TECHNICAL
SPECIFICATION

**ISO/TS
21432**

First edition
2005-07-15

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

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

Reference number
ISO/TS 21432:2005(E)

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Foreword

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Introduction

Neutron diffraction is a non-destructive method that can be employed for determining residual stresses in crystalline materials. It can also be used for establishing 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. Measurements of elastic strain are obtained which are then converted to stress. The purpose of this document is to provide the technical specification for reliably determining stresses that are relevant to engineering applications.

TECHNICAL SPECIFICATION **ISO/TS 21432:2005(E)**

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

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

1 Scope

This Technical Specification gives the standard test method for determining residual stresses in polycrystalline materials by neutron diffraction. It is applicable to homogeneous and inhomogeneous materials and to test pieces containing distinct phases.

The principles of the neutron diffraction technique are outlined. Advice is provided on the diffracting lattice planes on which measurements should be made for different categories of materials. Guidance is provided about the directions in which the measurements should be obtained and of the volume of material, which should be examined, in relation to material grain size and the stress state envisaged, when making measurements.

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

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

The methods of making individual elastic strain 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 (or applied) stress from the strain data and of how to estimate the uncertainty in the results.

2 Normative references

The following referenced documents are indispensable for the application 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, *Non-destructive testing — X-ray diffraction from polycrystalline and amorphous materials — Part 3: Instruments*[1]

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[1] To be published.

3 Terms and definitions

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

3.1
absorption
neutron capture by an atomic nucleus

NOTE Tables of nuclear capture cross sections can be found under e.g. http://www.webelements.com and links.

3.2
alignment
adjustment of position and orientation of the specimen and all components of the instrument such that reliable strain measurements by neutron diffraction can be performed at the desired location in the specimen

3.3
anisotropy
dependence of material properties on orientation

3.4
attenuation
reduction of neutron intensity

NOTE Attenuation can be calculated by using the so called "total neutron cross section", which comprises absorption and different nuclear scattering processes. The attenuation length is the distance within the material for which the primary neutron intensity is reduced by $1/e$.

3.5
background
intensity considered not belonging to the diffraction signal

NOTE Background dependence on scattering angle or time-of-flight is not uncommon and can have an influence on the peak position resulting from data analysis.

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

NOTE These include devices such as apertures, slits, collimators, monochromators and mirrors.

3.7
Bragg edge
sudden change in neutron intensity as a function of wavelength or diffraction angle corresponding to $\lambda=2\mathrm{d}_{h'k'l'}$ where $h'k'l'$ indicates a diffracting lattice plane

3.8
Bragg peak
intensity distribution of the diffracted beam for a specific hkl lattice plane

3.9
peak height
maximum intensity of the Bragg peak above the background

3.10
peak function
analytical expression to describe the shape of the diffraction line

3.11

peak position

single value describing the position of a Bragg peak

NOTE The peak position is the determining quantity to calculate strain.

3.12

diffraction

scattering based on interference phenomena

3.13

diffraction elasticity constants

elasticity constants associated with individual (*hkl*) lattice planes for a polycrystalline material

NOTE They are often called elastic constants and can be denoted as E_{hkl} (diffraction elastic modulus) and ν_{hkl} (diffraction Poisson's ratio).

3.14

diffraction pattern

distribution of scattered neutrons over the available range of wavelengths or times of flight and/or scattering angles

3.15

full width at half maximum

FWHM

width of the diffraction line at half the maximum height above the background

3.16

full pattern analysis

determination of crystallographic structure and/or microstructure from a measured diffraction pattern of a polycrystalline material

NOTE In general the full pattern analysis is termed after the method used (e.g. Rietveld refinement) See also single peak analysis.

3.17

gauge volume

volume from which diffraction data are obtained

NOTE This volume is determined by the intersection of the incident and diffracted neutron beams.

3.18

lattice parameters

linear and angular dimensions of the crystallographic unit cell

NOTE Most engineering materials have either cubic or hexagonal crystal structures. Hence the lattice parameters usually only refer to the lengths of the unit cell edges.

3.19

lattice spacing

d-spacing

spacing between adjacent crystallographic lattice planes

3.20

macrostress

type I stress

mean stress in a volume containing a large number of grains

NOTE Also called stress of type I.

3.21
microstress
mean stress deviation in a restricted volume from the macrostress level

NOTE There are two classes of microstress:

- the mean deviation from the macrostress determined over a grain or phase dimension (also called type II);
- the mean deviation from the type II stress determined over a volume of several atomic dimensions (also called type III).

3.22
monochromatic instrument
neutron instrument employing a narrow band of neutron energies (wavelengths)

3.23
monochromatic neutron beam
neutron beam with narrow band of neutron energies (wavelengths)

3.24
orientation distribution function
quantitative description of the crystallographic texture

NOTE The orientation distribution function is necessary to calculate the elasticity constants of textured materials.

3.25
polychromatic neutron beam
neutron beam containing a continuous range of neutron energies (wavelengths)

3.26
reference point
centroid of the instrumental gauge volume

NOTE See 6.5.

3.27
reproducibility
closeness of the agreement between the results of measurements of the same measurand carried out under changed conditions of measurements

[VIM: 1993]

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

NOTE 2 Reproducibility can be expressed quantitatively in terms of the dispersion characteristics of the results.

NOTE 3 Results are here usually understood to be corrected results.

3.28
scattering
coherent scattering
scattering of neutrons from ordered scattering centres producing constructive and destructive interference of the particle waves

3.29
incoherent scattering
scattering of neutrons in an uncorrelated way

3.30

single peak analysis

statistical procedure to determine the characteristics of a peak and the background from the measured diffraction data

3.31

texture

preferred orientation of crystallites (crystallographic texture) or reinforcements (morphological texture) within a specimen

3.32

through surface scan

procedure to determine the position of a specimen surface or interface

NOTE Sometimes also termed surface scan or intensity scan while its result is often called an entering curve.

3.33

time-of-flight

time needed by a neutron of a given speed (i.e. energy or wavelength) to cover the distance from a defined starting point to the detector

3.34

uncertainty of measurement

parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand

[VIM: 1993]

NOTE 1 The parameter may be, for example, a standard deviation (or a given multiple of it), or the half-width of an interval having a stated level of confidence.

NOTE 2 Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of the results of a series of measurements and can be characterized by experimental standard deviations. The other components, which also can be characterized by standard deviations, are evaluated from assumed probability distributions based on experience or other information.

NOTE 3 It is understood that the result of the measurement is the best estimate of the value of the measured, and that all components of uncertainty, including those arising from systematic effects, such as components associated with corrections and reference standards, contribute to the dispersion.

NOTE 4 Uncertainty needs to be distinguished from accuracy of a measurement, which can be influenced by a systematic bias.

3.35

wall scan

see-through surface scan

4 Symbols and abbreviated terms

4.1 Symbols

a,b,c	Lengths of the edges of a unit cell, here referred to as lattice parameters	nm
B	Background at peak position	—
d	Lattice spacing	nm
e	energy	
E	Elasticity modulus	GPa

E_{hkl}	Elasticity 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	
$hkil$	Alternative indices of a crystallographic lattice plane for hexagonal structures	
H	Peak height	—
I	Integrated neutron intensity of a Bragg peak above background	
k_{i}, k_{f}	Wave vector of the incident and scattered neutrons	nm^{-1}
L	Path length from neutron source to detector	m
l	Neutron attenuation length	mm
m_n	Neutron mass (1.67×10^{-27} kg)	kg
N_n	Total number of neutrons counted	
Q	Scattering vector ($k_{\mathrm{f}} - k_{\mathrm{i}}$)	nm^{-1}
t	Time of flight of neutrons from source to detectors	
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	—
$\varepsilon_{\mathrm{ij}}$	Components of elastic strain tensor	—
$\varepsilon_{\mathrm{hkl}}$	Normal elastic strain associated with the (hkl) diffracting lattice plane	—
λ	Wavelength of neutrons	nm
v	Poisson's ratio	
v_{hkl}	Poisson's ratio associated with the (hkl) diffracting lattice plane	
σ	Stress	MPa
σ_{ij}	Components of stress tensor	MPa
σ_{Y}	Yield stress	MPa
2θ	diffraction angle	degrees
ϕ, ψ, ω	Orientation angles	degrees

4.2 Subscripts

hkl, hkil	Indicate relevance to crystallographic lattice planes
x, y, z	Indicate components along the x-, y-, z-axes of the quantity concerned
$\phi\ \psi$	Indicate the normal component, in the ($\phi\ \psi$) – direction of the quantity concerned
0 (zero)	Indicates strain 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 Preamble

This Technical Specification is concerned with the determination of residual and/or applied stresses that are needed in engineering analysis. These are determined from neutron diffraction measurements of the lattice spacing between crystallographic planes. From changes in these spacings, elastic strains can be derived, from which stresses can be calculated. By translating a specimen or component through a neutron beam, stresses at different locations can be determined, provided enough strain measurements are obtained. In this clause the strain measurement process is summarized.

5.2 Outline of principle — Bragg's law

When illuminated by radiation of wavelength similar to interplanar spacings crystalline materials diffract this radiation as distinctive Bragg peaks. The angle at which a diffraction line occurs is given by Bragg's law of diffraction.

