TECHNOLOGY TRENDS ASSESSMENT

ISO/TTA 3

First edition
2001-09-15

Polycrystalline materials — Determination of residual stresses by neutron diffraction

Matériaux polycristallins — Détermination des contraintes résiduelles par diffraction neutronique

Reference number
ISO/TTA 3:2001(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

ISO/TTA

Page

FOREWORD

The International Organisation for Standardisation (ISO) is a worldwide federation of national standards bodies. It is responsible for preparing International Standards through ISO technical committees in association with other international organisations and national governmental and non-governmental agencies.

The Versailles Project on Advanced Materials and Standards (VAMAS) supports trade in high technology products through international collaborative projects aimed at providing the technical basis for drafting codes of practice and specifications for advanced materials. The scope of the collaboration embraces all agreed aspects of enabling science and technology which are required as a precursor to the drafting of standards for advanced materials. The VAMAS activity emphasises collaboration on pre-standards measurement research, intercomparison of test results, and consolidation of existing views on priorities for standardisation action.

ISO Technology Trend Assessment (ISO/TTA) documents are published under a memorandum of understanding concluded between ISO and VAMAS. They enable the technical innovations and developments emerging from a VAMAS activity to be published at an early stage prior to their incorporation into a Standard. Whilst ISO/TTAs are not Standards, it is intended that they will be able to be used as a basis for standards development in the future by the various existing standards agencies.

This particular ISO/TTA reports the findings of a comprehensive 'round-robin' study which was carried out by VAMAS Technical Working Area (TWA) 20 to investigate the feasibility of measuring residual stresses in crystalline materials by neutron diffraction. It was supported by a European (EU) project RESTAND (Residual Stress Standard using Neutron Diffraction) aimed at demonstrating that the techniques developed can be applied to real components.

EXECUTIVE SUMMARY

Neutron diffraction is a relatively new method for determining residual (and applied) stresses in crystalline materials. It is similar to the X-ray technique for surface determinations. However because neutrons are not charged, neutron diffraction can be used to obtain residual stresses non-destructively to a depth of several centimetres in most materials of practical interest. No standard is currently available for making these measurements.

An international project, under the auspices of VAMAS (Versailles Agreement on Advanced Materials and Standards), Technical Working Area 20 (TWA 20) was initiated in January 1996 to carry out the under-pinning research necessary to develop a standard. The investigation involved most of the neutron sources worldwide which are capable of making the measurements. A series of 'round-robin' specimens including a shrink-fit aluminium alloy ring and plug assembly, a ceramic matrix composite, a nickel alloy shot-peened plate and a ferritic steel weldment were examined. This study was supported by a European (EU) project RESTAND (Residual Stress Standard using Neutron Diffraction) which was started in December 1997 to demonstrate the usefulness of the technique to a range of practical applications and to develop confidence in the method for industry.

This document gives the background to the VAMAS TWA 20 and RESTAND projects. It outlines the main findings and indicates the precautions that are required to achieve accurate positioning and alignment of specimens (and components) in the neutron beam and the analysis required to obtain reliable results. It also shows that special attention is needed in dealing with near-surface measurements because of surface aberration effects. It is demonstrated that, provided the recommended procedures are followed, a positional tolerance of $\pm 0{,}1$ mm can be achieved with an accuracy in strain of $\pm 10^{-4}$ to give a resolution in residual stress of ± 7 to 20 MPa in most materials of practical interest.

BACKGROUND

Neutron diffraction is a technique that can be applied for determining residual (and applied) stresses in crystalline materials [1-3]. With the method, elastic strain is measured and stress calculated using the elastic properties of the material. The depth to which these measurements can be made non-destructively within specimens or components depends on their size and shape. It is also dependent on the neutron scattering and absorption characteristics of the materials of which they are made. Typically the depths to which these measurements can be obtained are up to a few centimetres in most materials of practical interest.

No standard or code of practice is available for making residual stress measurements by neutron diffraction. As a consequence VAMAS TWA 20 (Versailles Agreement on Advanced Materials and Standards, Technical Working Area 20) was set up in January 1996 with the aim of carrying out the under-pinning research necessary for preparing a standard. The specific objectives of TWA 20 were to:

- establish accurate and reliable procedures for making non-destructive residual stress measurements by neutron diffraction,
- examine a selection of samples in which residual stresses had been introduced by different techniques,
- conduct inter-laboratory comparisons to establish reproducibility,
- assemble the necessary information for preparing a draft standard for making the measurements.

