shielding allows continuous variation of the wavelength by setting the monochromator at the respective Bragg diffraction condition:

$$\lambda = 2d_{hkl} \sin \theta_{hkl}$$

This selection of monochromators and the possibility to vary automatically the take-off angles from $2\theta_M$ = 35° to 110° allows to find a good compromise between resolution and intensity for each measuring problem. In addition this particular feature results from the necessity for strain measurements that adjustment of a suitable wavelength offers the possibility to perform measurements around $2\theta = 90°$. This is important with regard to spatial resolution for measurements of components, since the volume element is kept cubic for all required measuring directions at the case of $2\theta = 90°$.

For high resolution experiments like for instance ceramic materials a good definition of the wavelength and therefore high take-off angles of the monochromator up to 120° are required. In cases where high intenisties are required, i.e. texture measurements of metallic materials, small take-off angles of the monochromator are preferable.

Sample stage

For strain mapping a high precision sample stage is necessary to align and adjust the components. The translation device (x,y,z) available at STRESS-SPEC allows sample positioning with an accuracy better than 10 μm for components as heavy as 300 Kg.

Slits and adjustment system

The gauge volume dimension can be defined continuously in the incoming (primary) beam up to 7 x 17 mm2 (WxH) and for the diffracted (secondary) beam side continuously up to 15 mm. Radial collimators with a FOV of 5 mm, 2 mm and 1 mm are available. For the precise adjustment of the sample a laser and and further optical systems are installed.

Detector

A two dimensional position sensitive ^{3}He- detector is used with a detection area of 25 x 25cm^2 divided in 256 x 256 Pixel. The distance between the sample table centre and detector is variable between 0.75 m until 1.7 m.

Description of the experiment

The material science diffractometer STRESS-SPEC is built and optimized for measuring the complete residual stresses tensor of relevant industrial components. To avoid complexity of this tutorial the analysis the complete residual stress tensor will not be carried out.
All measurements in this experiment will be carried out using the standard control and analysis software. Because of the instrument complexity an instrument scientist will be allays present during the tutorial.

Following experiments will be performed in this tutorial:

1 Experimental determination of the absorption cross sections of aluminum and iron - by means of intensity measurement of samples with different thickness.

2. Experimental extraction of diffraction elastic constants of different lattice planes Fe (220) and Fe (211) using a tensile text rig.
   - Mount the extensometer on the sample and the sample into the tensile rig.
   - Adjust the sample position.
   - While tensioning the sample start aquiring diffraction data for the (211) reflection. The measurements are carried out along the tension direction. The strains should be calculated using the first measuring point without force as a reference value. Repeat the measurements for the (220) reflection using a new sample.
   - Calculate the diffraction elastic constant of the measured lattice plane from the in-situ diffraction data.

3. Spatial resolved measurement of iron bar sample, which was deformed by four point bending, along its cross section.
   - Mount the sample at the sample table.
   - Align and adjust the sample. Choose a suitable Bragg reflection.
   - Write a scan script to measure the spatial resolved measurement steps along the sample cross section.
   - Fit the Bragg reflections for each measuring step. This data will be used to calculate the starin across the bar and from the strain the residual stress.

Analysis/Experimental Report
As usual at the end of an experimental work, the analysis of the results and a brief report of what was done is mandatory. If during the experiment a protocol is written, the effort will be considerably reduced when writing the report afterwards. Try to be concise and clear in the report and perform a proper error calculation.

In detail:

1. Calculate the absorption coefficient from the measured curves for each sample and compare the obtained value with the one found in preparation of this experiment.
2. Draw a strain-stress diagram from the measured data of the (220) and (211) reflections. Calculate for each set the (phase specific) diffraction elastic constants,

E_{hkl}. Compare the experimental calculated diffraction elastic constant with the macroscopic elastic module, of steel. Discuss your findings.

3. Calculate the residual stress profile along the cross section of the bent bar sample using microscopic elastic modulii obtained in 2. A reference value for calculating the stress can be obtained when assuming mechanical equilibrium along the cross section of the sample. Discuss your findings and why mechanical equilibrium is appropriate here.

Help: Additional and more detailed information on the four point bending test can be found in references [4,5].

References

[1] G.L. Squires, Introduction to the Theory of Thermal Neutron Scattering, Dover reprints (1978)

[2] M.T. Hutchings et al., Introduction to the Characterization of Residual Stress by Neutron Diffraction, Taylor&Francis (2004)

[3] V. Hauk, Structural and Residual Stress Analysis by Nondestructive Methods, Elsevier (1997)

[4] l. Spieß et al., Moderne Röntgenbeugung , B.G.Teubner Verlag (2005)

[5] Analysis of Residual Stress by Diffraction using Neutron and Synchrotron Radiation, M. E. Fitzpatrick and A. Lodini Taylor & Francis (2003)