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

Essais non destructifs — Méthode normalisée de détermination des contraintes résiduelles par diffraction de neutrons

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 135, *Non-destructive testing*, Subcommittee SC 5, *Radiographic Testing*.

This first edition cancels and replaces ISO/TS 21432:2005, which has been technically revised. It also incorporates the Technical Corrigendum ISO/TS 21432:2005/Cor 1:2008. Furthermore this document replaces ISO/TTA3:2001.

The main changes compared to ISO/TS 21432 are as follows:

— Figures 1 and 5 were replaced with updated, more suitable versions. The keys for several figures were updated in order to better reflect and explain the content of the figures.

— 5.4 was rearranged to emphasize the distinction between monochromatic instruments and time-of-flight instruments.

— The former Clause 7 became Clause 6 and vice versa. The new order reflects better the real order of steps taken in the preparation of a measurement.

— 7.6 was updated to provide additional details on the determination of the stress-free reference value.

— Clause 10 was slightly modified and the references to the ISO/IEC Guides relevant to uncertainty determination were updated.

— 11.7 was added in order to include uncertainties and errors in the reporting.

— A.5.4 was revised and amended to provide more information on grain size effects and the possibilities to mitigate these.

— A.9 was added to explain the calculation of stresses in the case of macroscopically anisotropic material.

— The Bibliography was updated by including a few new references.

— Throughout the document minor revisions of the text were implemented in order to correct small errors and to improve the clarity.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Neutron diffraction is a non-destructive method that can be employed for determining residual stresses in crystalline materials. It can also be used to determine internal stresses in samples subjected to applied stresses. The procedure can be employed for determining stresses within the interior of materials and adjacent to surfaces. It requires specimens or engineering components to be transported to a neutron source. Elastic strains are derived from the measurements, which in turn are converted into stresses. The purpose of this document is to provide an International Standard for reliably determining stresses that are relevant to engineering applications.

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

WARNING — This document does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document describes the test method for determining residual stresses in polycrystalline materials by neutron diffraction. It is applicable to both homogeneous and inhomogeneous materials including those containing distinct phases.

The principles of the neutron diffraction technique are outlined. Suggestions are provided on:

— the selection of appropriate diffracting lattice planes on which measurements should be made for different categories of materials,

— the specimen directions in which the measurements should be performed, and

— the volume of material examined in relation to the material grain size and the envisaged stress state.

Procedures are described for accurately positioning and aligning test pieces in a neutron beam and for precisely defining the volume of material sampled for the individual measurements.

The precautions needed for calibrating neutron diffraction instruments are described. Techniques for obtaining a stress-free reference are presented.

The methods of making individual measurements by neutron diffraction are described in detail. Procedures for analysing the results and for determining their statistical relevance are presented. Advice is provided on how to determine reliable estimates of residual stresses from the strain data and on how to estimate the uncertainty in the results.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 13925-3:2015, *Non-destructive testing — X-ray diffraction from polycrystalline and amorphous materials — Part 3: Instruments*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
neutron absorption
neutron capture by an atomic nucleus

Note 1 to entry: A table of nuclear capture cross-sections can be found in Reference [1].

3.2
alignment
adjustment of the specimen position and orientation and also of all the components of the instrument such that measurements can be performed precisely at the desired location in the specimen

3.3
anisotropy
dependence of material properties on the direction with respect to the sample

3.4
attenuation
reduction of the neutron beam intensity

Note 1 to entry: Attenuation can be calculated by using the so-called "total neutron cross-section", which comprises *neutron absorption* (3.1) and different nuclear scattering processes. The attenuation length is the distance within the material for which the primary neutron beam intensity is reduced by $1/e$.

3.5
background
intensity considered not belonging to the *diffraction* (3.13) signal

Note 1 to entry: Background dependence on the scattering angle or *time-of-flight* (3.34) is not uncommon and can have an influence on the *peak position* (3.11) resulting from data analysis.

3.6
beam-defining optics
arrangement of devices used to define the properties of a neutron beam such as the wavelength and intensity distributions, divergence and shape

Note 1 to entry: These include devices such as apertures, slits, collimators, monochromators and mirrors.

3.7
Bragg edge
sharp change in the neutron intensity as a function of the wavelength or monochromator take-off angle corresponding to the condition $\lambda = 2d_{hkl,}$ where *hkl* indicates an (*hkl*) diffracting lattice plane of the material under investigation

3.8
Bragg peak
intensity distribution of the neutron beam diffracted by a specific (*hkl*) lattice plane

3.9
peak height
maximum number of neutron counts of the *Bragg peak* (3.8) above the *background* (3.5)

3.10
peak function
analytical expression to describe the shape of the *Bragg peak* (3.8)

3.11
peak position
single value describing the position of a *Bragg peak* (3.8)

Note 1 to entry: The peak position is the determining quantity to calculate the strain.

3.12
peak intensity
integrated intensity
area under the *diffraction* (3.13) peak above the *background* (3.5), normally calculated from the associated fitted parameters of a selected *peak function* (3.10) and a background function

3.13
diffraction
scattering arising from coherent interference phenomena

3.14
diffraction elastic constants
E_{hkl}
ν_{hkl}
elastic constants associated with *diffraction* (3.13) from individual (*hkl*) lattice planes for a polycrystalline material

3.15
diffraction pattern
intensity distribution of neutrons diffracted from a crystalline material over the available wavelength, *time-of-flight* (3.34) and/or *diffraction* (3.13) angle ranges

3.16
full width at half maximum
FWHM
width of the *Bragg peak* (3.8) at half the *peak height* (3.9) above the *background* (3.5)

3.17
full pattern analysis
determination of the crystallographic structure and/or strain from a measured (multi-peak) *diffraction pattern* (3.15) of a polycrystalline material

Note 1 to entry: In general, the full pattern analysis is termed after the method used (e.g. Rietveld refinement). See also *single peak analysis* (3.31).

3.18
gauge volume
volume from which information is obtained

3.19
lattice parameters
linear and angular dimensions of the crystallographic unit cell

3.20
lattice spacing
***d*-spacing**
lattice plane spacing
distance between adjacent parallel crystallographic lattice planes

3.21
Type I stress
macrostress
stress that self-equilibrates over a length scale comparable to the structure or component, thereby spanning multiple grains and/or phases

3.22
Type II stress
stress that self-equilibrates over a length scale comparable to the grain size

Note 1 to entry: Stresses of Type II and Type III are collectively known as microstresses.

3.23
Type III stress
stress that self-equilibrates over a length scale smaller than the grain size

Note 1 to entry: Stresses of Type II and Type III are collectively known as microstresses.

3.24
monochromatic instrument
instrument employing a narrow band of neutron energies (wavelengths)

3.25
monochromatic neutron beam
monochromatic beam
neutron beam with narrow band of neutron energies (wavelengths)

3.26
orientation distribution function
quantitative description of the crystallographic *texture* (3.32)

Note 1 to entry: The orientation distribution function is necessary to calculate the elastic constants of textured materials.

3.27
polychromatic neutron beam
neutron beam containing a broad band of neutron energies (wavelengths)

3.28
reference point
centroid of the instrumental *gauge volume* (3.18)

Note 1 to entry: See 7.5.

3.29
reproducibility
closeness of agreement between indications or measured quantity values obtained under conditions of measurement, out of a set of conditions that includes different locations, operators, measuring systems, and replicate measurements on the same or similar objects

Note 1 to entry: A valid statement of reproducibility requires a specification of the conditions changed. These can include the principle of measurements, method of measurements, observer, measuring instrument, reference standard, location, conditions of use and time.

Note 2 to entry: Reproducibility can be expressed quantitatively in terms of the dispersion characteristics of the results.

Note 3 to entry: Results are here usually understood to be corrected results.

Note 4 to entry: This definition combines ISO/IEC Guide 99:2007, 2.25, 2.15, and 2.24.

3.30
incoherent scatterer
material scattering neutrons in an uncorrelated way thus giving rise to a strong *background* (3.5) signal and no *Bragg peaks* (3.8) or only some with low amplitude

3.31
single peak analysis
statistical procedure to determine the characteristics of a peak and the *background* (3.5) from the measured *diffraction* (3.13) data

3.32
texture
preferred orientation of crystallites, referred to as crystallographic texture, or other microstructural features, referred to as morphological texture, within a specimen

3.33
surface scan
wall scan
intensity scan
procedure to determine the position of a specimen surface or interface with respect to the *reference point* (3.28)

Note 1 to entry: The result is often called an entering curve.

3.34
time-of-flight
time needed by a neutron of a given speed to cover the distance from a defined starting point to the detector

3.35
uncertainty in a measurement
non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

Note 1 to entry: Measurement uncertainty includes components arising from systematic effects, such as components associated with corrections and the assigned quantity values of measurement standards, as well as the definitional uncertainty. Sometimes estimated systematic effects are not corrected for but, instead, associated measurement uncertainty components are incorporated.

Note 2 to entry: The parameter may be, for example, a standard deviation called standard measurement uncertainty (or a specified multiple of it), or the half-width of an interval, having a stated coverage probability.

Note 3 to entry: Measurement uncertainty comprises, in general, many components. Some of these may be evaluated by Type A evaluation of measurement uncertainty from the statistical distribution of the quantity values from series of measurements and can be characterized by standard deviations. The other components, which may be evaluated by Type B evaluation of measurement uncertainty, can also be characterized by standard deviations, evaluated from probability density functions based on experience or other information.

Note 4 to entry: In general, for a given set of information, it is understood that the measurement uncertainty is associated with a stated quantity value attributed to the measurand. A modification of this value results in a modification of the associated uncertainty.

[SOURCE: ISO/IEC Guide 99:2007, 2.26, modified — The term has been changed from "uncertainty of measurement", alternative terms "measurement uncertainty" and "uncertainty" have been removed.]