A European (EU) project RESTAND (Residual Stress Standard using Neutron Diffraction) was also started in December 1997 to demonstrate the application of the technique to industrial situations. This ISO/TTA presents the findings of these two investigations. It includes a draft procedure which can be used for making the measurements until a standard has been developed. Relevant committees concerned with the preparation of this standard are ASTM E 28.13, CEN/TC 138 AHG 7 and ISO/TC 135/SC 5.

Both the VAMAS TWA 20 and RESTAND investigations involved a series of 'round-robin' experiments. These were carried out by making measurements on the same samples at a number of neutron sources. Most of the sources world-wide that are capable of making the measurements participated. A list of the participants contributing to VAMAS TWA 20 is given in Table 1. Those participating in RESTAND are included in Table 2.

For the VAMAS TWA 20 project, four types of 'round-robin' sample were examined. These were a shrink-fit aluminium alloy ring and plug, a ceramic matrix composite, a nickel alloy shot-peened plate and a ferritic steel weldment. These examples were chosen to establish the range of application of the technique. They were investigated in the order mentioned. In each case a protocol was specified which each participating group was required to follow. Measurements were made at each neutron source independently. The results were then collected together and statistical analyses carried out to determine the reliability of the

measurements. Data were collected on steady state instruments which used a monochromatic beam of neutrons and also on time-of-flight instruments which employed a pulsed polychromatic beam. With a monochromatic source, measurements are made on specific crystallographic planes; with the time-of-flight method the entire spectrum can be analysed using profile refinement [4] to obtain strains. It has been found that comparable results are obtained from each type of instrument.

The ring and plug assembly was the first specimen to be measured because residual stresses had been introduced into it elastically, a discontinuity is obtained at the ring/plug interface and comparisons can be made with theory. The ceramic matrix composite was chosen to determine the feasibility of making measurements in a dual phase system containing fine fibres. The shot-peened plate was selected to establish whether steep stress gradients (of the order of 2000 MPa/mm) can be measured close to external surfaces and the ferritic weldment to determine whether reliable results can be obtained through regions of different microstructure (and possibly chemical composition).

The studies on the ring and plug assembly established the basic procedures that should be followed. The findings are contained in VAMAS report no. 38 [5]. It has been found that it is essential to ensure accurate positioning and alignment of a specimen in the neutron beam for reliable results to be obtained. A suitable shape and size of 'gauge volume' over which individual measurements should be made to achieve adequate resolution in regions of strain gradients has been identified. It is recommended that a minimum size of 8 mm^3 is adopted to encompass sufficient grains and to give neutron counting times which are not too long. In some cases cube-shaped sampling volumes are required. When there is no strain gradient in one direction a 'match-stick' shape, with the axis aligned along the direction of zero strain gradient, can be employed. If there is no strain gradient in two directions a plate-shaped volume can be used. For the shot-peened plate it has been established that steep stress gradients approaching 2000 MPa/mm can be measured with a 1x1x10 mm^3 'match-stick' sampling volume with its axis aligned in the plane of the plate. In regions away from interfaces and steep gradients a 3x3x3 mm^3 volume can be used. In the absence of stress gradients, such as may exist in the presence of uniform applied loads, the entire specimen may be bathed.

From statistical analysis of the data, it has been established in most cases, that a positional accuracy with a standard deviation of 0,1 mm can be achieved provided proper alignment procedures are adopted. It has also been ascertained that strains can be recorded away from surfaces to a tolerance of $\pm 10^{-4}$ corresponding to a stress of $\pm$ 7 to 20 MPa in most materials. Close to surfaces (or interfaces) and regions of variable microstructure, greater errors can be expected. Where the volume from which neutrons are being counted is traversed through a surface, there is the possibility that compensation is needed for the change in shape and size of the volume of material being sampled affecting the position at which the strain measured should be recorded. This is particularly important in the presence of steep stress gradients in highly absorbing materials where there are significant differences in neutron path lengths through different regions of the volume of material being examined. In this case, it is necessary to establish the neutron intensity weighted centroid of the material cross-section being measured and record the strain at this location. In traversing regions of variable microstructure and/or chemical composition it may be required to make allowance for a change in stress-free crystal lattice spacing with position.