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

where λ is the wavelength of the radiation, d_{hkl} is the spacing of the hkl lattice planes responsible for the Bragg peak and θ_{hkl} is the Bragg angle. The peak will be observed at an angle of $2\theta_{\mathrm{hkl}}$ from the incident beam, as shown schematically in Figure 1.

5.3 Neutron sources

Neutron diffraction uses neutrons generated by 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. $\lambda \geqslant 0.09$ nm. At reactor sources, a monochromatic beam of neutrons is usually produced by using a crystal monochromator to select a given neutron wavelength from the polychromatic beam. At spallation sources, the neutron beam usually consists of a series of short pulses each containing a spectrum of wavelengths The energy (and therefore wavelength) 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 time of flight (TOF). TOF measurements are, therefore, wavelength dependent (sometimes termed energy dispersive), with the entire diffraction pattern being recorded at any particular scattering angle. Short pulses of polychromatic neutrons can also be produced by one or more choppers at continuous sources or from long pulses.

5.4 Strain measurement

When a specimen is illuminated by a monochromatic parallel beam of neutrons of known wavelength, its lattice spacing can be determined from the observed Bragg angle using Bragg's law (1). If the specimen contains no strain, the lattice spacings correspond to the strain free (stress free) values for the material and are denoted as $d_{0,\mathrm{hkl}}$. In a stressed specimen lattice spacings are altered and a shift in each Bragg peak occurs allowing the elastic strains to be given by:

$$\varepsilon_{\mathrm{hkl}} = \frac{d_{\mathrm{hkl}} - d_{0,\mathrm{hkl}}}{d_{0,\mathrm{hkl}}} = \frac{\Delta d_{\mathrm{hkl}}}{d_{0,\mathrm{hkl}}} = \frac{\sin\theta_{0,\mathrm{hkl}}}{\sin\theta_{\mathrm{hkl}}} - 1 \qquad (2)$$

At a TOF instrument, pulses, containing neutrons spanning a range of velocities, and therefore wavelengths, are directed at the specimen. From the measured flight time t of detected neutrons, their wavelength is calculated using the de Broglie relationship to give:

$$\lambda = \frac{h}{m_{\mathrm{n}} \cdot L} \cdot t \qquad (3)$$

By substituting (3) into Bragg’s law (1), the time of flight for a particular wavelength and crystal plane becomes :

$$t_{\mathrm{hkl}} = 2\frac{m_{\mathrm{n}}}{h} \cdot L \cdot \sin\theta \cdot d_{\mathrm{hkl}} \qquad (4)$$

for a detector positioned at angle 2θ.

As the incident neutron beam is polychromatic, the reflections of all lattice planes normal to the direction in which the strain is measured are recorded. Each reflection is produced from a different family of grains oriented such that a specific *hkl* plane diffracts to the detector. The elastic strain can then be calculated from the flight time shifts in any of the observed reflections in a manner analogous to that described in equation (2) so that for a fixed angle 2θ:

$$\varepsilon_{\mathrm{hkl}} = \frac{\Delta d_{\mathrm{hkl}}}{d_{0,\mathrm{hkl}}} = \frac{t_{\mathrm{hkl}} - t_{0,\mathrm{hkl}}}{t_{0,\mathrm{hkl}}} = \frac{\lambda_{\mathrm{hkl}} - \lambda_{0,\mathrm{hkl}}}{\lambda_{0,\mathrm{hkl}}} \qquad (5)$$

It should be noted that simultaneous recording of reflections of various lattice planes can facilitate analysing the data by multi-peak fitting or full pattern analysis (see 6.3.2).

For both monochromatic and TOF instruments, the direction in which strain is measured is along the scattering vector, $Q = k_{\mathrm{f}} - k_{\mathrm{i}}$, which bisects the angle between incident and diffracted beams and is perpendicular to the diffracting planes as shown in Figure 1.

5.5 Neutron diffractometers

A monochromatic instrument typically used for strain measurement 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 then given spatial definition by the use of appropriate beam defining optics to produce a beam of controlled dimensions. This beam is then diffracted from the specimen and captured by a neutron detector. An example of a diffraction peak from a monochromatic instrument is shown in Figure 3.

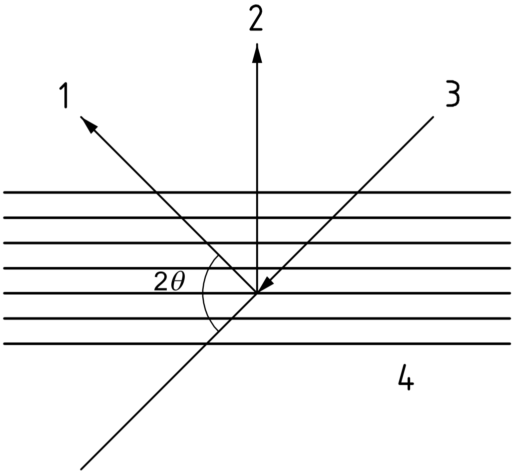
At TOF-diffractometers typically used at pulsed sources, each pulse provides a diffraction profile across a large range of lattice spacings. A typical TOF-diffractometer used for strain measurement in two directions simultaneously at a pulsed source is shown in Figure 4. As a fixed scattering angle is used, most instruments at spallation sources use radial (focussing) 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 6.5). 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 more than one Q (strain) direction to be measured simultaneously. 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 6.3.2).

5.6 Stress determination

Stress and elastic strain are second rank tensors that are related through a solid's elasticity constants. Since neutron diffraction can measure the elastic strain within a defined volume in a crystalline solid, it is possible to calculate the mean stress in that volume provided the relevant material elasticity constants are known. Full determination of the strain tensor requires measurements of the elastic strain in at least six independent directions. If the principal strain directions within the body are known, measurements along these three directions are sufficient. For plane stress or plane strain conditions, a further reduction to two directions is possible. Measurement along one direction only is needed in the case of uni-axial loading.

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

By sequentially moving the specimen through the volume (termed a gauge volume, see clause 6.5) in space identified by the intersection of the incident and diffracted beams, the spatial variation in elastic strain and, following measurement in other directions, stress can be mapped within a specimen or component.



Key

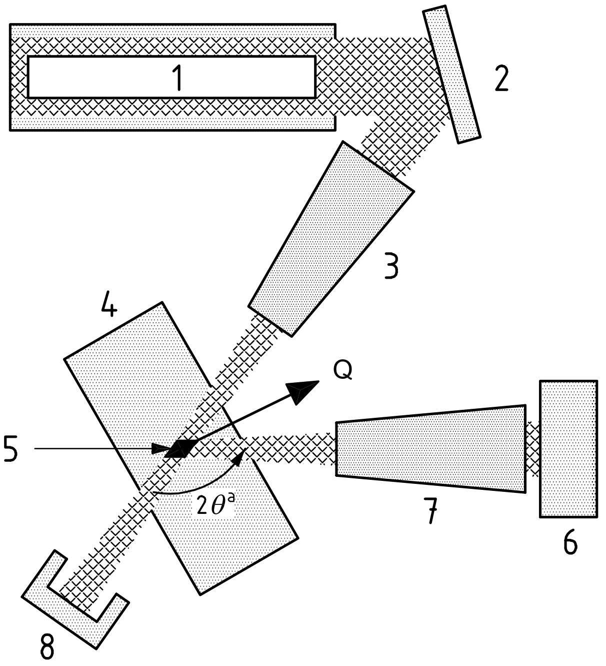
1 diffracted wave vector k_f

2 incident wave vector k_i

3 scattering vector Q

4 diffracting planes

Figure 1 — Schematic illustration of Bragg scattering geometry



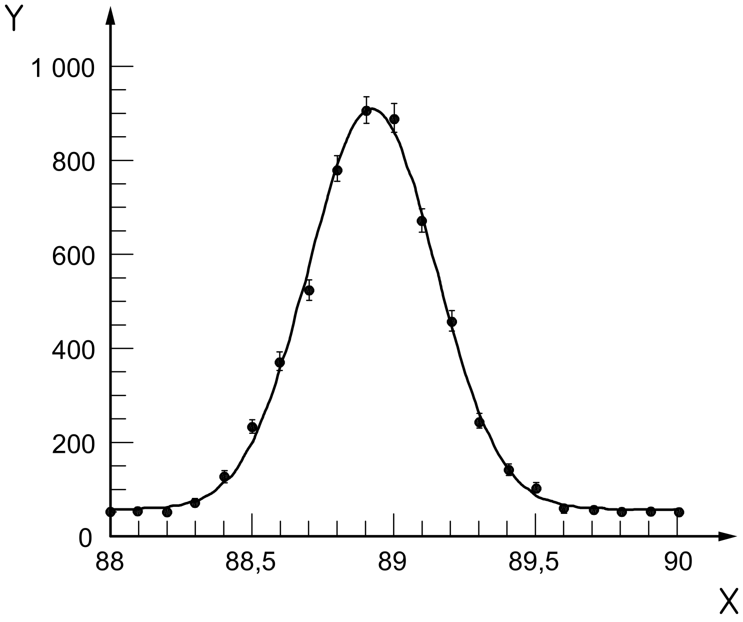
Key

1 neutron beam from 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

[a] Scattering angle.