4 Symbols and abbreviated terms

4.1 Symbols and units

a,b,c	Lengths of the edges of a unit cell, here referred to as lattice parameters	nm
B	Background at the peak position	—
d	Lattice plane spacing	nm
E	Macroscopic elastic modulus	GPa
E_{hkl}	Elastic modulus associated with the (hkl) diffracting lattice planes	GPa
g	Strain gradient	mm^{-1}

h	Planck's constant	Js
hkl	Indices of a crystallographic lattice plane NOTE In the remainder of the document (hkl) will be used bearing in mind that each plane of the family {hkl} will diffract under the same conditions.	
$hkil$	Alternative Miller index notations of a crystallographic lattice plane for hexagonal structures	
H	Peak height above the background	—
I	Integrated neutron intensity of a Bragg peak above the background	
$\vec{k}_i, \vec{k}_f$	Wave vectors of the incident and diffracted neutrons	nm^{-1}
L	Path length from the neutron source to the detector	m
l	Neutron attenuation length	mm
m_n	Neutron mass ($1{,}67 \times 10^{-27}$ kg)	kg
N_n	Total number of neutrons counted	
$\vec{Q}$	Scattering vector ($\vec{k}_f - \vec{k}_i$)	nm^{-1}
t	Time of flight of neutrons from source to detectors	s
T	Temperature	°C or K
u	Standard uncertainty	—
x,y,z	Axes of the specimen co-ordinate system	
α	Coefficient of thermal expansion	K^{-1}
Δ	Variation of, or change in, the parameter that follows	
ε	Elastic strain	—
ε_{ij}	Components of the elastic strain tensor	—
ε_{hkl}	Normal elastic strain associated with the (hkl) diffracting lattice plane	—
λ	Wavelength of neutrons	nm
ν	Poisson's ratio	
ν_{hkl}	Elastic constant corresponding to Poisson's ratio associated with the (hkl) diffracting lattice plane	
$\vec{\vec{\sigma}}$	Stress	MPa
σ_{ij}	Components of the stress tensor	MPa
σ_Y	Yield stress	MPa
2θ	diffraction angle	degrees
ϕ, ψ	Orientation angles	Degrees

4.2 Subscripts

hkl, *hkil*	Indicate relevance to crystallographic (*hkl*) or (*hkil*) lattice planes
x, y, z	Indicate components of the quantity concerned along the x-, y-, z-axes
$\phi\ \psi$	Indicate the normal component, in the ($\phi\ \psi$) – direction of the quantity concerned
0 (zero)	Indicates stress-free value of the quantity concerned
ref	Indicates reference value of the quantity concerned

4.3 Abbreviated terms

PSD	Position sensitive detector
TOF	Time-of-flight
IGV	Instrumental gauge volume
NGV	Nominal gauge volume
SGV	Sampled gauge volume

5 Summary of method

5.1 General

This document is concerned with the determination of residual stresses that are needed in engineering analyses. The stresses are determined from neutron diffraction measurements of lattice spacings. From changes in these spacings, elastic strains can be derived, from which stresses can be calculated. By step-wise translation of a specimen or component across the reference point, stresses at different locations inside the specimen can be determined. In this clause the measurement process is summarized.

5.2 Outline of the principle — Bragg's law

When the lattice of a crystalline material is illuminated with penetrating neutron radiation with wavelength(s) similar to the interplanar spacings, this radiation will be diffracted as distinct Bragg peaks. The angle at which such a peak occurs is given by Bragg's law of diffraction, see Formula (1).

$$2d_{hkl}.\sin\theta_{hkl} = \lambda \tag{1}$$

where

λ is the wavelength of the neutrons;

d_{hkl} is the spacing of the (*hkl*) lattice planes responsible for the Bragg peak;

θ_{hkl} is the Bragg angle.

The peak will be observed at an angle $2\theta_{hkl}$ from the incident beam direction, as shown schematically in Figure 1.

5.3 Neutron sources

Neutron diffraction uses beams of neutrons generated by either fission or spallation; the former is predominantly employed in steady-state nuclear reactors and the latter in pulsed spallation sources.

In both cases the neutrons produced are moderated to bring their energies to the thermal range, i.e. $\lambda \geq 0{,}09$ nm. At reactor sources, a monochromatic neutron beam is usually extracted by using a crystal monochromator to select a given narrow neutron wavelength band from the originally polychromatic beam. At spallation sources, the neutron beam usually consists of a series of short pulses each containing a wavelength spectrum. The velocity (and therefore wavelength in accordance with the de Broglie principle) of each neutron can be determined by measuring the distance it has travelled to the detector and the time it has taken to travel this distance, called the TOF. TOF measurements are, therefore, wavelength dispersive, with the entire diffraction pattern being recorded at all available detector positions.

5.4 Strain determination

5.4.1 General

It is evident from Bragg's Law that if λ and θ are known experimentally, the lattice spacing, d, can be determined. We define the lattice strain as the relative change in d with respect to the stress-free lattice spacing, d_0. Thus, for the lattice planes identified by Miller Indices hkl, the lattice strain is given by Formula (2):

$$\varepsilon_{hkl} \equiv \frac{d_{hkl} - d_{0,hkl}}{d_{0,hkl}} = \frac{\Delta d_{hkl}}{d_{0,hkl}} \tag{2}$$

By virtue of the diffraction process, the measurement direction is along the scattering vector, $\vec{Q} = \vec{k}_{\mathrm{f}} - \vec{k}_{\mathrm{i}}$, which bisects the angle between incident and diffracted beams and is perpendicular to the diffracting planes as shown in Figure 1.

5.4.2 Monochromatic instrument

When a specimen is illuminated by a monochromatic neutron beam of a fixed wavelength, its lattice spacing can be determined from the observed Bragg angle, θ, by substituting Bragg's Law [see Formula (1)] into the strain equation [see Formula (2)]. Thus, elastic lattice strains can be determined directly from the corresponding Bragg angles, θ_{hkl}, and $\theta_{0,hkl}$, and given by Formula (3):

$$\varepsilon_{hkl} = \frac{\sin\theta_{0,hkl}}{\sin\theta_{hkl}} - 1 \tag{3}$$

5.4.3 TOF instrument

At a TOF instrument the incident beam contains neutrons spanning a range of velocities (i.e. wavelengths) arriving at the sample in pulses. In this case all lattice planes normal to the scattering vector will diffract neutrons to the detector. The diffraction corresponding to each hkl peak is produced by different families of grains. By using the de Broglie relationship and Bragg's Law [see Formula (1)] the TOF t_{hkl} for a particular wavelength, crystal plane and a detector positioned at angle 2θ, takes on the form as given in Formula (4):

$$t_{hkl} = 2\frac{m_{\mathrm{n}}}{h} \times L \times \sin\theta \times d_{hkl} \tag{4}$$

Solving for d_{hkl} and $d_{0,hkl}$ and substituting into the strain equation [Formula (2)] for a fixed angle 2θ, the elastic strain can be calculated from flight time change using Formula (5):

$$\varepsilon_{hkl} \equiv \frac{t_{hkl} - t_{0,hkl}}{t_{0,hkl}} = \frac{\lambda_{hkl} - \lambda_{0,hkl}}{\lambda_{0,hkl}} \tag{5}$$

or by averaging over all planes using a full pattern analysis such as the Rietveld refinement procedure[2] (see 7.3.2 and A.7.3).

5.5 Neutron diffractometers

A monochromatic instrument typically used for strain determination at a steady state source is shown schematically in Figure 2. The polychromatic neutron beam is first monochromated to a chosen wavelength by diffraction from a suitable monochromator. This monochromatic beam is spatially defined by appropriate beam-defining apertures to produce a beam of controlled dimensions. The neutrons in this beam are diffracted from the specimen volume illuminated and captured by a neutron detector. An example of a diffraction peak from a monochromatic instrument is shown in Figure 3.

At TOF diffractometers, typically used at pulsed sources, each pulse produces a diffraction profile across a range of lattice spacings. A typical TOF diffractometer used for strain determination in two directions simultaneously at a pulsed source is shown in Figure 4. As a fixed scattering angle is used, most instruments at spallation sources use radial collimation. This allows neutrons to be detected over a wider solid angle than would be possible using a slit, yet ensuring that most of the detected neutrons come from a defined gauge volume (see 7.5). The signals from the individual elements of the detector array are combined taking into account their different angular positions. Two or more detectors with radial collimators can be used to enable the simultaneous measurement of more than one $\vec{Q}$ (strain) direction. A typical diffraction pattern from such an instrument is shown in Figure 5, which also shows the result of a Rietveld profile refinement where a crystallographic model of the structure is fitted to the diffraction data using a least squares analysis (see 7.3.2).

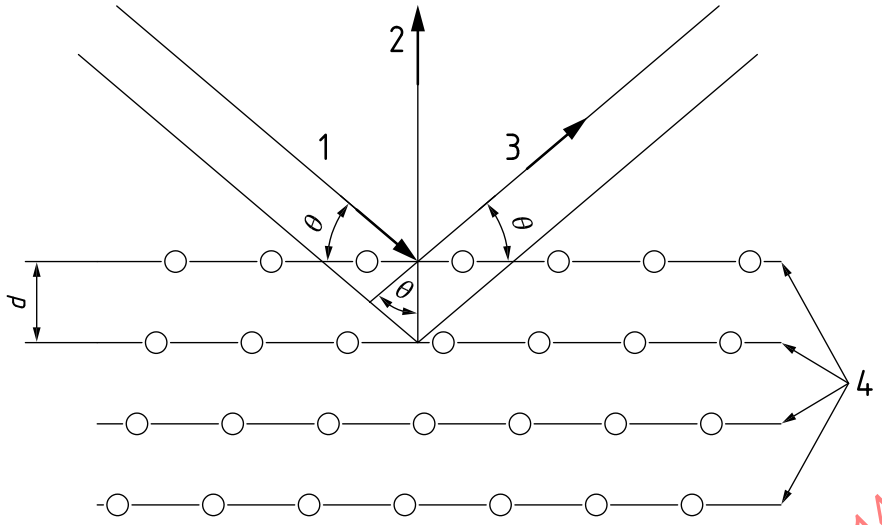
Additional information concerning the fitting of the diffraction data is provided in 9.5.