A main aim of the RESTAND project was to develop industrial confidence in the application of the neutron diffraction technique for residual stress measurement. Measurements have been made on felt and fibre-reinforced composites for heat insulation and thermal shock resistance, on deep-rolled crankshafts to represent complex shapes, a quenched component and through fusion, linear-friction and friction-stir welds for power generation and aerospace applications. For composites, with fibre and matrices of similar composition, it has been found that it is sometimes necessary to separate out the effects of overlapping peaks. With complex shapes such as crankshafts, care is needed to avoid orientations which involve long neutron path lengths to minimise attenuation.

Similarly curved surfaces can exaggerate surface aberrations. In all cases, it has been determined when using a monochromatic beam of neutrons, that measurements should be restricted to those planes which give high peaks close to a diffraction angle of 90° and which represent bulk material behaviour. It is also recommended that a check is made for force and moment equilibrium, where possible, to provide additional confidence in the results.

The remainder of this document contains the proposed protocol for making the measurements. It includes the scope of the method, an outline of the technique, the calibration and measurement procedures recommended, and details of how the strain data should be analysed to calculate stresses and establish the reliability of the results obtained.

REFERENCES

[1] Allen, A, Andreani, C, Hutchings, M.T. and Windsor, C.G. 'Measurements of internal stress within bulk materials using neutron diffraction' NDT International 14, 1981.

[2] Stacey, A., MacGillivray, H.J., Webster, G.A., Webster, P.J. and Ziebeck K.R.A. 'Measurement of residual stresses by neutron diffraction' J. Strain Analysis, I.MechE 20, 1985.

[3] Hutchings, M.T. and Kravitz, A.D. (Eds) 'Measurement of residual and applied stress using neutron diffraction' Proc. NATO ARW Oxford, March 1991, Kluwer, Netherlands 1992.

[4] Rietveld, H.M. 'A profile refinement method for nuclear and magnetic structures' J. App. Crystal 2, 1969, 65-71.

[5] Webster, G.A. (Ed) 'Neutron diffraction measurements of residual stress in a shrink-fit ring and plug' VAMAS report no. 38, Jan. 2000.

Participants in VAMAS TWA 20

G. A. Webster (Chairman)	Imperial College, London, UK.
R. C. Wimpory (Secretary)	Imperial College, London, UK.
P. Brand	NIST, Gaithersburg, USA.
M. A. M. Bourke	LANSCE, Los Alamos, USA.
M. Ceretti	LLB, Saclay, France.
M. R. Daymond	ISIS, Rutherford Appleton Laboratory, UK.
L. Edwards	Open University, Milton Keynes, UK.
J. H. Fox	CRL, Chalk River, Canada.
T. Gnäupel-Herold	University of Maryland / NIST, Gaithersburg, USA.
T. M. Holden	CRL, Chalk River, Canada.
C. R. Hubbard	ORNL, Oak Ridge, USA.
D. J. Hughes	University of Salford, Salford, UK.
M. T. Hutchings	AEA Technology plc, Abingdon, UK.
M. W. Johnson	ISIS, Rutherford Appleton Laboratory, UK.
A. Krawitz	University of Missouri, Missouri, USA.
A. Lodini	LLB, Saclay, France.
T. Lorentzen	Riso National Laboratory, Roskilde, Denmark.
P. Lukas	NPI, Prague, Czech Republic.
G. Mills	University of Salford, Salford, UK.
N. Minakawa	JAERI, Japan.
C. Ohms	JRC, Petten, Netherlands.
M. Ono	KURSS, Kyoto University, Japan.
J. Pang	University of Manchester/UMIST, Manchester, UK.
E. A. Payzant	ORNL, Oak Ridge, USA.
R. L. Peng	Studsvik NRL, Studsvik, Uppsala University, Sweden.
T. Pirling	ILL, Grenoble, France.
H. J. Prask	NIST, Gaithersburg, USA.
H. G. Priesmeyer	LANP, GKSS, Geesthacht, Germany.
A. Pyzalla	HMI, Berlin, Germany.
W. Reimers	HMI, Berlin, Germany.
R. B. Rogge	CRL, Chalk River, Canada.
J. Root	CRL, Chalk River, Canada.
S. Spooner	ORNL, Oak Ridge, USA.
A. Venter	Safari Atomic Energy Corporation, Pretoria, South Africa.
D. Q. Wang	Studsvik NRL, Studsvik, Uppsala University, Sweden.
X. L. Wang	ORNL, Oak Ridge, USA.
P. J. Webster	University of Salford, Salford, UK.
R. A. Winholtz	University of Missouri, Missouri, USA.
P. J. Withers	University of Manchester/UMIST, Manchester, UK.
J. S. Wright	ISIS, Rutherford Appleton Laboratory, UK.
T. Youtsos	JRC, Petten, Netherlands.