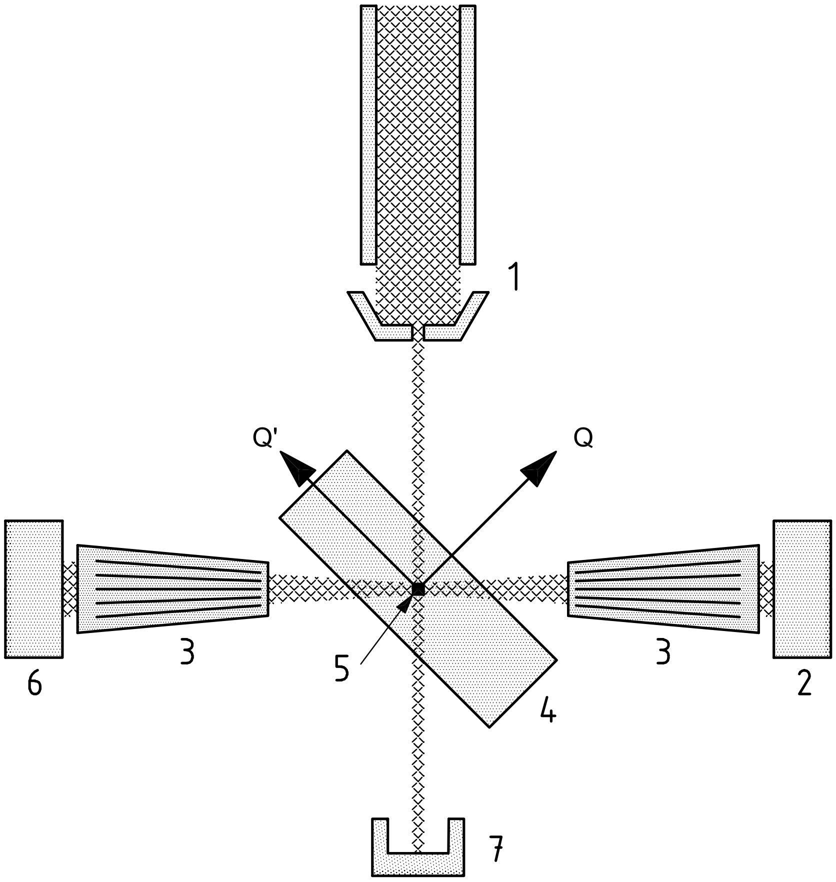
Figure 2 — Schematic illustration of a steady state source based diffractometer for strain measurement



Key

1 2θ, degrees
2 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	beam from 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 strain measurement

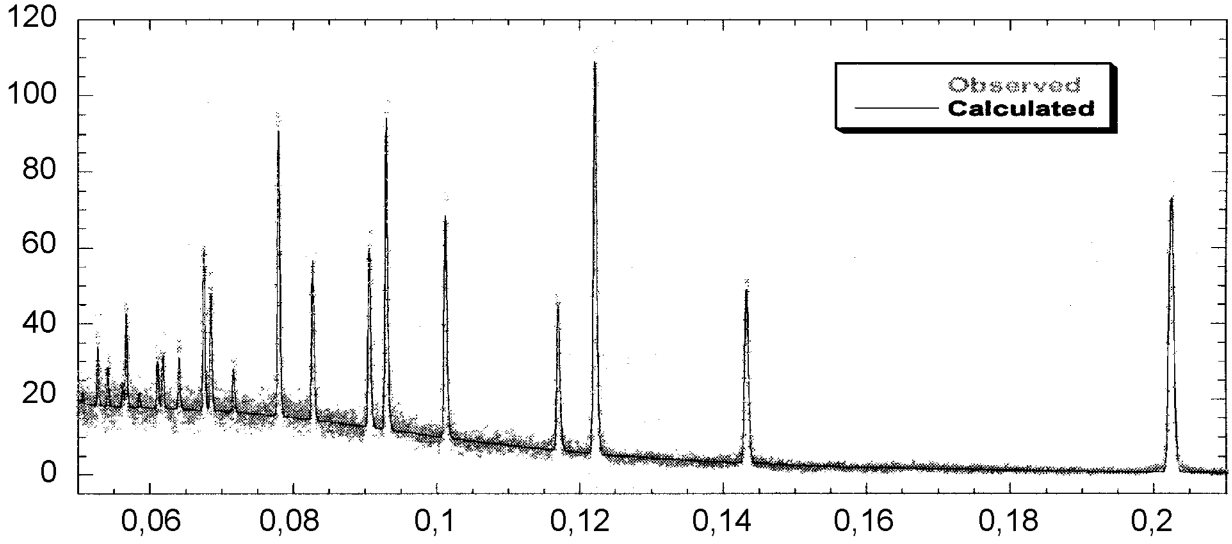


Figure 5 — Example of a diffraction pattern from a pulsed source. The solid line is the result of a Rietveld fit to the data as described in 6.3.2

6 Preparations for measurements

6.1 Preamble

Prior to an actual strain measurement it is necessary to align the instrument and/or verify its alignment. Then appropriate conditions for the diffraction measurement have to be chosen and the specimen has to be positioned accurately on the diffraction instrument. Also the size and shape of the volume from which diffraction will be observed have to be determined, and the value has to be assessed of the d-spacing with respect to which the strain will be determined.

6.2 Alignment and calibration of the instrument

It is necessary to align and calibrate the diffractometer being used (see A.4.2). When using a monochromatic beam instrument, it is necessary to ensure that a constant wavelength is maintained throughout the entire set of measurements and that the detector angular response has been calibrated (see EN 13925-3:—2) Annex C). At a TOF-diffractometer, both the flight path and detector angular response should be calibrated. In both cases this is done using a standard stress free specimen typically silicon, ceria or alumina powders. Such specimens are chosen because they diffract neutrons well, 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 wavelength. One way of doing this is to use an incoherent scatterer, such as vanadium.

6.3 Choice of diffraction conditions

6.3.1 Monochromatic instruments

6.3.1.1 Choice of 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 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 which can cause artificial peak shifts. These 'problematic' wavelengths have been tabulated in [1] for several common metals over a range commonly used for strain measurements. For cubic materials, in particular, scattering angles of 90° should be avoided since for all $\{hkl\}$ diffraction planes there is an alternate $\{h'k'l'\}$ which would cause a Bragg edge related effect.

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

6.3.1.2 Choice of 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 [2]. This may be illustrated by loading and unloading a tensile bar, in situ, in a neutron diffractometer whilst measurements of stress and strain are recorded, as indicated in Figures 6 and 7. In these figures, stress recorded by a load cell in series with a test bar is plotted against elastic strain measured by neutron diffraction.

It is evident, within the elastic region as shown in Figure 6, that a linear response is obtained whichever set of lattice planes is used to make the measurements. This demonstrates that any hkl reflection can be employed for determining stress in this region, provided the appropriate diffraction elastic constants are chosen. Generally, these are neither the bulk elastic constants nor the single crystal values, but a polycrystalline

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2) To be published.

aggregate value associated with a particular *hkl* plane. These constants can be obtained either experimentally as Figure 6 demonstrates, or can be calculated (see clause 9). The calculation methods include the Reuss [3], Voigt [4], Neerfeld-Hill [5], [6] and self-consistent methods, e.g. Kröner [7]. 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 needs to be taken into account. See references [8] and [9] for discussion on the importance of texture.

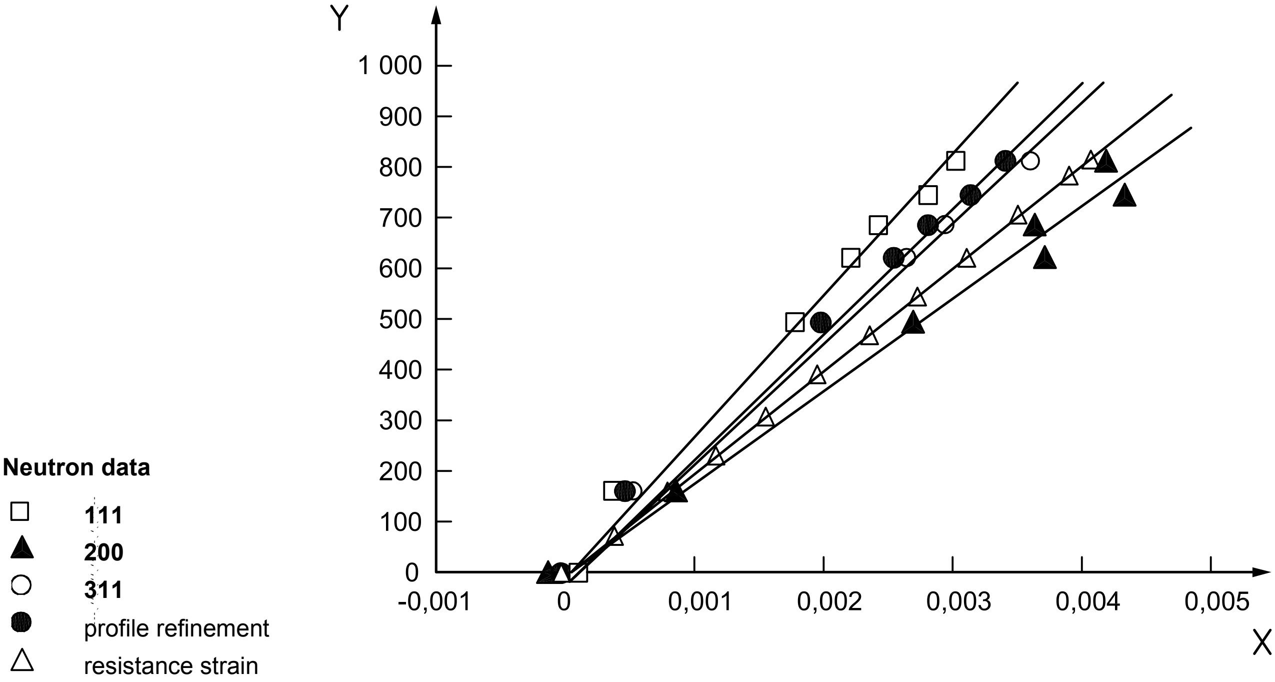
Plastic deformation begins at different stresses recorded by the load cell in differently oriented grains, as illustrated in Figure 7. This is demonstrated by a non-linear response on loading followed by linear elastic unloading. The consequence is that a different residual elastic strain may be measured on each *hkl* plane on unloading to zero applied stress. These are usually called intergranular strains. For no remaining load on the test bar, the engineering (macroscopic) residual stress shall be zero to satisfy equilibrium conditions. Non-zero residual strains at zero load for any crystallographic plane will translate into a residual stress. Consequently it is important, for engineering residual strain measurements, that a crystallographic plane is chosen which gives essentially zero residual strain on unloading [e.g. plane (220) or (311) in Figure 7].