5.6 Stress determination

Stress and strain are second rank tensors that are related through a solid's elastic constants. Since by means of neutron diffraction elastic strains within a defined volume in a crystalline solid can be determined, it is possible to calculate the mean stress in that volume provided that the relevant material elastic constants are known. Deriving the full strain tensor requires the determination of elastic strains in at least six independent directions. If the principal strain directions within the body are known, measurements along these three orthogonal directions are sufficient to determine the principal stresses. For plane stress or plane strain conditions, a further reduction to two directions is possible. Measurement along one direction is sufficient in the case of uni-axial loading. Clause 9 and A.9 provide further details on the calculation of stresses from derived strains.

Stresses and strains in a specimen are usually direction and position dependent. This leads to the need to obtain strains at a number of locations in more than one direction. This in turn requires the accurate positioning of the specimen with respect to the collimated neutron beam and the detectors. This is usually accomplished by mounting the specimen on linear translation and rotation tables.

By a step-wise movement of the specimen in relation to the volume in the space identified by the intersection of the incident and diffracted beams (termed a gauge volume, see 7.5), the spatial variation in elastic strain and, following measurements in other directions, stress can be mapped within a specimen or component.



Key

1 incident wave vector $\vec{k}_i$

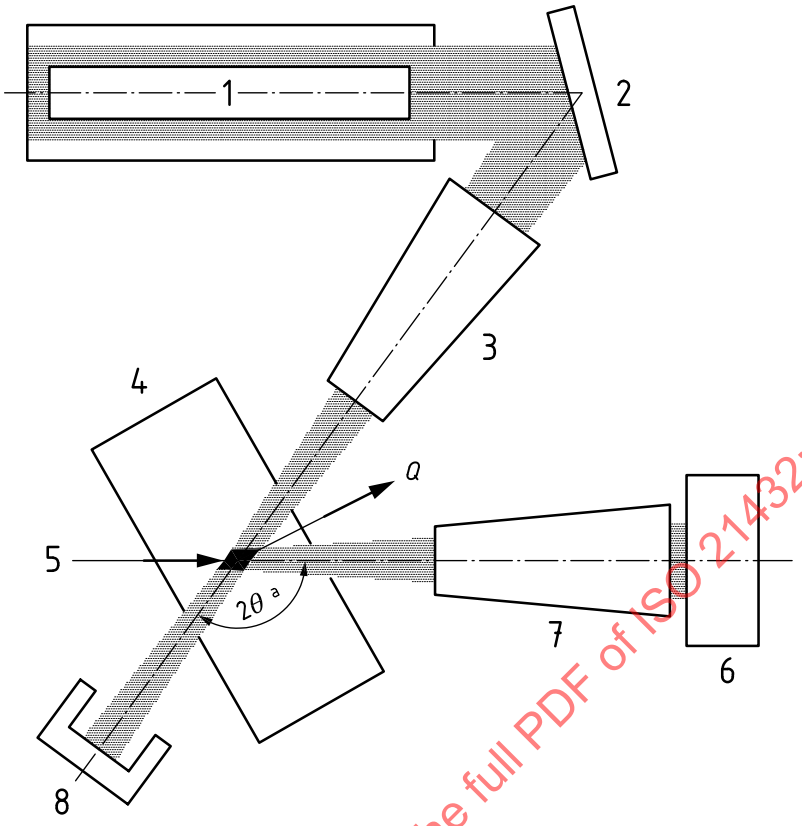
2 scattering vector $\vec{Q}$

3 diffracted wave vector $\vec{k}_f$

4 diffracting planes of spacing d

θ one-half of the diffraction angle

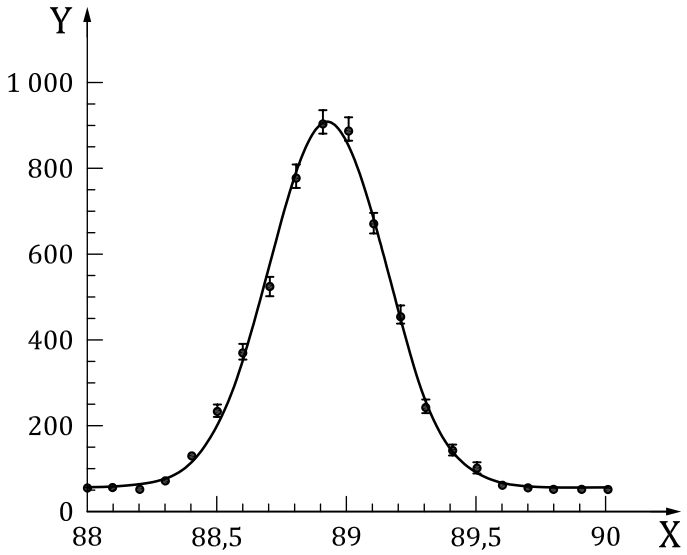
Figure 1 — The Bragg diffraction geometry



Key

1 neutron beam from the source
2 monochromator
3 beam-defining optics for the incident beam and shielding
4 specimen
5 gauge volume
6 detector
7 beam-defining optics for the diffracted beam and shielding
8 beam stop
Q scattering vector
2θ [a] scattering angle

Figure 2 — Schematic illustration of a steady state source based diffractometer for elastic strain determination

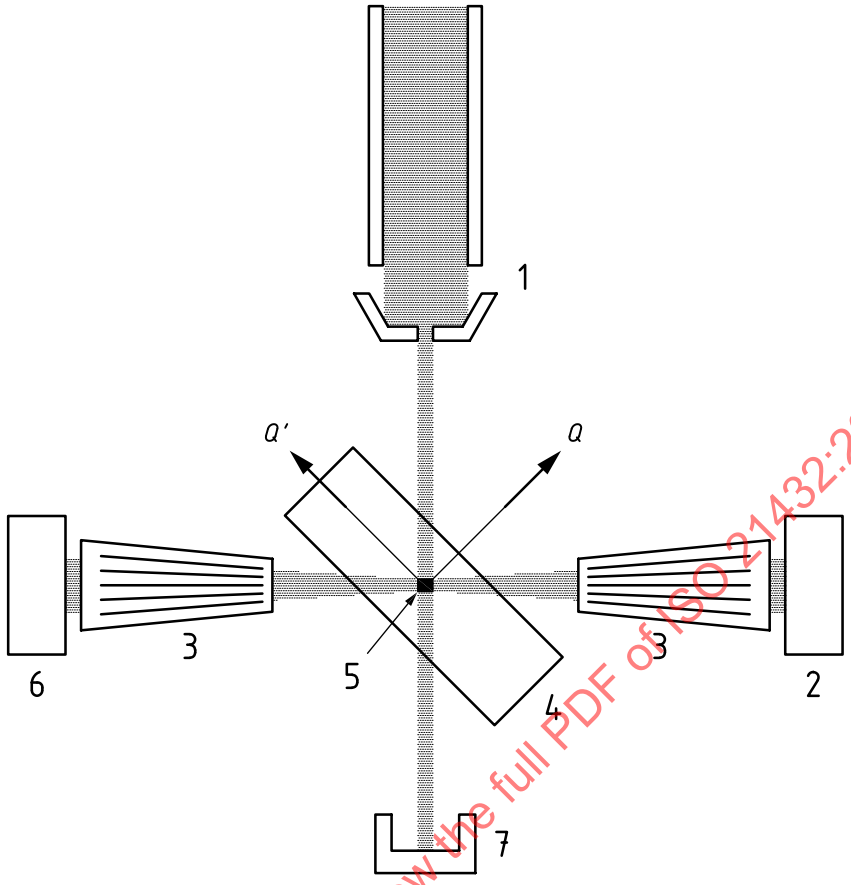


Key

X 2θ, degrees

Y neutron counts

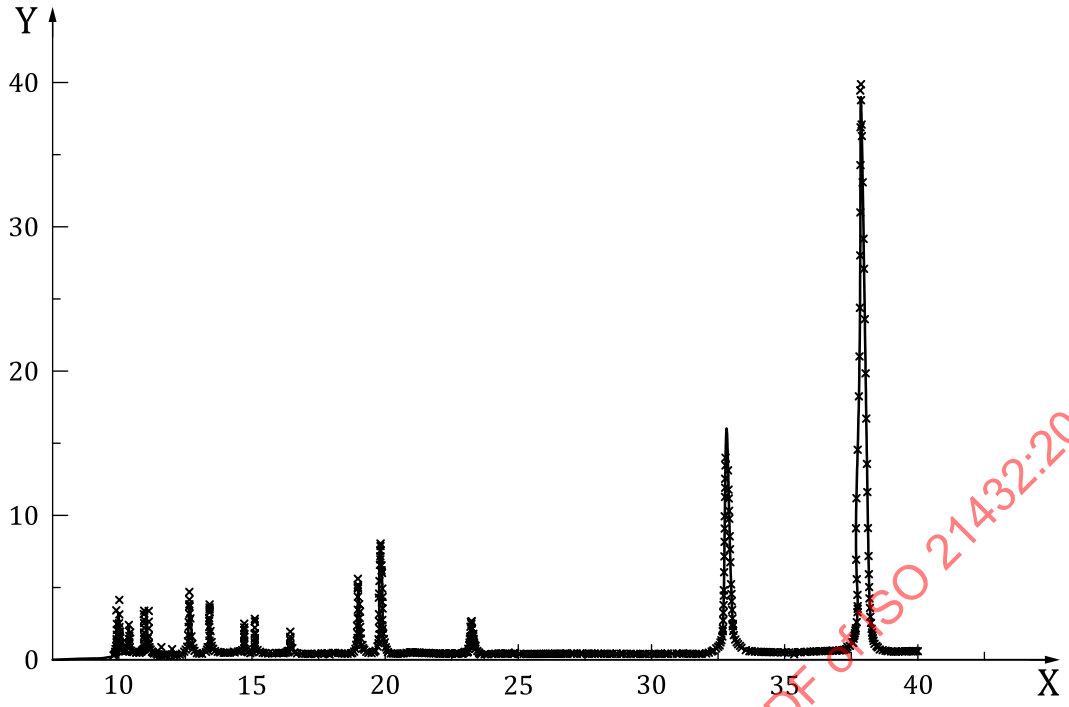
Figure 3 — Example of a Bragg peak from a reactor (steady state source) based diffractometer fitted with a Gaussian distribution



Key

Q	scattering vector, right detector
Q'	scattering vector, left detector
1	neutron beam from the source
2	right detector
3	radial collimator
4	specimen
5	gauge volume
6	left detector
7	beam-stop

Figure 4 — Schematic illustration of a pulsed-source TOF diffractometer for elastic strain determination



Key

X time-of-flight, ms
Y intensity
× data
— Rietveld profile fit

Figure 5 — Example of a diffraction pattern from a pulsed source

6 Purpose, geometry and material

6.1 General

The design of an experiment depends upon the purpose of the measurement, the dimensions and shape of the specimen, the materials from which it is made and how it has been fabricated and used.