Participants in RESTAND

T. Youtsos (Co-ordinator)	JRC, Petten, Netherlands.
C. Ohms (Secretary)	JRC, Petten, Netherlands.
C. Achmus	Volkswagen AG, Wolfsburg, Germany.
K. M. Beinborn	Sintec Keramik GmbH, Halblech (Buching), Germany.
N. Bonner	Rolls Royce, Derby, UK.
R. Burguete	BAE Systems, Bristol, UK.
E. Calvet	European Commission, Brussels, Belguim.
M. R. Daymond	ISIS, Rutherford Appleton Laboratory, UK.
L. Edwards	Open University, Milton Keynes, UK.
A. Hewat	ILL, Grenoble, France.
D. J. Hughes	University of Salford, Salford, UK.
M. T. Hutchings	AEA Technology plc, Abingdon, UK.
M.W. Johnson	ISIS, Rutherford Appleton Laboratory, UK.
T. Lorentzen	Riso National Laboratory, Roskilde, Denmark.
P. Lukas	NPI, Prague, Czech Republic.
G. Mills	University of Salford, Salford, UK.
E. Oliver	University of Manchester/UMIST, Manchester, UK.
J. Pang	University of Manchester/UMIST, Manchester, UK.
R. L. Peng	Studsvik NRL, Studsvik, Uppsala University, Sweden.
T. Pirling	ILL, Grenoble, France.
A. Pyzalla	HMI, Berlin, Germany.
W. Reimers	HMI, Berlin, Germany.
P. Schmidt	Schunk Kohlenstofftechnik GmbH, Heuchelheim, Germany.
G. A. Webster	Imperial College, London, UK.
P. J. Webster	University of Salford, Salford, UK.
R. Weiss	Schunk Kohlenstofftechnik GmbH, Heuchelheim, Germany
R. C. Wimpory	Imperial College, London, UK.
P. Withers	University of Manchester/UMIST, Manchester, UK.
J. S. Wright	ISIS, Rutherford Appleton Laboratory, UK.

Introduction

Neutron diffraction is a non-destructive method 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 test pieces to be transported to a neutron source. Measurements of the lattice spacing or lattice parameter are obtained which are then converted to strain and stress.

POLYCRYSTALLINE MATERIALS - DETERMINATION OF RESIDUAL STRESSES BY NEUTRON DIFFRACTION

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

1. Scope

1.1 This Technology Trends Assessment specifies a test method for determining residual stresses in polycrystalline materials by neutron diffraction. It may be applied to homogeneous and inhomogeneous materials and to test pieces containing distinct phases.

1.2 The principles of the neutron diffraction technique are outlined. Advice is provided on the crystalline 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.

1.3 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.

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

1.5 The methods of determining individual elastic strains 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. Symbols and abbreviations

2.1 Symbols

Symbol	Definition	Units
a,b,c	Lattice parameter The value of the lengths of the sides of a unit cell	nm
a_0, b_0, c_0	Strain-free lattice parameter	nm
B	Background The value of counts that constitutes the height of the background on a neutron detector	-
d	Lattice spacing The perpendicular distance between adjacent parallel lattice planes (crystallographic planes), also called d-spacing	nm
d_0	Strain-free lattice spacing	nm
E	Elastic modulus	GPa
E_{hkl}	Diffraction elastic modulus	GPa
hkl, hkil	Miller indices of crystallographic plane	-
H	Peak height This is the height of the Bragg peak above that of the background	nm
I	Integrated neutron intensity above background	-
k_i, k_d	Incident and diffracted neutron wave-vectors	nm^{-1}
L	Neutron attenuation length	mm
N	Number of measurements	-
N_n	Total number of neutrons counted	-
Q	Scattering vector ($k_d - k_i$)	nm^{-1}
t	Time-of-flight of neutrons from source to detector	µs
u, u_c	Standard and combined standard uncertainty	-
u_d, u_{d0}	Uncertainty in d and d_0, respectively	nm
u_λ, u_θ	Uncertainty in λ and θ, respectively	nm
s_ε	Measured standard deviation in strain	-
S_1, S_2	Elastic compliance constants	MPa^{-1}
w	Slit width	mm
x,y,z	Coordinate axes (relevant to sample)	-
Y	Measurand, the quantity being measured.	-
Δa, Δb, Δc	Change in lattice parameter	nm
Δd	Change in lattice spacing	nm
Δt	Change in time of flight of neutrons from source to detector	µs
$\Delta\theta$	Change in Bragg angle	degrees, rads
$\Delta\lambda$	Change in wavelength of neutrons	nm
ε	Strain	-
ε_{ij}	Components of strain tensor	-
λ	Wavelength of neutrons	nm
ν	Poisson's ratio	-