If a suitable *hkl* plane is not known, or a new material is being examined, an appropriate plane can be determined by loading a tensile bar into the plastic region as shown in Figure 7.

Nevertheless, in some cases it is necessary and appropriate to employ *hkl* planes that are sensitive to intergranular strains. In such cases compensation has to 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 [10]. Examples of *hkl* planes with high and low sensitivity to intergranular strains for a range of materials are listed in Table 1.

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 [11], Fe [12], Cu [13]), fcc (Al [14], [15]), Ni [2]	111, 311, 422	200
bcc (Fe [15])	110, 211	200
hcp (zircaloy [16], Ti [17])	$10\bar{1}2$, $10\bar{1}3$ (Pyramidal)	0002 (basal) $10\bar{1}0$, $1\bar{2}10$ (prism)
hcp (Be [18])	$20\bar{2}1$, $11\bar{2}2$ (2nd order pyramidal)	$10\bar{1}2$, $10\bar{1}3$ (basal, prism and 1st order pyramidal)

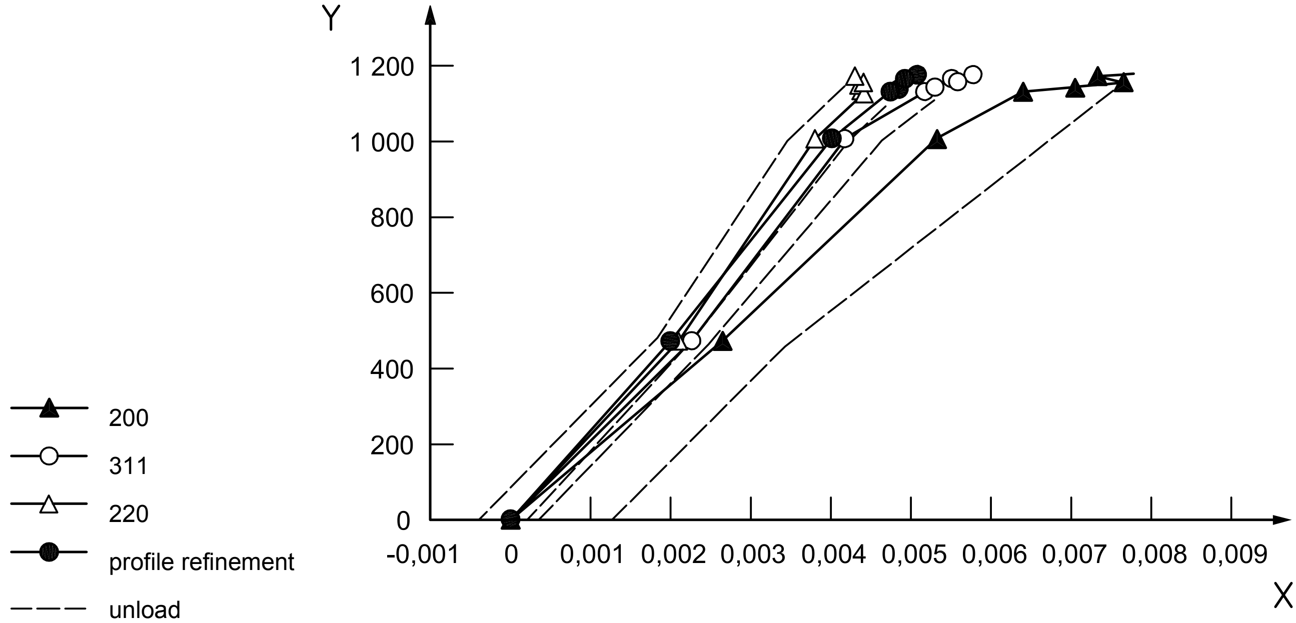


Key

1 stress (MPa)

2 strain

Figure 6 — Elastic response of different crystallographic planes for nickel alloy [2]



Key

1 stress (MPa)

2 strain

Figure 7 — Effect of yielding on response of different crystallographic planes to loading and unloading of a tensile bar of a nickel alloy [2]

6.3.2 TOF instruments

At a TOF instrument many peaks are recorded simultaneously. In this case strains can be determined from one or more selected individual *{hkl}* planes as described for monochromatic instruments (6.3.1) or by averaging over all planes using a full pattern analysis such as the Rietveld refinement procedure [19]. In this latter case, strain is obtained from changes in the lattice parameters defining the unit cell dimensions. It has been shown that this procedure gives adequately small residual stress in tensile bars after unloading from the plastic region to several percent plastic strain and is suitable for determining residual stresses for engineering purposes [2, 20] (see also Figure 7).

For cubic materials with lattice parameter a_0, strain is given by:

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

where the lattice parameter *a* is the value obtained from the full pattern analysis (it replaces the lattice spacing d in equation (2)). For non-cubic materials it is necessary to identify a suitable strain parameter, e.g. in untextured hexagonal materials an appropriate expression for the strain ε is:

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

where ε_a and ε_c are the strains determined from the *a* and *c* lattice parameters respectively in the same way as in equation (6) [18].

6.4 Positioning procedures

The initial alignment procedure requires the determination of the location of the centroid of the IGV (see 6.5 and Fig. 8.b). 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.

Accurate specimen positioning is required, as described in A.2. The level of accuracy required depends to some extent on the type of measurements being made, but typically should be within ± 0,1 mm. Highest positioning accuracy is most important in the case of large strain gradients and where measurements are being made close to surfaces. It is important that the uncertainty in positioning is known.

Alignment can be carried out for example by optical or mechanical means, or by using through surface scans (see annex A.2.3). All three methods are capable of determining the position of a specimen edge relative to the neutron beam to an uncertainty of 0,1 mm.

6.5 Gauge volumes

The nominal gauge volume (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.a). The centroid of the NGV is the geometric centre of this volume (see 6.4).

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

The instrumental gauge volume (IGV) is the volume of space defined by the actual neutron beam paths through the defining apertures, taking into account beam divergence and the beam intensity profile (Figure 8.b). A common method of determining the IGV involves scanning a small probe through it (see annex A.4.1 for details). 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. Note that the IGV and NGV are properties of the diffractometer itself.

Finally, the sampled gauge volume (SGV) is the intersection of the IGV with the specimen phase under investigation (see Figure 8.c). It is the volume over which the average strain is obtained. This average is affected by:

- 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.

It is for the above reasons that the centroid of the SGV will be at a different position to that of the IGV as shown in Figure 8.c.

The SGV and its centroid should be determined for each measurement. The position at which the average strain in this volume is obtained is then the intensity weighted position of the centroid of the SGV. It is important that the measured strain is reported at this position. The effect of the weighting will be most significant at surfaces and interfaces, and in highly attenuating materials. The consequences of the SGV centroid being offset from the reference point are discussed in annexes A.4.5 and A.5.

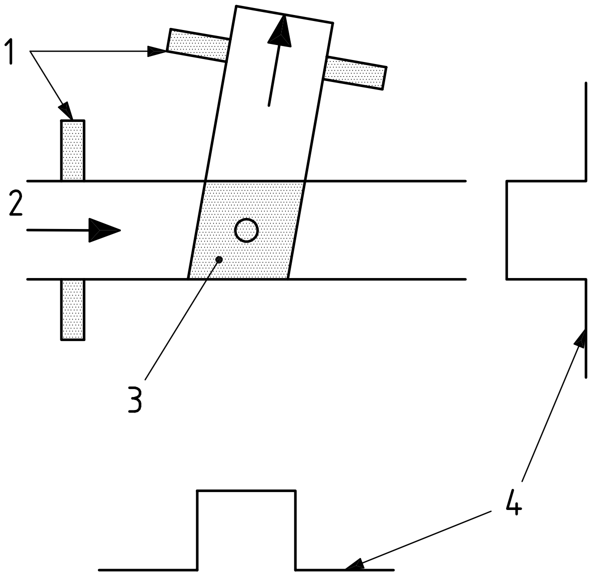
6.6 Determination of a strain free or reference lattice spacing

Since diffraction measurements allow the determination of lattice spacings, in order to measure elastic strains it is necessary to have a reference value, relative to which the strains can be determined. In some cases it is possible to determine a strain 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) will be possible. It should be noted that actual values of stress can only be determined when strains are calculated relative to d_0. Use of d_{ref} should only be made when values of d_0 are not available.