6.2 Purpose of the measurement

The purpose and scope of the measurements shall be defined as this determines many decisions that have to be made.

6.3 Geometry

The as-made dimensions and shape of the specimen shall be known and considered in the preparation of the measurement.

6.4 Composition

Standard material designations that indicate the chemical composition and processing route shall be used to enable appropriate experimental conditions to be chosen. Furthermore, for multiphase materials, including composites, the chemical composition, fraction, orientation and morphology of each phase shall also be considered for their influence on the stress determination.

6.5 Thermal/mechanical history

The processing route used to shape, form or join the specimen, including heat treatment, shall be considered in designing the experiment. In the case of parts removed from service use, the previous operating conditions may also be relevant.

6.6 Phases and crystal structures

The phases in the material to investigate shall be known. The crystallographic structure of phases used in the measurements shall be known.

6.7 Homogeneity

Information about any spatial variation in the composition, microstructure or phase distribution is relevant to the experiment. This may affect the confidence in the measurements obtained at a particular location in a specimen or component and whether it is valid in taking the results to be representative of the specimen or component as a whole. In particular, inhomogeneities in the microstructure and composition can lead to variations in the stress-free lattice spacing with position in the specimen or component (see 7.6).

6.8 Microstructure

Large dimensions of grains or composite reinforcements can result in point-to-point fluctuations in diffraction peak intensities, which may indicate that an insufficient number of grains are being sampled. Consequently the grain size in relation to the gauge volume employed and to the stress distributions shall be established in such cases.

6.9 Texture

The presence of crystallographic texture affects the diffraction peak intensity and the conversion of strain to stress. If the material is known to possess a texture, as a result of processing or use, it should be characterised and possible implications reported.

7 Preparations for measurements

7.1 General

Prior to an actual measurement it is necessary to carry out or verify the instrument alignment. Then appropriate conditions for the diffraction measurement shall be chosen and the specimen shall be accurately positioned on the diffraction instrument. Also the dimensions of the volume from which the diffraction data will be collected shall be chosen.

7.2 Alignment and calibration of the instrument

It is necessary to align and good practice to calibrate the diffractometer being used (see A.5.2). When using a monochromatic beam instrument, it shall be ensured that a constant wavelength is maintained throughout the entire set of measurements and that the detector angular response has been calibrated in accordance with EN 13925-3:2015, Annex C. At a TOF-diffractometer, both the flight path and detector angular response should be calibrated. For both types of instrument, calibration is performed using a standard specimen typically silicon, ceria or alumina powders. Such specimens are chosen because they diffract neutrons strongly, have known and well-defined lattice parameters and have small intrinsic peak widths. If intensity information is required at a TOF instrument it is necessary to determine the incident neutron flux and the detector efficiency as a function of the wavelength. One way of doing this is to use an incoherent scatterer, such as vanadium.

7.3 Choice of diffraction conditions

7.3.1 Monochromatic instruments

7.3.1.1 Choice of the wavelength

At monochromatic instruments the user shall choose the neutron wavelength for a particular experiment from the range of wavelengths available. The wavelength and diffraction plane should be selected such that an efficient execution of the experiment is achieved for a diffraction angle near 90°. However, if the chosen wavelength is close to twice the *d*–spacing of any diffraction plane in the specimen, "Bragg edge" related spectrum distortion can occur that can cause peak shifts, which can be falsely interpreted as strains. These "problematic" wavelengths have been tabulated in Reference [3] for several common metals. For cubic materials, in particular, diffraction angles too close to 90° should be avoided since for all (*hkl*) diffraction planes there is an alternate (*h'k'l'*) which can cause a Bragg edge related effect.

The efficiency with which a measurement can be performed depends on parameters such as spatial resolution, incident beam intensity at the chosen wavelength, diffracted neutron intensity, peak width and separation of the peak under investigation from adjacent peaks. With respect to these factors a diffraction angle quite different from 90° can be more efficient than one close to 90°.

7.3.1.2 Choice of the diffracting lattice plane

In the presence of elastic and plastic anisotropy in a material, different (*hkl*) planes may exhibit different responses to a macroscopic stress field[4]. This can be demonstrated by loading and unloading a tensile bar, in situ, in a neutron diffractometer whilst measurements of the stress and strain are recorded, as indicated in Figures 6 and 7. In these figures, the stress recorded by a load cell in series with a test bar is plotted against the elastic strain derived from neutron diffraction measurements.

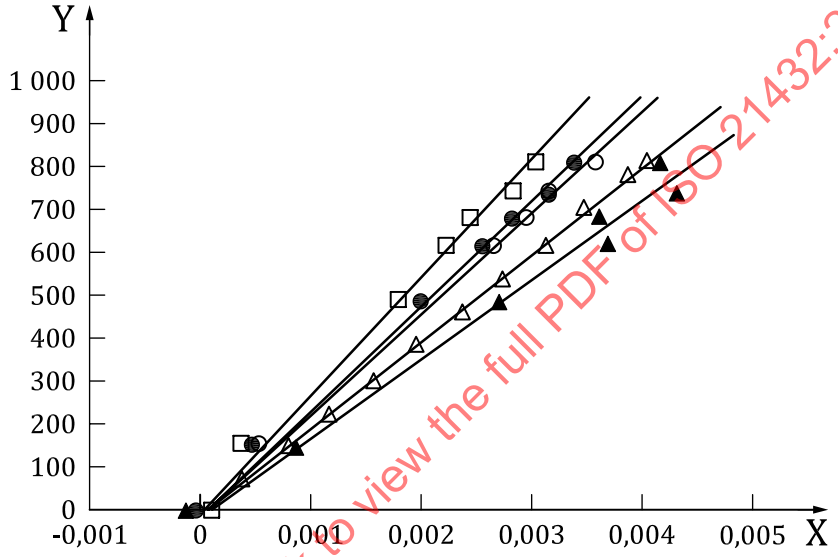
Figure 6 shows that a linear response is obtained in the elastic region irrespective of the set of lattice planes used to make the measurements. This demonstrates that any (*hkl*) reflection can be employed for determining the stress in this region, provided that the appropriate diffraction elastic constants are chosen. Generally, these are neither the macroscopic elastic constants nor the single crystal values, but a polycrystalline aggregate value associated with a particular (*hkl*) plane. These constants can be obtained either experimentally as Figure 6 demonstrates, or calculated (see Clause 9). The calculation methods include the Reuss[5], Voigt[6], Neerfeld-Hill[7][8] and self-consistent methods, e.g. Kröner[9]. Normally the Neerfeld-Hill method provides reliable approximations and is much simpler to implement than the self-consistent approaches. Regardless of the method used, the crystallographic texture of the specimen shall be taken into account. See references [10] and [11] for discussions on the importance of texture.

Plastic deformation begins at different stresses recorded by the load cell in differently oriented grains, as illustrated in Figure 7. This is demonstrated by a non-linear response on loading followed by linear elastic unloading. The consequence is that a different residual elastic strain may be observed for each (*hkl*) plane upon unloading to zero applied stress. These strains are usually called intergranular strains. After complete unloading of the test bar, the engineering (macroscopic) residual stress shall be zero to satisfy equilibrium conditions. The stress will be calculated from the observed residual strains at zero applied load. However, this can be a superposition of Type I and Type II stress. Therefore, if Type I stresses are to be determined, it is important to use crystallographic planes that do not bear significant Type II (intergranular) strain [e.g. plane (220) or (311) in Figure 7].

In some cases it is necessary and appropriate to employ (*hkl*) planes that are sensitive to intergranular strains. In such cases compensations shall be made for the intergranular strains. One suitable approach is to obtain the d_0 value from coupons that are taken from the specimen under investigation and are sufficiently small not to contain macrostresses[12][13]. Examples of (*hkl*) planes with high and low sensitivity to intergranular strains for a range of materials are listed in Table 1. These data are based on intergranular strains developed in plastically deformed uniaxial loading specimens. In general, the deformation history of a particular material or component may be more complicated.

Table 1 — Examples of planes exhibiting high and low sensitivity to intergranular strains for materials of different symmetry

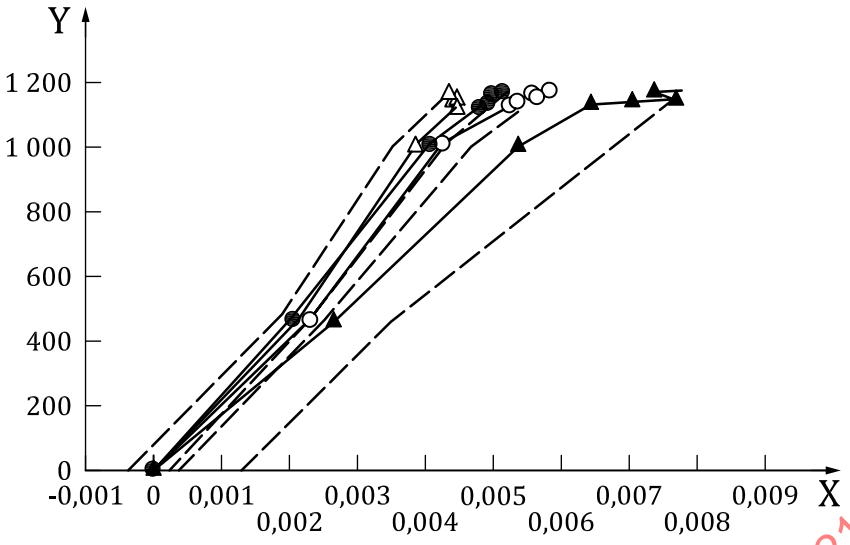
Material	Planes with low sensitivity to intergranular strains	Planes with high sensitivity to intergranular strains
fcc (Ni[14], Fe[15], Cu[16]), fcc (Al[17][18]), Ni[4]	(111), (311), (422)	(200)
bcc (Fe[18])	(110), (211)	(200)
hcp (zircaloy[19], Ti[20])	$(10\bar{1}2)$, $(10\bar{1}3)$ (Pyramidal)	(0002) (basal) $(10\bar{1}0)$, $(1\bar{2}10)$ (prism)
hcp (Be[21])	$(20\bar{2}1)$, $(11\bar{2}2)$ (2^{nd} order pyramidal)	$(10\bar{1}2)$, $(10\bar{1}3)$ (basal, prism and 1^{st} order pyramidal)



Key

□ (111) planes
▲ (200) planes
○ (311) planes
● profile refinement
△ load cell indication
X recorded lattice strain
Y applied stress (MPa)

Figure 6 — Elastic response of different crystallographic planes for nickel alloy[4]



Key

—▲—	(200) planes
—○—	(311) planes
—△—	(220) planes
—●—	profile refinement
– – –	unload
X	recorded lattice strain
Y	applied stress (MPa)

Figure 7 — Effect of yielding on the response of different crystallographic planes to the loading and unloading of a tensile bar of a nickel alloy [4]

7.3.2 TOF instruments

When a full pattern analysis such as the Rietveld refinement procedure[2] is used, the strain is obtained from changes in the lattice parameters defining the unit cell dimensions.