Ω	Angular rotation about reference point The angular motion of the goniometer of the diffraction instrument in the scattering plane	degrees
σ	Stress	MPa
σ_{ij}	Components of stress tensor	MPa
σ_Y	Yield stress	MPa
θ	Bragg angle	degrees, rads
θ_0	Strain-free Bragg angle	degrees, rads
$\theta, \phi, \psi, \omega$	Angular rotations	degrees, rads

Subscripts

a,c	Relevant to lattice parameter
hkl, hkil	Relevant to crystallographic plane
xx, yy, zz	Relative to Cartesian co-ordinate axis
ϕ, ψ	Relevant to strain axis
0	Strain-free reference
ref	Reference value

Reference point
Location in space at which all measurements are made. This will normally correspond with a point on the axis of rotation of the diffractometer.

2.2 Abbreviations

DED	Detector to reference point distance	mm
DEC	Diffraction elastic constant	GPa
DSD	Detector slit to reference point distance The distance from the centre of the exit slit (or equivalent optic) to the reference point.	mm
DSH	Detector slit height The height of the exit slit (or equivalent optic).	mm
DSW	Detector slit width The width of the exit slit (or equivalent optic).	mm
FWHM	Full width at half maximum The width of the diffraction peak at half the maximum height above the background	degrees, µs, nm
ISD	Incident slit to reference point distance	mm
ISH	Incident slit height	mm
ISW	Incident slit width	mm
PSD	Position sensitive detector	-
TOF	Time-of-flight The time-of-flight of neutrons from source to detection	µs
IGV	Instrumental gauge volume	mm^3
NGV	Nominal gauge volume	mm^3
SGV	Sampled gauge volume	mm^3

3 Summary of method

3.1 Outline of Principle – Bragg's law

Neutron diffraction can be used to measure components of strain directly from changes in crystal lattice spacing. When illuminated by radiation of wavelength similar to interplanar spacings, crystalline materials diffract this radiation as distinctive Bragg peaks. The angle at which any given peak occurs can be calculated using Bragg's law of diffraction,

$$2d_{hkl}\, sin\theta_{hkl} = \lambda \qquad (1)$$

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

3.2 Neutron sources

Neutrons can be generated by fission or spallation: the former is predominantly employed in reactor and the latter in spallation sources. In both cases the neutrons produced are moderated to bring their energies to the thermal ($\leqslant$ 100meV) range. At reactor sources, a continuous monochromatic beam of neutrons is usually produced by using a crystal monochromator to select a given neutron wavelength from a "white" polychromatic neutron beam by Bragg diffraction. At spallation sources, the neutron beam usually consists of a series of short pulses containing a spread of wavelengths that arrive at the sample over a very small period of time (of order 20 ms). The energy (and therefore wavelength) of each neutron can be determined on detection from the distance it has travelled to the detector and the time taken to do so, called the time-of-flight (TOF). TOF measurements are, therefore, energy dispersive, with the entire diffraction pattern recorded at any particular scattering angle.