Lattice spacings are sensitive to a number of causes, apart from stress and instrumental aberrations, and these shall be taken into account. The most important of these are chemical composition and temperature. The optimum method of determining d_0 (or d_{ref}) will depend on the particular application under consideration. Methods include

- measurement in a material at a position known to contain negligible stress,
- measurement on a powder, which is representative of the material being examined. This is particularly suitable for multiphase materials,
- measurement on small coupons, cut from large blocks of material. This is relevant to welds, since use of multiple coupons allows determination of spatial and directional variations in d_0 through a weldment to be obtained [10],
- calculation of d_0 by imposing force and moment equilibrium. This is possible when sufficient measurements across an appropriate section have been made in a component in which there is no d_0 variation across that section. It is recommended however that experimental methods are used where possible, and that equilibrium is employed mainly as a check for consistency, and
- calculation 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 accurate near surface strain measurements are possible.

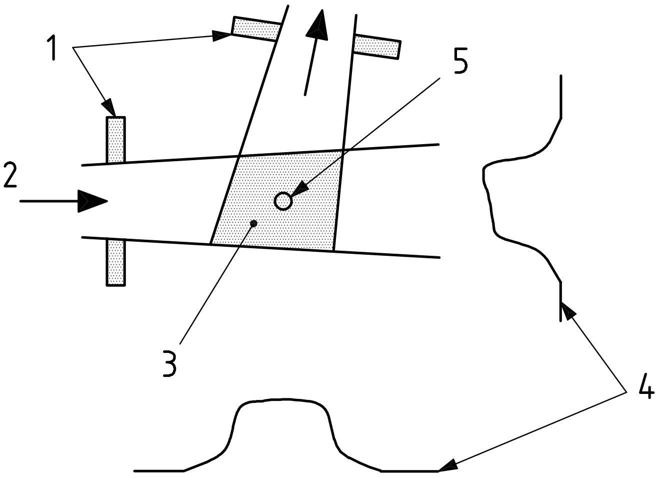
Care shall be taken with the preparation of “stress-free” material to avoid the introduction of residual stresses, or modification of the microstructure, during manufacture.



Key

1 apertures
2 incident neutrons
3 nominal gauge volume
4 neutron intensity profile

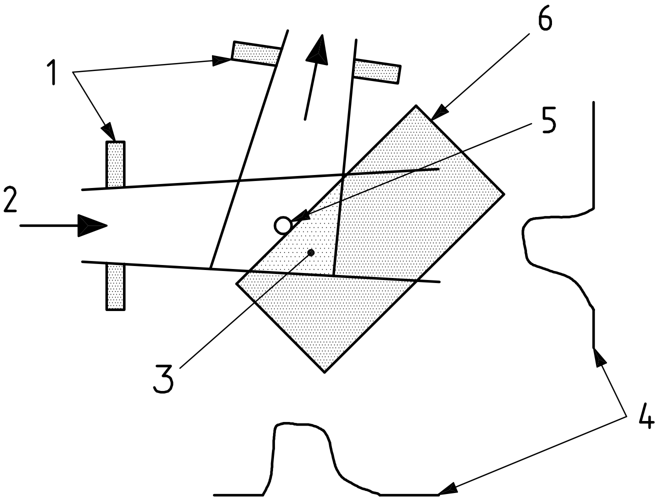
a)



Key

1 apertures
2 incident neutrons
3 instrumental gauge volume
4 neutron intensity profile
5 reference point

b)



Key

1 apertures
2 incident neutrons
3 instrumental gauge volume
4 neutron intensity profile
5 reference point
6 specimen

c)

Figure 8 — Plan views of the a) nominal b) instrumental and c) sampled gauge volumes. “O” indicates the centroids of the NGV and the IGV, and “X” the centroid of the SGV. The centroid of the IGV is the Reference Point

7 Material characterization

7.1 Preamble

A number of factors concerning the thermal and mechanical history experienced by the specimen or component to be examined can affect the state of residual strain in the material, its measurement, and its conversion to stress. Those aspects that have bearing on the measurements shall be reported. Most of these aspects are cited below. In some instances, it may be appropriate to carry out preliminary diffraction measurements to establish the scope of the investigation. This type of information is required to estimate, for example, diffraction elasticity constants, beam attenuation, diffracted beam intensity, background intensity, and possible issues with regard to radioactive activation.

7.2 Composition

Standard material designations that indicate 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 stress determination.

7.3 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, the previous operating conditions may also be relevant.

7.4 Phases and crystal structures

The phases in the alloys, ceramics, and composites shall be known. The crystallographic structure of phases used in the measurements shall be specified.

7.5 Homogeneity

Information about any spatial variation in composition or phase distribution is relevant to the experiment. This may affect confidence in making measurements 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 6.6).

7.6 Microstructure

The number of grains in the gauge volume is important in determining the quality of a diffraction pattern. 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 is being sampled. Consequently the grain size in relation to the gauge volume employed and to the stress distributions measured shall be known.

7.7 Texture

The presence of crystallographic texture will affect diffraction peak intensity and the conversion of strain to stress. If the material is known to possess texture, as a result of processing or use, it shall be characterised.

8 Recording requirements and measurement procedure

8.1 Preamble

Three parameters shall be determined with appropriate accuracy: a) strain, b) direction of the strain measurement, 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. Supplementary details for this clause are provided in annexes A and B.

8.2 Recording requirements

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

8.2.1 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 and width, and distance to 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:

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

f) wavelength and how it was determined;

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

h) resolution of the detector.

Parameters for TOF instruments:

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

j) wavelength range and how it was determined;

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

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

m) time resolution or channel width;

n) incident intensity as a function of wavelength.

8.2.2 General information — specimen

Specimen related information:

a) specimen material; chemical composition, crystal structure;

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

8.2.3 Specific information required for each strain measurement

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

Information related to specific measurements:

For monochromatic instruments:

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

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

For TOF instruments:

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

d) times of flight for the lattice planes in the strain free condition, $t_{0,\mathrm{hkl}}$ (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:

e) specimen orientation relative to the scattering vector Q ± uncertainty;

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

g) strain ± uncertainty;

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

For single peak fits:

i) angle or time increment;

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

    1) FWHM ± uncertainty;

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

    3) background B ± uncertainty.

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

k) peak profile(s) used, and relevant parameters including:

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

  2) peak profile asymmetry;

l) background fit used ;

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

8.3 Specimen co-ordinates

The co-ordinate system used to define 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 6.4) The orientation of the specimen co-ordinate system shall be defined in relation to the co-ordinate system used to define Q. The reference point position shall be defined as accurately as is practicable. Details are given in A.2.

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 coordinate 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, which require long neutron path length within the specimen, it may be necessary to remove material to make the measurement possible.

Details are given in A.3.

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.4.

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.5.

8.9 Temperature

The specimen temperature shall be monitored and controlled such that changes in lattice dimensions are either small relative to the uncertainty specified for the strain measurement, or can be accounted for. Details are given in A.7.

9 Calculation of stress

9.1 Preamble

With neutron diffraction elastic strains are measured and stresses calculated. As in X-ray diffraction, only normal strains are measured; shear strains shall be calculated, if needed, along with stresses.

Essentially all diffraction investigations of stresses and strains are based on continuum mechanics using Hooke's law for stress calculations. As discussed in 6.3, the only major alteration is the use of specific diffraction elasticity constants rather than the overall aggregate average. Hence, the average elasticity constants in the generalized Hooke's law are simply exchanged with the appropriate diffraction elasticity constants ($E_{\mathrm{hkl}}, \nu_{\mathrm{hkl}}$). 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 measurements made along mutually orthogonal co-ordinate axes, x, y and z at that point. In this case the stresses become:

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

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

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

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 equations reduce to:

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

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

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

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

Measurements of strain in sufficient orientations at any location are required for stresses at that location to be determined.

9.3 Stress state determinations

When the principal directions of the stress state are not known strain measurements in at least six independent orientations are needed to identify the complete strain state over a selected gauge volume representative of a given location. The stress components can be obtained from measured normal strains $\varepsilon_{\varphi\psi}$ using the formulation:

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

In equation (14) subscripts xy, xz and yz correspond to the shear components of stress. The angles are indicated in Figure 9.