For cubic materials with lattice parameter a_0, the strain is given by Formula (6):

$$\varepsilon = \frac{a - a_0}{a_0} \tag{6}$$

where

ε is the strain;

a is the lattice parameter value under strain obtained from the full pattern analysis [it replaces the lattice spacing d in Formula (2)];

a_0 is the stress-free lattice parameter.

For non-cubic materials it is necessary to identify a suitable strain parameter, e.g. in hexagonal materials free of texture, an appropriate expression for the strain is given in Formula (7):

$$\varepsilon = \frac{2\varepsilon_a + \varepsilon_c}{3} \tag{7}$$

where

ε is the strain;

ε_a is the strain determined from the a lattice parameter in the same way as in Formula (6)[21];

ε_c is the strain determined from the c lattice parameter in the same way as in Formula (6)[21].

7.4 Positioning procedures

The initial alignment procedure requires the determination of the location of the centroid of the IGV, see 7.5 and Figure 9. This location is defined as the reference point to which all measurements are referred. It should ideally coincide with the centre of rotation of the specimen table.

An accurate specimen positioning is required, as described in A.3. The level of accuracy required depends to some extent on the objectives of the measurements to be made. Positioning accuracy is most important in the case of large strain gradients and where measurements are made close to surfaces or interfaces. It is important that the uncertainty in positioning is declared.

Alignment can be carried out for example by optical or mechanical means, or by using surface scans (see A.3.4). All three methods can determine the position of a specimen edge relative to the reference point to an uncertainty of 0,1 mm.

Experiment planning and control software that model the sample and the instrument exist[22]. When such systems are used in concert with data acquisition, the resulting uncertainty in positioning — which is a function of the instrument, the model and the computation — shall be established and reported.

7.5 Gauge volumes

The NGV is defined as that volume of space that is occupied by the intersection of parallel beams of neutrons, which are transmitted through the defining apertures (e.g. slits, collimators) for both the incident and diffracted neutrons (Figure 8). The centroid of the NGV is the geometric centre of this volume.

For a system which incorporates radial collimators the concept is identical, except that each radial collimator slit contributes to the NGV.

The IGV is the volume of space defined by the actual neutron beam paths through the defining apertures, taking into account the beam divergence and the beam intensity profile (Figure 9). Methods of determining the IGV by probing it with a scattering element are presented in A.5.1. The IGV dimensions can also be defined in terms of the FWHM of the beam intensity profile. Whatever practice is adopted shall be specified.

The difference between the IGV and the NGV may be particularly evident when small volumes are being sampled due to a relatively more significant contribution from the penumbra. Note that the IGV and NGV are properties of the diffractometer itself.

Finally, the SGV is the intersection of the IGV with the specimen phase under investigation (see Figure 10). That is, a spatially dependent sub-volume weighted by the scattering of the phase investigated. Consequently it is the volume over which the average strain is obtained.

Additionally, the centroid of the SGV (i.e. the intensity-weighted centre of the scattering) can be at a different position from that of the IGV as shown in Figure 10 due to several factors:

— partial filling of the IGV with the specimen phase under investigation;

— attenuation of the neutron beam within the specimen;

— wavelength and intensity distribution in the neutron beam.

The SGV and its centroid should be determined for each measurement. It is important that the derived strain be reported at this position.

The effect of the weighting can become significant in cases such as at surfaces and interfaces, in highly attenuating materials, large grained materials, and at texture or microstructure gradients. The asymmetric presence of a strong incoherent scatterer can also affect results.

The consequences of the SGV centroid being offset from the reference point are discussed in A.5.5 and A.6. Additional information can be found in Reference [23].

7.6 Methods for establishing the macroscopically stress-free or reference lattice spacing

Since diffraction measurements allow the determination of lattice spacings (see 5.4 and 7.3.2), in order to determine elastic strains it is necessary to obtain a reference value, relative to which the strains can be determined. In some cases it is possible to determine a stress-free lattice spacing d_0. In other cases only a reference lattice spacing d_{ref} (the lattice spacing to which other measurements will be compared) is possible. It should be noted that actual values of stress can only be determined when strains are calculated relative to d_0. The use of d_{ref} should only be made when values of d_0 are not available.

Lattice plane spacings may be influenced by a number of causes apart from the stress. The most important of these are chemical composition and temperature. The optimum method of determining d_0 (or d_{ref}) depends on the particular problem under consideration.

In some cases it may be sufficient to use a single stress-free or reference value. In other cases it may be necessary to use spatially and/or directionally dependent values[24].

When a single value can be used it may be determined by:

— measurement in a material at a position known to contain a negligible stress;

— measurement on a powder, which is representative of the material being examined. This is particularly suitable for multiphase materials.

In the case of spatial and directional variations in d_0, these values can be obtained by:

— measurement on small coupons, cut from large blocks of material. This is particularly relevant to welds, since the use of multiple coupons allows the determination of spatial and directional variations in d_0 through a weldment[12][13][19].

Other approaches include:

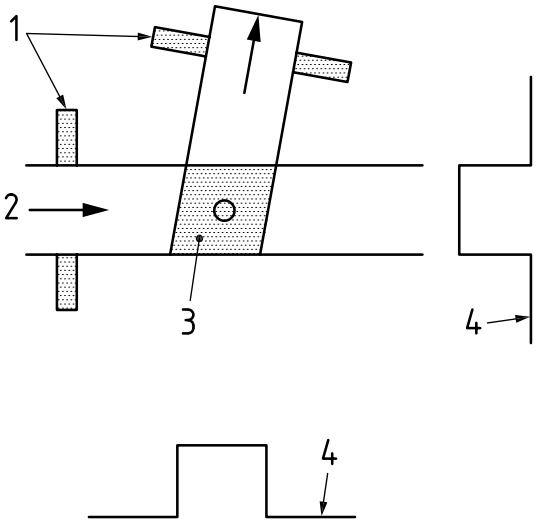
— determination of d_0 by imposing force and moment equilibrium (noting that experimental methods should be used wherever possible, and that equilibrium should be employed mainly for validation purposes), and

— determination of d_0 by ensuring zero stress perpendicular to a free surface. This is only suitable when there is no variation in d_0 away from the surface and when an accurate determination of near surface strains is possible.

It is good practice to confirm that measured stress results satisfy force and moment equilibrium across a section of a component. This requires that sufficient measurements across an appropriate section have been made.

During the preparation of a "stress-free" sample of material, care shall be taken to avoid the introduction of residual stresses or modification of the microstructure or of the chemical composition.

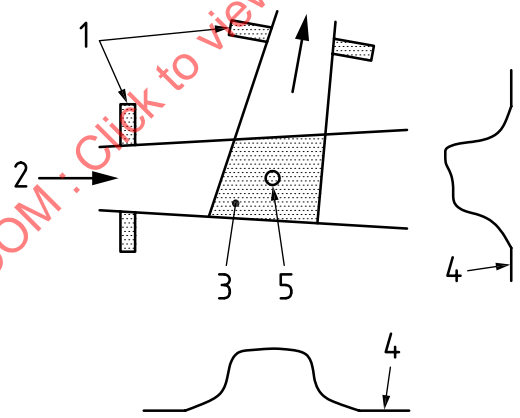
It is necessary to centre the reference sample accurately at the IGV centroid.



Key

1 apertures
2 incident neutrons
3 NGV
4 neutron intensity profile
○ centroid of the NGV

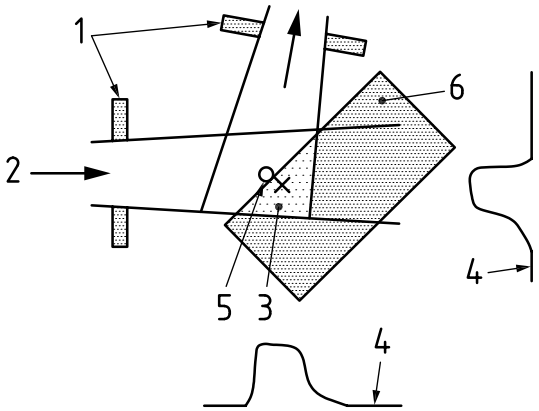
Figure 8 — Plan view of the nominal gauge volume.



Key

1 apertures
2 incident neutrons
3 IGV
4 neutron intensity profile
5 reference point (centroid of the IGV)

Figure 9 — Plan view of the instrumental gauge volume



Key

1 apertures
2 incident neutrons
3 SGV
4 neutron intensity profile
5 reference point (centroid of the IGV)
6 specimen
× centroid of the SGV

Figure 10 — Plan view of the sampled gauge volume.

8 Measurement and recording requirements

8.1 General

Three parameters shall be determined with appropriate accuracy:

a) the strain,

b) the direction of the strain component, and

c) the position in the specimen at which the measurement is made.