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

9.3.1 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 equation (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 the simplified form of equation (14):

$$\varepsilon_{\varphi\psi} = \frac{1+\nu_{\mathrm{hkl}}}{E_{\mathrm{hkl}}}(\sigma_\varphi-\sigma_{\mathrm{zz}})\sin^2\psi - \frac{\nu_{\mathrm{hkl}}}{E_{\mathrm{hkl}}}(\sigma_{\mathrm{xx}}+\sigma_{\mathrm{yy}}+\sigma_{\mathrm{zz}}) + \frac{1+\nu_{\mathrm{hkl}}}{E_{\mathrm{hkl}}}\sigma_{\mathrm{zz}} \tag{15}$$

since

$$\sigma_\varphi = \sigma_{\mathrm{xx}}\cos^2\phi + \sigma_{\mathrm{yy}}\sin^2\phi + \sigma_{\mathrm{xy}}\sin^2\phi$$

Since in equation (15), the desired value, $(\sigma_\varphi-\sigma_{\mathrm{zz}})$, 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. [21]. 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 elasticity constants

The diffraction elastic constants, E_{hkl}, ν_{hkl}, are required by equation (8) to equation (15). As explained in 6.3, it is possible only in special cases to use the 'macroscopic' values of the elasticity modulus, and Poisson’s ratio, that have been determined by usual mechanical methods. This is because the values of the diffraction elasticity 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 [2]. In case of crystallographic texture, the diffraction elastic constants are orientation dependent and such experiments shall be performed in sufficient number of directions. If no experimental data can be made available, estimates should be obtained based on appropriate models [3-7, cf. 6.3].

If the required diffraction elastic constants are not available for the specimen material, they may be determined by a uniaxial loading experiment. Since 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 fitting).

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 elasticity 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 elasticity constants [8].

9.5 Data analysis

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 in strain determination can be compromised by improper consideration of the items listed below and in the appendices.

9.5.1 Peak fitting function

When a monochromatic beam is used the peak position is normally determined 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 [19], can be used to extract strains (see A.6.3).

9.5.2 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 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, it is recommended that a fixed gradient should be used. If the background is not constant the fitting function and its parameters should be stated.

9.5.3 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.

9.5.4 Distorted peak profiles

Unless proper corrections can be made, caution should be adopted in dealing with 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. In the study of multiphase materials, overlapping profiles are sometimes unavoidable. Multiple peak fitting strategies can be used for analysis based on procedures described in annex A.6.

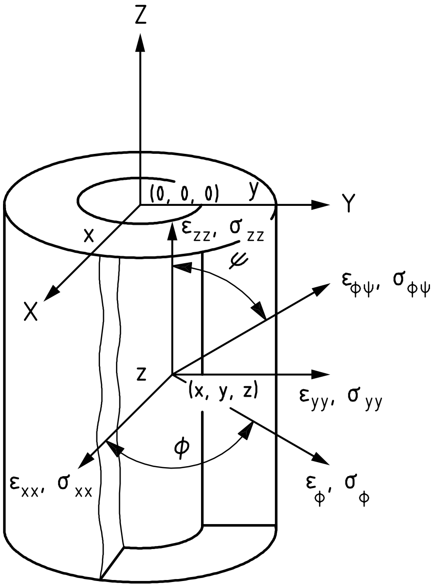


Figure 9 — Stress and strain components at a measurement point (x, y, z) in the specimen co-ordinate system (X, Y, Z)

10 Reliability of results

The determination of the uncertainty in a measurement is as important as the result itself, and without it the reliability of a measurement cannot be estimated. Clause 11 lists the quantities to be reported for a stress or strain measurement. For all these quantities the uncertainties shall be reported. It is recommended that such uncertainties are determined and reported in accordance with the ISO ‘Guide to the expression of uncertainty in measurement’ [22]. Additional guidelines are given in [23]. 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 Preamble

The basic reporting philosophy is to describe the experimental procedure adopted, the results of the measurements and how the data were analyzed. 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 will be subject to the agreement between client and provider.

11.2 Strain or stress values

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 measurements were made, i.e. the weighted centroid of the IGV or SGV;
- 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, shall be stated.

11.2.1 Stress free or reference lattice spacing

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

11.2.2 Conversion of strain to stress

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

11.2.3 Elasticity constants

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

11.2.4 Positioning

The uncertainty in 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:

- neutron source;
- instrument at source;
- wavelength and monochromator description (monochromatic instrument) or wavelength range (TOF instrument);
- instrument calibration procedure and calibration measurement results.

11.4 General measurement procedures

The following aspects of the measurements shall be reported:

- methods used to translate and orientate the specimen;
- method used to locate surfaces and other reference positions;
- manner in which the gauge volume(s) is determined;
- diffraction peak fitting function and procedure used;
- methods used to process data, e.g., smoothing, outlier elimination;
- method used to demonstrate reliability of the results.

11.5 Specimens/materials properties

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

- specimen geometry;
- composition;

- thermal/mechanical history;
- phases and crystal structures;
- homogeneity;
- sizes and shapes of grains, second phase particles or reinforcements;
- 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.

Annex A
(informative)

Measurement procedures

This annex to clause 8 presents procedures that are used by experienced practitioners to facilitate compliance with the Technical Specification.

Many of these procedures were clarified within two international pre-normative research projects (VAMAS TWA 20 [24-25] and RESTAND [26]). Further information is available in the following texts [27 – 30].

A.1 Specimen co-ordinates

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

A.1.1 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 orthogonal axes are appropriate.

EXAMPLE

Railway rails: longitudinal, transverse and vertical.

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

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

A.1.2 Specimens of irregular shape

In the general case of a specimen with 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.2 Positioning of the specimen

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

A.2.1 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.2.2 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. Positioning should be as accurate as is practicable, preferably to within 10 % of the relevant gauge volume dimensions. The location of the gauge volume in the measuring plane may be determined from the intensity profile of scattered neutrons as described in A.4.1.

A.2.3 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.1). Specimen positioning accuracy should generally be similar to that of the reference point. The specimen position may be determined by optical or mechanical means. The specimen can also be positioned relative to the gauge volume using wall scans. To define the specimen position it is helpful to have a fiduciary 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 $< \pm 0.1°$ (± 1.7 mrad). Specimen alignment is particularly important when scanning through steep strain gradients, interfaces, surfaces or when large translation scans are required.

NOTE 1 The limit of alignment by unaided eye by a skilled experimenter is $\approx 0.5°$ so optical or mechanical devices are essential when 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 of measured strain. 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 may 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 of 2000×10^{-6} per mm will result in a systematic uncertainty of $\pm 100 \times 10^{-6}$ in strain.

NOTE 3 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 through surface 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.

A.3 Number and location of strain measuring positions

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.3.1 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 about the specified location will be needed to assure reliability in the results.

A.3.2 Strain mapping

For 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 specific features as necessary. 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 adequate spatial resolution.

A.3.3 Material removal to facilitate measurements at difficult locations

The geometry of a specimen may make it difficult or impossible to perform measurements at particular locations of interest because of beam attenuation or because the specimen might not fit on the instrument used. In such cases the user could consider removing material from the specimen to overcome such problems. 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 redistribution.

A.4 Gauge volume

A.4.1 Instrumental gauge volume (IGV) determination

Because of beam divergence and other inevitable uncertainties of the experimental set-up, the IGV should be determined experimentally as illustrated in 6.5.

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 one beam, the other beam shall be sufficiently wide, 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 8). In case of a monochromatic instrument, the shape of the gauge volume depends on the scattering 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 instrumental gauge volume intensity profile may be illustrated by means of three 1-dimensional intensity profiles or a 3-dimensional intensity contour map. Such plotting also illustrates the level of beam uniformity across the beam width. However, for most practical purposes, the IGV may be described by just 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.4.2 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 [31] 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 previous methods by replacing the scatterer by a narrow slit and placing a detector behind it.

NOTE 1 The radial distance of incident and diffracted beam defining optics from the reference point should be set as appropriate. In the case of slits they 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.

NOTE 2 If the beam positioning reproducibility cannot be guaranteed after a gauge volume change the gauge positioning procedure shall be repeated.

A.4.3 Gauge dimensions

Gauge dimensions should be chosen so as to permit detail to be resolved as necessary. If gauge dimensions exceed the size of or distance between features of significance 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 instrumental gauge volume [32].

A.4.4 Grain size

Strain determination by diffraction methods in polycrystalline materials requires that there is a sufficient number of grains within the subset scattering into the detector. If only a few grains are sampled significant systematic errors in peak position will be introduced. Increasing the gauge volume dimensions as much as is practicable, commensurate with spatial resolution and attenuation considerations, may improve the statistical averaging but may also increase the peak position uncertainty due to coupling between position and angular sensitivity of the detector. Oscillation of limited amplitude of the specimen may be a more satisfactory way of increasing the number of contributing grains to produce a better average rather than increasing the gauge volume size.

A.4.5 Attenuation

Neutron attenuation can cause shifts in peak position, which unless accounted for, result in apparent strains that are not caused by the stress state. With reference to Figure 8 it is clear that neutrons scattering from different parts of the gauge volume can have different path lengths. For a specimen with significant attenuation the centroid of the SGV can be shifted from the centroid of the IGV because more detected neutrons come from the parts of the SGV for which the total path length is shortest. Generally, this is only a concern in reflection geometry (Figure 8) unless the specimen shape itself is asymmetric with respect to the neutron paths.

For monochromatic instruments, as explained elsewhere, there is in fact a spread of wavelengths about its mean. Furthermore, this spread of wavelengths is not uniform across the beam in the plane of diffraction. Consequently, the mean wavelength of the diffracted neutrons for highly attenuating materials can be different from the mean wavelength of neutrons in the incident beam. This effect leads to an incorrect determination of *d*-spacing and thus strain.

These effects are of particular concern in the case of measurements, which include surfaces and interfaces (see A 5.3) [33].

A.4.6 Counting time

The statistical quality of the data is a function of the number of neutrons counted which is related to the size of the SGV and the counting time. To minimize counting times, and background noise effects, gauge dimensions should be made as large as is practicable commensurate with spatial resolution, grain size and attenuation considerations.

A.5 Other gauge volume considerations

A.5.1 The SGV and its position

When scanning through surfaces or interfaces the proportion of the IGV occupied by the specimen material (or materials) varies, as does the shape of the occupied portion as the surface or interface is traversed. Consequently the centroid of the SGV is not at the centroid of the IGV, and erroneous strain values may result. Geometrical corrections for the offset of the SGV centroid from the reference point should be made when scanning through surfaces or interfaces.

Where appropriate, the effect of attenuation should be taken into account for the reasons described in A.4.4.

A.5.2 Instrument induced aberrations

Instrumental characteristics can affect peak shifts. Furthermore, although PSDs record peaks faster than single detectors, their use may introduce errors in the strain measured if the observed data are not properly interpreted. The reason is, that the detected peak position is sensitive to the shift of the centroid of the SGV. In contrast, high resolution single detector instruments generally exhibit lower sensitivity to the offset of the centroid of the SGV from the reference point than that of instruments employing PSDs. This is of particular concern in the case of scanning through surfaces and interfaces.

At monochromatic instruments using crystal monochromators, there is a relation between the angle at which a neutron enters the gauge volume and its wavelength. When scanning through surfaces or interfaces the gauge volume is not fully immersed in the phase under investigation. A slit, used as a beam defining device of the incident beam, covers a part of the monochromator and therefore reduces the angular range for neutrons to enter the SGV. Therefore the wavelength-band and the mean wavelength in the SGV change with the measuring position. This leads to changes in detected peak width and peak position. Errors in strain, induced by this effect can be considerable, and exceed the strain present [34].

On the diffracted beam side a slit can truncate the peak shape, which leads to false detected peak width and peak position.

A.5.3 Techniques to reduce aberrations

Slits, used as beam defining optics, should in general be positioned as close as possible to the gauge volume in order to minimize the above described instrumental aberrations. This is not always possible due to sample size and shape limitations.

A reliable way to reduce this error is to introduce a radial focusing collimator in the incident beam of a crystal monochromator instrument. This collimation ensures the same wavelength band at each point in the IGV. Thus, for any measurement position the same mean wavelength is obtained as if the gauge volume were fully immersed. Measurements based on this set-up only suffer from the much smaller geometrical effect, which depends mainly on the dimension of the diffracted beam width [32].

On the diffracted beam side, peak truncation can be avoided by using such a radial collimator. This transmits the diffracted peak on the PSD without distortions and allows peak shape analysis even at surfaces [33].

Another approach to correct for such errors is to simulate the experiment by computer models [35]. Although such models are improving, it is always good practice to reduce instrumental errors as much as possible.

A.6 Data analysis

For monochromatic beam instruments the study of single isolated peaks is sought. In this case fitting the diffraction line profile with a Gaussian function is generally quite effective. In some cases only overlapping diffraction line profiles are available. The analysis of overlapping diffraction line profiles should be treated with care. The following techniques have been used under appropriate conditions with some success.

A.6.1 Analysis of two overlapping diffraction lines

In the case of two overlapping peaks, such as are shown in Figures A.1 and A.2, it is sometimes possible to extract an accurate d-spacing from a least-squares fit to that part of the data where the peak of interest dominates. In practice, a full analytical peak profile is fitted to a part of an experimental peak profile to estimate the centroid of the full experimental peak profile. Some suggestions have been made concerning the conditions under which such an analysis can be applied [36]. Some of the relevant parameters, which affect the reliability of the fit, are the relative FWHMs and intensities of the two peaks, the separation of their centroids, and the background level.

A.6.2 Analysis of several overlapping diffraction lines

It is possible to fit parts of a diffraction pattern containing several overlapping diffraction lines by analyzing that part of the diffraction pattern. In general the uncertainty in the result of the fitting procedure is increased and the reliability of the results decreases. However, if some of the fitting parameters can be fixed or related to each other, reliability can be improved. For instance, if the volume fractions of the phases of a two phase material and the relative peak intensities in the constituent phases are known, the intensity ratio of their diffraction patterns can be calculated and fixed.

A.6.3 Full pattern analysis

In situations where multiple diffraction lines have been recorded, as is routine at a TOF source, it is common practice to carry out a full pattern analysis in addition to, or instead of, a single or multipeak analysis. A typical full profile analysis method is that originally proposed by Rietveld [19]. In a Rietveld refinement the crystal structure(s) of the material is (are) assumed, and from this a diffraction pattern is predicted. The predicted pattern is compared with the measured pattern and the crystal structure is varied in a least squares procedure in order to optimise agreement between the measured and the calculated patterns.

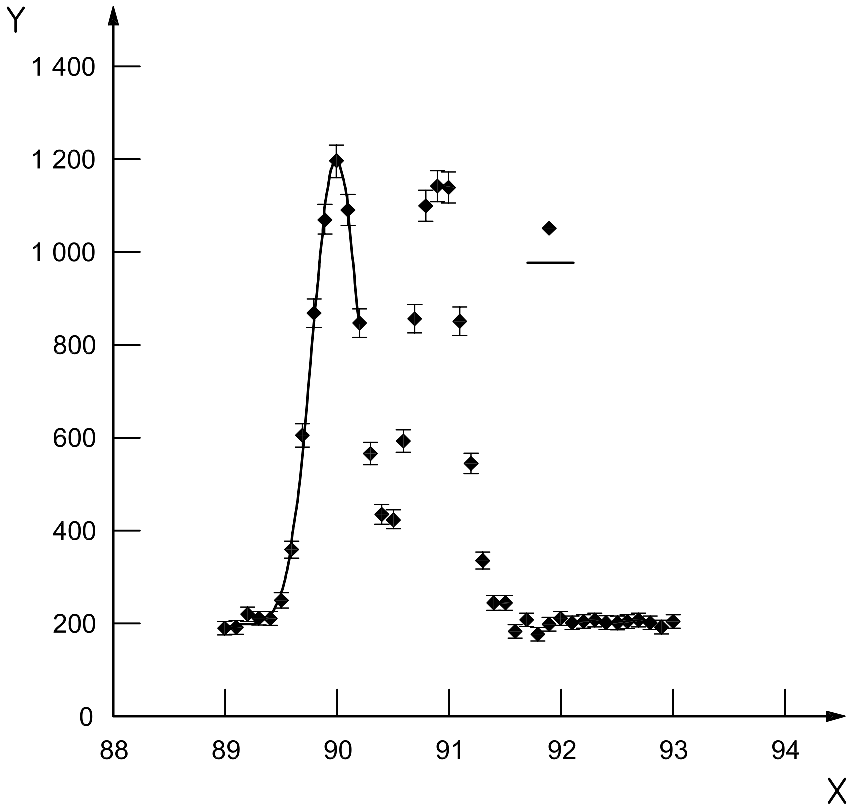
The advantage of a full pattern analysis is that by simultaneously fitting the entire pattern a lower uncertainty in the parameter values determined can be obtained, compared to fitting individual diffraction lines. In addition, information on the microstructure can be obtained, such as the presence of texture, volume fractions of phases and strain anisotropy. This requires the inclusion of appropriate models that incorporate the microstructure into the refinement procedure. Finally, it is observed that the lattice parameter obtained by Rietveld refinement is generally insensitive to the plastic anisotropy effects described in 6.3.

When using a Rietveld refinement the elasticity constants for determination of stress from strain are the bulk Young's modulus and Poisson's ratio adjusted to account for the texture present. In cubic materials strain is determined directly from eq. (6) using the lattice parameter value obtained by the refinement process. This is not the case in lower symmetry materials, where values for more than one lattice parameter are obtained from the refinement process. In this case a texture weighted strain parameter should be determined (cf. 6.3).

A.7 Temperature measurement and recording

Lattice spacing is affected not only by stress, but also by temperature. Thermal expansion is a property characteristic to each material and a change in temperature results in a "thermal strain", which is indistinguishable from a mechanical strain. The linear coefficient of thermal expansion, α, for many engineering materials lies within the range of ca.$10\text{-}20 \times 10^{-6}$ K^{-1}. Thus temperature variations of as little as 5 to 10 °C could induce strain in the range of 50 to 200×10^{-6}. Therefore, for all strain measurements it is important to monitor and control the temperature such that variations do not cause significant changes in the lattice dimensions.

NOTE Temperature variations may also have an influence on performance of the measuring equipment.