Sufficient information shall be recorded such that the experimental approach and data analysis can be understood, evaluated and reproduced. Measurement and analysis methodologies as well as experimental considerations are presented in Annex A. Discussion of the determination of uncertainties in a measurand is provided in Annex B.

8.2 Recording requirements

8.2.1 General

In general, the project title, persons involved in the investigation and the dates of the measurements shall be provided together with the following information.

8.2.2 General information — instrument

Instrument related information:

a) individuals responsible for the instrument;

b) neutron source and location, name and type of instrument;

c) temperature ± variation;

d) optics components in incident and diffracted beams; for slits, height, width and distance to the reference point shall be specified; for radial collimators, focal length, foil length and thickness, angle between foils, all aperture dimensions and collimator oscillation parameters shall be quoted.

Parameters for monochromatic instruments:

a) the type of monochromator, its crystal and reflection used, the type of detector, the distance from the monochromator to the reference point, the distance from the detector to the reference point;

b) the wavelength and how it was determined;

c) the vertical and horizontal gauge intensity profile if critical to the measurement;

d) the resolution of the detector.

Parameters for TOF instruments:

a) the total flight path length L, the distance from the detector to the reference point, the type of detector, the angular range of the detector;

b) the wavelength range and how it was determined;

c) the vertical and horizontal gauge intensity profile if critical to the measurement;

d) the number of Bragg peaks used or d-spacing range used in the analysis of data;

e) the time resolution or channel width;

f) the incident intensity as a function of the wavelength.

8.2.3 General information — specimen

Specimen related information:

a) specimen material, chemical composition, crystal structure;

b) diagram of the specimen showing dimensions, fiducial marks or reference locations and specimen co-ordinates.

8.2.4 Specific information required for each diffraction measurement

All original data shall be recorded and available. The methods by which the data have been processed shall also be recorded and available.

Information related to specific measurements:

For monochromatic instruments:

a) the peak position $2\theta_{hkl}$ ± uncertainty;

b) the peak position for the lattice planes in the stress-free condition $2\theta_{0,hkl}$ (or reference peak position $2\theta_{\mathrm{ref},hkl}$) ± uncertainty.

For TOF instruments:

a) the times of flight t_{hkl} or lattice parameter(s) in the case of full pattern analysis ± uncertainty;

b) the times of flight for the lattice planes in the stress-free condition, $t_{0,hkl}$ (or reference times of flight $t_{\mathrm{ref},hkl}$) or reference lattice parameter(s) in the case of full pattern analysis ± uncertainty.

For any type of instrument:

a) the specimen orientation relative to the scattering vector $\vec{Q}$ ± uncertainty;

b) the specimen and the gauge volume positions relative to the reference point ± uncertainty;

c) the strain ± uncertainty;

d) the *d*-spacing measurement ± uncertainty (if absolute values are required).

For single peak analysis:

a) the angle or time increment;

b) the peak profile function used and the parameter values obtained, including:

   1) the FWHM ± uncertainty;

   2) the peak height H or integrated intensity I ± uncertainty;

   3) the background B ± uncertainty.

For multiple peak fits or full pattern analysis (e.g. Rietveld refinement):

a) the peak profile(s) used and relevant parameters including:

   1) the width, as a function of the wavelength or diffraction angle;

   2) the peak profile asymmetry;

b) the background fit used;

c) a description of how texture, elastic and plastic anisotropy are taken into account.

8.3 Specimen co-ordinates

The co-ordinate system used to define the location and direction within a specimen shall be clearly specified and shall relate to the shape of the specimen and/or to the principal stress directions, if known.

NOTE For most applications on regular-shaped specimens or components, rectangular or polar co-ordinates aligned with respect to symmetry features are appropriate.

8.4 Positioning of the specimen

The position of a specimen shall be defined relative to the instrument reference point (see 7.4) The orientation of the specimen co-ordinate system shall be defined in relation to the co-ordinate system used to define $\vec{Q}$. The reference point position shall be defined as accurately as is practicable. Details are given in A.3.

8.5 Measurement directions

Measurements along at least six independent directions are generally required in order to determine the strain/stress tensor. Nevertheless, three measurements along any three mutually orthogonal co-ordinate axes (e.g. the specimen co-ordinate system) yield the respective normal components of the stress tensor. Therefore important information can be obtained without knowing the principal stress directions and without making measurements in more than three independent orientations (see 5.6).

8.6 Number and location of measuring positions

The number and locations of measurements shall be related to the strain detail that is required, to the shape and dimensions of features of interest of the strain profile and to the size of the gauge volume used.

For test locations that require a long neutron path length within the specimen, it may be necessary to remove some material to make the measurement possible.

Care shall be taken in such cases because material removal results in a redistribution of the stresses inside the specimen, which can affect the stress at the location of interest.

More details are given in A.4.

8.7 Gauge volume

Gauge volumes are defined by appropriate beam-defining optics in the incident and diffracted beams, and the directions and divergences of those beams. The choice of gauge volume dimensions shall relate to the shape and the dimensions of features of interest of the strain profile and to material parameters such as grain size and attenuation lengths. Details are given in A.5.

8.8 Gauge volume centroid considerations

The SGV centroid position shall be determined taking into account instrumental aberrations and attenuation. Special attention is required when scanning through surfaces or interfaces. Details are given in A.6.

8.9 Temperature

The specimen temperature shall be monitored and controlled such that changes in lattice dimensions either are small relative to the uncertainty specified for the obtained strain or can be accounted for. Details are given in A.8.

9 Calculation of stress

9.1 General

With neutron diffraction elastic strains can be determined and stresses calculated from these strains. Only normal strains can be derived from the measured data; shear strains can be calculated, if needed.

Essentially all diffraction investigations of stresses and strains are based on continuum mechanics using Hooke's law for stress calculations. As discussed in 7.3, the only major alteration is the use of specific diffraction elastic constants rather than the overall aggregate average. Hence, the average elastic constants in the generalized Hooke's law are simply exchanged with the appropriate diffraction elastic constants (E_{hkl}, ν_{hkl}). When these diffraction elastic constants are calculated from the single crystal elastic constants C_{ij} using suitable models or measured by means of in-situ uniaxial tests, care shall be taken. The procedure for calculating stresses in isotropic materials is described in 9.2 to 9.4.

9.2 Normal stress determinations

The normal stresses at a point can be determined from strain components along three mutually orthogonal co-ordinate axes, x, y and z, at that point. In this case the stresses are given in Formulae (8), (9) and (10):

$$\sigma_{xx} = \frac{E_{hkl}}{(1+\nu_{hkl})(1-2\nu_{hkl})}\left[(1-\nu_{hkl})\varepsilon_{xx} + \nu_{hkl}(\varepsilon_{yy}+\varepsilon_{zz})\right] \tag{8}$$

$$\sigma_{yy} = \frac{E_{hkl}}{(1+\nu_{hkl})(1-2\nu_{hkl})}\left[(1-\nu_{hkl})\varepsilon_{yy} + \nu_{hkl}(\varepsilon_{xx}+\varepsilon_{zz})\right] \tag{9}$$

$$\sigma_{zz} = \frac{E_{hkl}}{(1+\nu_{hkl})(1-2\nu_{hkl})}\left[(1-\nu_{hkl})\varepsilon_{zz} + \nu_{hkl}(\varepsilon_{xx}+\varepsilon_{yy})\right] \tag{10}$$

where the indices

xx, yy, and zz indicate the co-ordinate axes, along which the normal strains and stresses are obtained.

When the co-ordinate axes are coincident with the principal directions of deformation, these stresses are the principal stresses.

For plane stress conditions, where one of these stresses (say σ_{zz}) is zero these formulae reduce to Formulae (11) and (12):

$$\sigma_{xx} = \frac{E_{hkl}}{(1-\nu_{hkl}^2)}[\varepsilon_{xx} + \nu\varepsilon_{yy}] \tag{11}$$

$$\sigma_{yy} = \frac{E_{hkl}}{(1-\nu_{hkl}^2)}[\varepsilon_{yy} + \nu\varepsilon_{xx}] \tag{12}$$

For plane strain conditions with $\varepsilon_{zz} = 0$, the corresponding expressions for σ_{xx} and σ_{yy} are obtained by substituting $\varepsilon_{zz} = 0$ in Formulae (8), (9) and (10), and σ_{zz} becomes as given in Formula (13):

$$\sigma_{zz} = \nu_{hkl}(\sigma_{xx} + \sigma_{yy}) \tag{13}$$

For the case of strong macroscopic anisotropy see A.9.

9.3 Stress state determinations

9.3.1 General

When the principal directions of the stress state are not known, determination of strain in at least six independent orientations is in general needed to identify the complete strain state at a given location. The full stress tensor components can be derived from these normal strains $\varepsilon_{\varphi\psi}$ using Formula (14):

$$\varepsilon_{\Phi\Psi} = \left(\frac{1+\nu_{hkl}}{E_{hkl}}\right)\left[\begin{array}{l}(\sigma_{xx}\cos^2\Phi + \sigma_{yy}\sin^2\Phi + \sigma_{xy}\sin 2\Phi)\sin^2\Psi \\ +\sigma_{xz}\cos\Phi\sin 2\Psi + \sigma_{yz}\sin\Phi\sin 2\Psi + \sigma_{zz}\cos^2\Psi\end{array}\right] \\ -\frac{\nu_{hkl}}{E_{hkl}}\left[\sigma_{xx}+\sigma_{yy}+\sigma_{zz}\right] \tag{14}$$

where

Φ and Ψ are the orientation angles (see Figure 11), and

$\varepsilon_{\Phi\Psi}$ is the normal strain in the direction defined by the orientation angles Φ and Ψ.

In Formula (14) subscripts *xy*, *xz* and *yz* correspond to the shear components of stress.

It is desirable to choose the six measurement directions so that they are oriented by the largest angular separations possible.