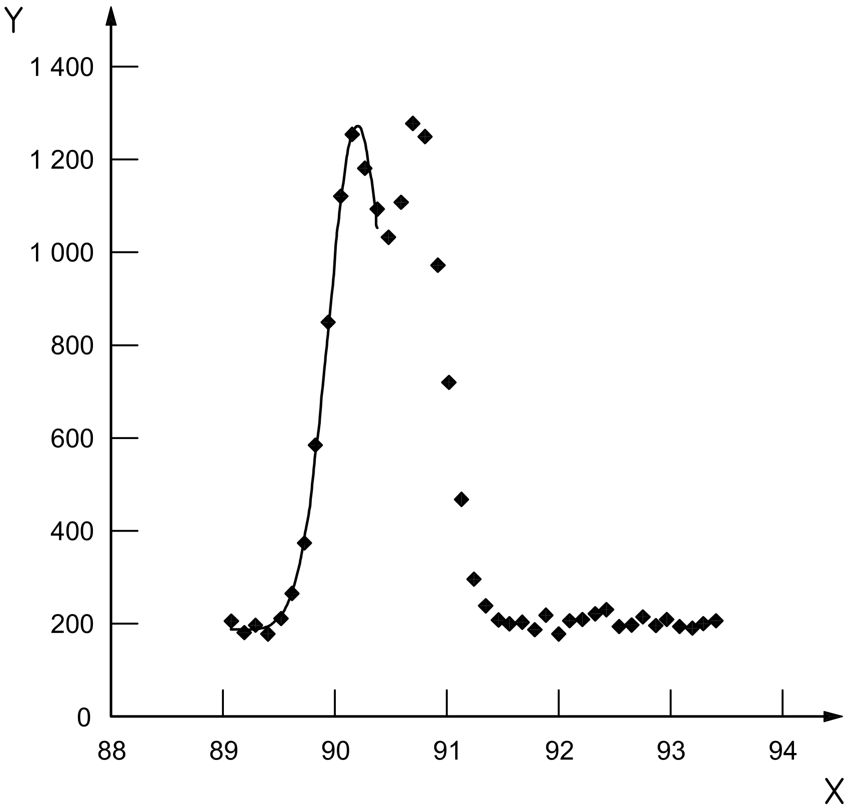


Key

X 2θ
Y count

◆ simulated data
—— fitted result

Figure A.1 — Example of peaks with little overlap, where the line profile parameters can be determined independently



Key

X 2θ

Y count

◆ simulated data

—— fitted result

Figure A.2 — Example of diffraction lines with extensive overlap, where the line profile parameters may not be determined independently

Annex B
(informative)

Determination of uncertainties in a measurand

B.1 Introduction

The uncertainty in a measured parameter is an estimate of the lack of knowledge in the true value of the parameter. There are many possible sources of uncertainty, after corrections for known systematic effects have been made.

The experimenter shall make an assessment of the potential contributions to the uncertainty in the measurement. If the assessment suggests that the contribution could have a significant influence on the uncertainty, it shall be reported and the uncertainty should be estimated. A detailed description of the determination of uncertainty is given in [22,23].

The accumulated value of uncertainty *u* in an estimated parameter *y* is obtained from the values of all the independent uncertainties *u(x)* contributing to the measurement:

$$u^2(y) = \sum_{i=1}^{N} \left(\frac{\partial y}{\partial x_{\mathrm{i}}}\right)^2 u^2(\times_{\mathrm{i}}) \tag{B.1}$$

where x_{i} are the N parameters, on which *y* depends and u^2 is the variance.

B.2 Uncertainty in stress determination

Stress depends on strain and elastic constants (see clause 9). Therefore the uncertainties in strain and the elastic constants are needed for the estimation of the uncertainty in the stress value reported.

For instance, the variance of the stress component (σ_{xx}) computed from equation (8) is given by:

$$\begin{aligned} u^2(\sigma_{\times\times}) &= \left(\frac{\partial\sigma_{\times\times}}{\partial\varepsilon_{\times\times}}\right)^2 u^2(\varepsilon_{\times\times}) + \left(\frac{\partial\sigma_{\times\times}}{\partial\varepsilon_{\mathrm{yy}}}\right)^2 u^2(\varepsilon_{\mathrm{yy}}) + \left(\frac{\partial\sigma_{\times\times}}{\partial\varepsilon_{\mathrm{zz}}}\right)^2 u^2(\varepsilon_{\mathrm{zz}}) \\ &+ \left(\frac{\partial\sigma_{\times\times}}{\partial E_{\mathrm{hkl}}}\right)^2 u^2(E_{\mathrm{hkl}}) + \left(\frac{\partial\sigma_{xx}}{\partial \nu_{\mathrm{hkl}}}\right)^2 u^2(\nu_{\mathrm{hkl}}) \end{aligned} \tag{B.2}$$

B.3 Uncertainty in strain determination

Factors, which may affect the uncertainty in strain measurements, include:

- uncertainty in measurement of d, θ or t and their respective stress free (d_0, θ_0 or t_0) or reference values (d_{ref}, θ_{ref} or t_{ref});
- uncertainty in wavelength of the incident beam;
- variations in temperature or chemical composition of the specimen as a function of position or time;
- uncertainty in measurement position within the specimen.

Causes of these uncertainties include: movement of optical components or detector, non-uniformity in wavelength of incident beam, attenuation in the specimen, incomplete filling of the gauge volume and temperature variations.

Based on equation (B.1) the estimated variance in strain $u^2(\varepsilon)$ is given by:

$$u^2(\varepsilon) = \sum_{i=1}^{N} (\frac{\partial \varepsilon}{\partial x_i})^2 u^2(x_i) \qquad \text{(B.3)}$$

where x_i are the N parameters which have influence on the strain, each with their estimated variance $u^2\ (x_i)$.

Based on equation (2), equation (B.3) yields

$$u(\varepsilon)^2 = \frac{u(d)^2}{{d_0}^2} + \frac{u(d_0)^2}{{d_0}^2} \approx \frac{u(d)^2}{d^2} + \frac{u(d_0)^2}{{d_0}^2} \qquad \text{(B.4)}$$

NOTE If the same reference is used to determine a series of strain measurements, their *relative* uncertainties are given simply by:

$$u(\varepsilon)^2 \approx \frac{u(d)^2}{d^2} \qquad \text{(B.4a)}$$

That is, when determining relative uncertainties, the uncertainty in the reference is not included in the calculation of uncertainty in strain. Instead it should be reported separately as an uncertainty of the “zero point” of the strain scale. Thus, the relative changes in strain are better defined than the absolute values of strain, which is made clear by reporting the uncertainties separately.

B.4 Uncertainty in lattice spacing

Using Bragg’s law (1), equation (B.1) yields

$$u(d)^2 = (\frac{\partial d}{\partial \lambda})^2 u(\lambda)^2 + (\frac{\partial d}{\partial \theta})^2 u(\theta)^2 \qquad \text{(B.5)}$$

Moreover, the uncertainty in position x and temperature T can also influence the uncertainty in the observed lattice spacing. Thus, the estimate of the total uncertainty in the measured lattice spacing becomes:

$$u(d)^2 = (\frac{\partial d}{\partial \lambda})^2 u(\lambda)^2 + (\frac{\partial d}{\partial \theta})^2 u(\theta)^2 + (\frac{\partial d}{\partial T})^2 u(T)^2 + (\frac{\partial d}{\partial x})^2 u(x)^2 \qquad \text{(B.6)}$$

NOTE For cubic materials it is allowed to replace the lattice spacing d with the lattice parameter a in equation (B.5) and equation (B.6).

The contribution from the uncertainty in temperature to the uncertainty in d is proportional to the coefficient of thermal expansion α of the material.

A contribution from the uncertainty in positioning may be significant in regions of high strain gradient and a series of measurements may be required for that to be assessed.

B.5 Example

Based on the above analysis the cumulative uncertainty in strain is calculated below for a hypothetical measurement on an aluminium specimen:

- a diffraction peak in aluminium is measured to be at $\theta = 39.850° \pm 0.008$. The uncertainty of 0.008 is that obtained from the least squares peak fitting routine;
- the reference peak position is 39.900°± 0.005;
- the wavelength is 0.30000 ± 0.00001 nm. This 0.00001 nm uncertainty is believed to include all instrumental uncertainties;
- the uncertainty in the temperature during the d measurement is ± 3 °C;
- the uncertainty in the temperature during the d_0 measurement is ± 1 °C;
- the uncertainty in the position of the specimen is ± 0.10 mm in each of the x, y and z directions;
- the strain gradient g is estimated to be 500×10^{-6} mm in the x direction, and negligible in the y and z directions;
- the coefficient of thermal expansion, α is 24×10^{-6} °C;
- for this particular experiment, all other effects have been assessed as having a negligible influence on the measurements.

In this case equation (B.6) becomes:

$$\frac{u(d)^2}{d^2} \approx (\frac{u(\lambda)}{\lambda})^2 + (u(\theta)\cot\theta^2 + (u(T)\alpha^2 + (u(\times)g)^2 \qquad \text{(B.7)}$$

while for the independently measured d_0 we find for u (d_0):

$$\frac{u(d_0)^2}{{d_0}^2} \approx \left(\frac{u(\lambda)}{\lambda}\right)^2 + \left(u(\theta_0)\cot\theta_0\right)^2 + \left(u(T)\alpha\right)^2 \qquad \text{(B.8)}$$

The strain gradient term is absent in equation (B.8) as a d_0 measurement should not be allowed at a location exhibiting either strain or strain gradients.

Calculating the strain from equation (2) and the uncertainty from equation (B.4), equation (B.7) and equation (B.8), the example yields a strain of $1045 \pm 222 \times 10^{-6}$. The uncertainty in strain is 192×10^{-6} if the uncertainty in the reference measurement is not included in the calculation (equation (B.4a) and equation (B.7)).

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