9.3.2 The $\sin^2\psi$ method

When one of the principal directions is known at the location of interest within the specimen, e.g. *z*, the $\sin^2\psi$ method may be applied. In such a case the shear stresses σ_{xz} and σ_{yz} are equal to zero, and Formula (14) may be simplified. In this instance, it is possible to calculate the difference between the

normal stress in a given direction in the x-y plane, σ_{Φ}, and σ_{zz} from Formula (15), which is the simplified form of Formula (14):

$$\varepsilon_{\Phi\Psi} = \frac{1+\nu_{hkl}}{E_{hkl}}(\sigma_{\Phi} - \sigma_{zz})\sin^2\Psi - \frac{\nu_{hkl}}{E_{hkl}}(\sigma_{xx} + \sigma_{yy} + \sigma_{zz}) + \frac{1+\nu_{hkl}}{E_{hkl}}\sigma_{zz} \quad (15)$$

since

$$\sigma_{\Phi} = \sigma_{xx}\cos^2\Phi + \sigma_{yy}\sin^2\Phi + \sigma_{xy}\sin 2\Phi \quad (16)$$

Since in Formula (15), the desired value, $(\sigma_{\Phi}-\sigma_{zz})$, is a linear function of $\sin^2\psi$, this approach is frequently called "the $\sin^2\psi$ method" and is commonly used in stress determination in conventional X-ray diffraction[25]. The wider the range of ψ the more reliable the stress determination. This method is useful when it is not possible to measure in certain directions.

9.4 Choice of elastic constants

The diffraction elastic constants, E_{hkl}, ν_{hkl}, are required by Formula (8) to Formula (15). As explained in 7.3, it is possible only in special cases to use the "macroscopic" values of the elasticity modulus and Poisson's ratio, which have been determined by usual mechanical methods. This is because the values of the diffraction elastic constants are likely to depend on the chemical composition and the presence and quantities of other phases and/or lattice defects (e.g. dislocations after plastic deformation). Therefore, preferably, values obtained from diffraction experiments during uniaxial loading should be used[4]. In the case of crystallographic texture, the diffraction elastic constants are orientation dependent and such experiments shall be performed in a sufficient number of directions. If no experimental data can be made available, estimates should be obtained based on appropriate models, see References [3] to [7] and 7.3.

If the required diffraction elastic constants are not available for the specimen material, they may be determined by a uniaxial loading experiment. Since the chemical composition, phase volume fractions, texture, microstructure and even temperature can have an influence on the elastic properties, care shall be taken so that the specimen used in the uniaxial loading test and the specimen under investigation have comparable material characteristics. For the same reason, comparable experimental conditions should be applied in both tests. It is also recommended to use identical data evaluation procedures (e.g. single peak analysis).

The "macroscopic" values of E and ν can be used for cubic and hexagonal phases in texture-free specimens, if the elastic strain is determined by a full pattern analysis.

In the case of a specimen containing texture, modification of these estimates may be needed. The stronger and sharper the texture, the more required is the modification of the elastic constants. It is possible that texture can be taken into account for all crystal systems by measuring the so-called orientation distribution function (ODF) and by introducing it into the calculation of the diffraction elastic constants[10].

9.5 Diffraction data analysis

9.5.1 General

The position of a Bragg peak is determined by fitting a suitable mathematical function to the experimental data. This function simulates the peak shape of the diffracted spectrum including the background. It shall be borne in mind that the accuracy of strain determination can be compromised by an improper consideration of the items listed below and in the Annexes.

9.5.2 Peak fitting function

When a monochromatic beam is used the peak position is normally determined through single peak analysis by fitting a Gaussian function to the data.

At spallation sources, the peak profile is intrinsically asymmetric. The peak fitting function is normally a convolution of an exponential decay function and a Voigt function.

When a multi-peak spectrum is obtained, a full pattern analysis, such as a Rietveld refinement[2], can be used to extract strains (see A.7.3).

9.5.3 Background function

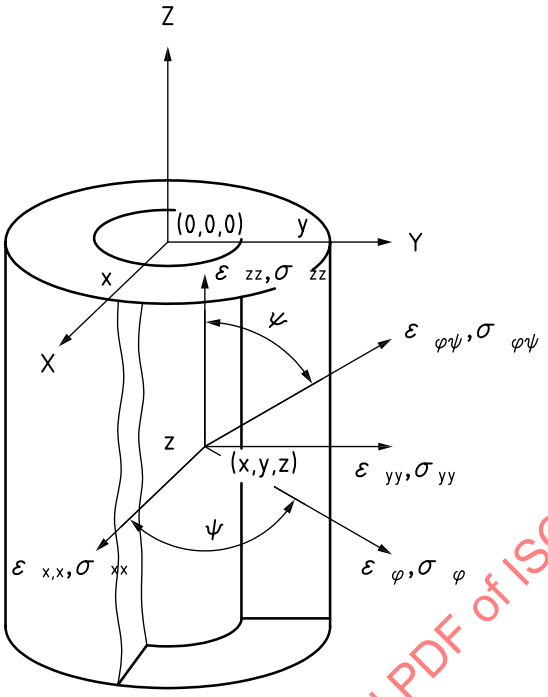
The function used to fit the background depends on the instrumental set-up and the types of neutron source. Because the slope of a background that varies as a function of the diffraction angle or TOF, and the peak position may be interdependent, care shall be taken in such cases. Unless the background can be determined independently of the peak profile, a fixed gradient should be used. If the background is not constant, the fitting function and its parameters should be stated.

9.5.4 Peak to background ratio

As the ratio of the peak height H to the background B decreases, it becomes more difficult to separate the peak position from effects caused by fitting the background, particularly if the background is not constant.

9.5.5 Distorted peak profiles

Unless proper corrections can be made, reflections with peak profiles that are distorted due to peak overlap or sample effects, such as material inhomogeneities and stacking faults, or due to instrumental effects, should be treated with caution. In the study of multiphase materials, overlapping profiles are sometimes unavoidable. Multiple peak fitting strategies can be used for the analysis based on the procedures described in A.7.



Key

ε_{xx}	normal strain in the direction of X
σ_{xx}	normal stress in the direction of X
ε_{yy}	normal strain in the direction of Y
σ_{yy}	normal stress in the direction of Y
ε_{zz}	normal strain in the direction of Z
σ_{zz}	normal stress in the direction of Z
Φ, Ψ	orientation angles
$\varepsilon_{\Phi\Psi}$	normal strain in the direction defined by the orientation angles Φ and Ψ
$\sigma_{\Phi\Psi}$	normal stress in the direction defined by the orientation angles Φ and Ψ
ε_{Φ}	normal strain in the direction defined by the orientation angle Φ at $\Psi = 90°$
σ_{Φ}	normal stress in the direction defined by the orientation angle Φ at $\Psi = 90°$
(0,0,0)	origin of co-ordinate system (X,Y,Z)

Figure 11 — Stress and strain components at a measurement point (x, y, z) in the specimen co-ordinate system (X, Y, Z)

10 Reliability

The difference between a measurement result and the true value of the measurand comprises contributions from random uncertainties and systematic errors (ISO/IEC Guide 99[46]).

The determination of the uncertainty in a measurement is as important as the result itself, and without it the accuracy of a measurement cannot be estimated. Clause 11 lists the quantities to be reported when stresses and strains are determined by neutron diffraction. Uncertainties should be determined and reported in accordance with ISO/IEC Guide 98-3[26]. Additional guidelines are given in Reference [27]. An abbreviated summary of the nomenclature and method of calculating the combined standard uncertainty of a measurement is given in Annex B.

11 Reporting

11.1 General

The basic reporting philosophy is to describe the experimental procedure adopted, the results of the measurements and how the data were analysed. Thus, the reader will have sufficient information to reproduce, understand, evaluate and further interpret the results. A rigid reporting format is not put forth as there is great variability in materials, available information and objectives of studies. The exact format and contents of a test report are subject to the agreement between the client and the provider.

11.2 Strain or stress values

11.2.1 General

The strain or stress values resulting from the measurements shall be reported, as follows:

— the strain or stress components and the values determined, including their uncertainties;

— the locations at which the measurements were made, i.e. the centroid of the IGV or the centroid of the SGV as appropriate;

— the size and shape of the IGV or SGV;

— the sources of uncertainties and the way in which they affect the reliability of the results.

11.2.2 Stress-free or reference lattice spacing

The values, uncertainties and method used to obtain the reference or stress-free lattice-spacing(s), or unit cell parameter values for use in determining the relative or absolute strains, shall be described.

11.2.3 Conversion of strain to stress

The relations and assumptions used to convert strain to stress shall be reported.

11.2.4 Elastic constants

If the determined strains are converted to stresses, the values of the diffraction elastic constants used shall be provided and their source stated.

11.2.5 Positioning

The uncertainty in the positioning of the specimen shall be reported and its influence on the strain or stress values shall be estimated.

11.3 Neutron source and instrument

The following information shall be provided:

— the neutron source;

— the instrument at the source;

— the wavelength and the monochromator description (monochromatic instrument) or the wavelength range (TOF instrument);

— the instrument calibration procedure and the calibration measurement results.

11.4 General measurement procedures

The following aspects of the measurements shall be reported:

— the methods used to translate and orientate the specimen;

— the method used to locate the surfaces and other reference positions;

— the manner in which the gauge volume(s) is determined;

— the diffraction peak fitting function and the procedure used;

— the methods used to process the data, e.g., smoothing, outlier elimination;

— the method used to demonstrate the reliability of the results.

11.5 Specimens/materials properties

The following aspects of the material being studied should be reported when available:

— the specimen geometry;

— the composition;

— the thermal/mechanical history;

— the phases and crystal structures;

— the homogeneity;

— the sizes and shapes of grains, second phase particles or reinforcements;

— the texture.

11.6 Original data

Original data shall be included in the report if required. Reported data resulting from any smoothing procedure, to which the original data have been subjected, shall be described as such.

11.7 Uncertainties and errors

Those factors contributing to the uncertainties in stress and strain should be quantified and reported in accordance with Clause 10 and as illustrated in Annex B.

In addition, identified sources of systematic errors should be reported together with any measures taken to mitigate their impact.

Annex A
(informative)

Measurement and analysis methodologies

A.1 General

This annex presents procedures that are used by experienced practitioners to facilitate compliance with this document.

Many of these procedures were clarified within two international pre-normative research projects (VAMAS TWA 20[28][29] and RESTAND[30]). Further information is available in References [31] to [34].

A.2 Specimen co-ordinates

A.2.1 General

For most applications on regularly shaped specimens, rectangular or polar co-ordinates aligned with respect to specimen symmetry features are appropriate.

A.2.2 Specimens with elements of symmetry

Most components have significant elements of symmetry. Many have rectangular, circular or axial features. For such specimens, co-ordinates should be defined relative to the symmetry directions as follows:

a) rectangular specimens: along the orthogonal symmetry directions x, y, z normal to the faces;

b) cylindrical specimens: a cylindrical co-ordinate system that is aligned with the axial, radial and hoop (tangential) directions;

c) extended constant cross-section specimens: rolled, drawn and extruded components may have constant but sometimes complex cross-sections. For extended specimens, co-ordinates parallel to the long axis and along the orthogonal axes are appropriate.

EXAMPLES

Railway rails: longitudinal, transverse and normal.

Rods and pipes: axial, radial and hoop (tangential).

Regular polygonal cross-sections: triangular, square, hexagonal etc.; axial, normal to faces, parallel to faces.

A.2.3 Specimens of an irregular shape

In the general case of a specimen with an irregular shape, co-ordinates should be along three suitable directions, preferably orthogonal. It may be appropriate to employ one co-ordinate system throughout or several systems that are suitable for a series of local scans.

A.3 Positioning of the specimen

A.3.1 General

The specimen should be positioned relative to the reference point of the instrument.

A.3.2 The reference point

The reference point position shall be determined as accurately as is practicable, preferably to within 10 % of the minimum dimension of the gauge volume that is to be used.

A.3.3 The gauge volume

It is good practice to position the centroid of the IGV, the reference point, at the centre of rotation of the specimen table. The positioning should be as accurate as is practicable, preferably to within 10 % of the applied gauge volume dimensions. The location of the gauge volume in the measurement plane may be determined from the intensity distribution (wall scans) of scattered neutrons as described in A.5.1.

A.3.4 The specimen

The location and the orientation of the specimen should be described relative to the reference point and to the direction of the scattering vector using appropriate co-ordinates (see A.2). The specimen positioning accuracy should generally be similar to that of the reference point. The specimen position may be determined optically, mechanically, by wall scans, by virtual models, or by combined methods (see also 7.4). To define the specimen position, it is helpful to have a fiducial mark and directions indicated on the specimen. The fiduciary mark should be sufficiently fine and sharp so that the required positioning accuracy can be achieved. If orthogonal translators are used for positioning, they should be accurately orthogonal, preferably to < ±0,1° (±1,7 mrad). Specimen alignment is particularly important when scanning through steep strain gradients and interfaces, surfaces, or when large translation scans are required.

If it is not possible to position the specimen to the required accuracy using optical or mechanical methods, the position of the IGV relative to the specimen surface should be determined using surface (wall) scans. These provide a peak intensity profile, called an "entering curve", as a specimen surface is translated through the gauge volume that gives an experimental measure of the position of the surface relative to the gauge volume. It is necessary to repeat wall scans for each measurement orientation, and at a number of locations along a surface where there is significant translation parallel to that surface. Care should be taken when surface treated, textured, large grained or highly absorbing materials are probed. In these cases, the entering curve can be substantially different from that expected in the absence of these features.

NOTE 1 The limit of alignment by unaided eye of a skilled experimenter is ≈0,5°, optical or mechanical devices are therefore essential when a more precise alignment is needed.

NOTE 2 Any inaccuracy in positioning the specimen with respect to the reference point will introduce a systematic error in the location where the strain is determined. If the reference point does not coincide with the centre of specimen rotation, a rotation of the specimen will also result in an effective displacement of the gauge volume position relative to the specimen. This can introduce significant errors in stress determination, particularly in cases of steep strain or composition gradients. For example, an uncertainty in positioning Δx leads to a systematic uncertainty in strain $\Delta\varepsilon$ at that point given by $\Delta\varepsilon = (\partial\varepsilon/\partial x)\Delta x$. Thus, a positioning uncertainty of ±50 µm in a region with a strain gradient $\partial\varepsilon/\partial x$ of 2 000 × 10^{-6} per mm will result in a systematic uncertainty of ±100 × 10^{-6} in strain.

A.4 Number and location of diffraction measurement positions

A.4.1 General

The number and location of data points within a specimen should be sufficient to enable significant or specified strain changes to be resolved. The specific number and locations of points will depend upon the detail that is required, the variation in the strain pattern and the size of the gauge volume.

A.4.2 Measurements at one location

In some cases, measurements only at one location may be specified. These measurements are reliable in regions of material and strain uniformity. However, in cases of non-uniformity, interpolation from

additional measurements in close proximity of the specified location will be needed to assure the reliability in the results.

A.4.3 Strain mapping

For an efficient strain mapping it may be useful to obtain an outline pattern using first a coarse matrix of regularly distributed points and then to increase the point density in the vicinity of the specific features that may be of interest. When the strain gradient or its variation is large along a measurement direction, it may be necessary to increase the mapping density in that direction in order to obtain an adequate spatial resolution.

A.4.4 Material removal to facilitate measurements at difficult locations

The specimen geometry or shape may make it difficult or impossible to perform measurements at particular locations of interest due to beam attenuation or the specimen not fitting on the instrument used. In such cases the user could consider removing the material from the specimen to overcome such limitations. Any modification of the specimen or component can alter or introduce stresses in the region of interest. Significant care shall be taken when alterations are required. This requires careful checking by experimental techniques, such as strain gauges, and/or mathematical methods, such as finite element analysis, to establish to what extent the material removal process causes stress alteration in the region of the intended measurements.

A.5 Gauge volume

A.5.1 IGV determination

Because of beam divergence and other inevitable uncertainties of the experimental set-up, the IGV should be determined experimentally as described in 7.5.

Complete IGV parameters can be obtained by scanning a fine-wire probe through the gauge cross-section. At each scan position, the integrated intensity scattered by the probe is recorded. This is called an intensity profile.

The wire can be a Bragg-scatterer (e.g. steel or copper) or an incoherent scatterer (e.g. nylon). On a monochromatic instrument, it is best to use the former, whilst the latter works very well with a polychromatic beam as on TOF instruments. While scanning through the primary beam, the secondary beam shall be sufficiently wide, and vice versa, not to influence the intensity profile of the former.

These scans yield the intensity profiles and the dimensions, shape and position of the IGV and therefore the position of the reference point (see Figure 9). The shape of the gauge volume depends on the detector angle. Therefore, the Bragg angle of the reflection used for the gauge volume determination should be as close as possible to the angle of the reflection used for the measurement. The dimensions of the probe should be sufficiently small; otherwise attenuation corrections are required.

The IGV intensity profile may be illustrated by means of three 1-dimensional intensity profiles or a 3-dimensional intensity contour map. Such plots also illustrate the level of beam uniformity across the beam width. However, for most practical purposes, the IGV may be described by three dimensions and the diffraction angle. The dimensions quoted should correspond to the FWHMs of the intensity profiles of the incident and diffracted beams. The full widths of the intensity profiles should be provided to give an indication of the sharpness of the IGV boundaries.

A thin metal sheet can be used to map the gauge volume in the scattering plane. It is scanned through the gauge volume, once with its surface normal being parallel to the scattering vector and once with it being perpendicular. The scan direction is given by its surface normal.

A.5.2 Alignment of beam-defining optics

The beam-defining optics should be aligned such that the reference point will be in the desired position. To this end the above-described scans and other techniques[35] can be used to perform the necessary corrections in the positioning of the beam-defining optics.

Alternatively, a cylindrical scatterer of the dimensions of the gauge volume can be placed at the preferred gauge volume position and each beam-defining optics component be scanned across its respective beam while recording the intensity profile. The centroid of the profile determines the correct position of the optics component.

The primary beam can also be aligned very efficiently by applying the methods discussed while replacing the scatterer by a narrow slit and placing a detector behind it.

The distances of the incident and diffracted beam-defining optics from the reference point should be set as appropriate. In the case of slits, these distances should be as small as is practicable so that the effects of divergences are minimized, whilst still permitting movement of the specimen with minimal risk of collision when scanning. When radial collimators are used, they should be aligned such that their focus coincides with the reference point.

If the beam positioning reproducibility cannot be guaranteed after a gauge volume change, the gauge positioning procedure shall be repeated.

A.5.3 Gauge dimensions

Gauge dimensions should be chosen so as to permit the detail to be resolved as necessary. If gauge dimensions exceed the size of or distance between features of significance, the detail is lost in the strain patterns.

It is important to describe the beam-defining optics in order to be capable to reproduce the measurement and to estimate the IGV[36].

A.5.4 Grain size

When the size of the grains becomes large relative to the SGV, the statistical sampling can become too small for a reliable determination of an average lattice spacing of grains within the volume contributing to the scattering. For example, at one location in the sample a large grain can make a disproportionately strong contribution to the measured intensity, but if incorrectly oriented, the peak centre will not provide a correct measurement of the lattice spacing, skewing the SGV average. Conversely, at a nearby location, the portion of that grain in the SGV may become small resulting in a negligible contribution.

A very large single grain within the gauge volume can make a very strongly biased contribution to a diffraction peak. This can result in large point-to-point variations in the intensity along a line of measurements that are significantly greater than the calculated uncertainty in the intensity of the individual measurement. Likewise, for a given measurement location, rotation of the sample about the IGV centre will also lead to large angle-to-angle intensity fluctuations if the grains are large, while for sufficiently small grains, the fluctuations will be comparable to the intensity uncertainty and therefore appear smoothly varying, even for a sample with a moderate crystallographic texture.

The solution is to increase the number of contributing grains for good statistical sampling. This can be achieved with a larger SGV, by angular oscillation of the sample about the IGV centre, or spatial oscillation. Normally other factors drive the choice of the IGV, and angular oscillation is more efficient than spatial oscillation.

The angular scan test noted above can be repeated with increasing mitigation measures until the intensity fluctuations are comparable to the uncertainty in the intensity of the individual measurement thereby confirming good statistical sampling of the grains.

Another example of a mitigation technique is the use of a 180° rotation, which can significantly reduce the effect[37].