3.3 Strain measurement

If a sample is illuminated by a monochromatic beam of neutrons, then its lattice spacing can be determined if the incident wavelength of the diffracting neutrons is known. When a specimen is stressed, its lattice spacing is altered. Any elastic strain will therefore be apparent as a shift in the value of $2\theta_{hkl}$ for a particular reflecting plane illuminated by a fixed wavelength. Differentiation of equation (1) gives:

$$\Delta\theta_{hkl} = -\left(\frac{\Delta d}{d_0}\right) tan\theta_0 \qquad (2)$$

where $\Delta\theta_{hkl}$ and Δd are the changes in the Bragg angle and lattice spacing, respectively, from their corresponding values θ_0 and d_0 for the stress-free sample of the material. The strain in the hkl set of planes is therefore given by:

$$\varepsilon = \left(\frac{\Delta d}{d_0}\right) = -\Delta\theta \cot\theta_0 \tag{3}$$

where $\Delta\theta$ is in radians. The direction in which strain is measured is along the scattering vector Q, which is perpendicular to the diffracting planes as shown in Figure 1.

At a time-of-flight (TOF) instrument, neutron pulses, each with a continuous range of velocities and therefore wavelengths, are directed at a specimen. By measuring the flight times t of detected (diffracted) neutrons, their wavelengths are calculated and diffraction spectra recorded. The incident spectra are polychromatic, thus all possible lattice planes are recorded in each measurement. The scattering vectors for all reflections recorded in one detector lie in the same direction, and thus measure the strain in that same direction. Each reflection is produced from a different family of grains, that are oriented such that a specific hkl plane diffracts to the detector. Strain can then be calculated from the shift in a given reflection in a manner analogous to that described in equation (3) to give:

$$\varepsilon_{hkl} = \frac{\Delta t}{t_0} = \frac{\Delta d}{d_0} = \frac{\Delta\lambda}{\lambda_0} \tag{4}$$

where a different value of strain will be obtained for each hkl peak.

However, since an entire diffraction spectrum is obtained in each measurement direction, more usually the strain is determined from the whole diffraction pattern using a Rietveld [1] style analysis where a crystallographic model of the structure is fitted to the entire diffraction spectrum. Strain is then given by:

$$\varepsilon = \frac{\Delta a}{a_0} \tag{5}$$

where Δa is the change in lattice parameter, from the lattice parameter a_0 of a stress-free sample of the material.

3.4 Neutron Diffractometers

A typical diffractometer used for stress measurement at a continuous source is shown schematically in Figure 2. The white neutron beam is first monochromated to a chosen wavelength by Bragg diffraction from a suitable single crystal monochromator. It is normal to arrange that this beam passes over the centre of rotation of the sample table (the reference point for the instrument) about which the neutron detector also rotates. The beam is diffracted from the sample to a detector. Both the incident and diffracted beams are given spatial definition by the use of suitable beam optics to

produce a beam of controlled, usually rectangular, dimensions. A multi-detector or position sensitive detector (PSD) may be used instead of a rotating single neutron detector to detect the whole diffraction peak simultaneously. A typical diffraction peak from a monochromatic instrument is shown in Figure 3.

At pulsed sources, each pulse provides a diffraction profile across a very large range of lattice spacings. As a fixed scattering angle is used, most instruments at spallation sources use radial focussing collimation that allows neutrons to be detected over a wider solid angle than would be possible using a traditional slit, yet ensuring that all collected neutrons come from the defined sampling volume. The signal from the individual elements of the focussing collimator is combined by software taking into account the slightly different diffraction angles. Multiple radial collimators are often used to enable more than one Q (strain) direction to be measured simultaneously. A typical diffractometer used for strain measurement in two perpendicular directions simultaneously at a pulsed spallation source is shown in Figure 4. A typical diffraction spectrum 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.

3.5 Stress determination

Stress and elastic strain are second rank tensors which are related through the elastic constants of a solid. Since neutron diffraction can be used to determine the elastic strain within a defined volume in a crystalline solid, it is possible to calculate the stress in that volume provided the relevant material elastic constants are known. Determination of the full strain tensor requires measurements of the elastic strain along at least six independent directions. If the principal strain directions within the body are known this can be reduced to three orthogonal directions. For plane stress or plane strain conditions, a further reduction to two directions is possible. For uni-axial loading measurement along one direction is sufficient.

The need to measure the strain in a given volume along a number of directions leads to the requirement that the specimen is accurately positioned with respect to the neutron beam collimation and the detectors. This is usually accomplished by mounting the specimen on motorised linear translation and rotation tables. By sequentially moving the specimen through this volume, which is fixed in space by the intersection of the incident beam and detector collimation, the spatial variation in elastic strain and, following measurement in other directions, stress can be mapped within a specimen or component.

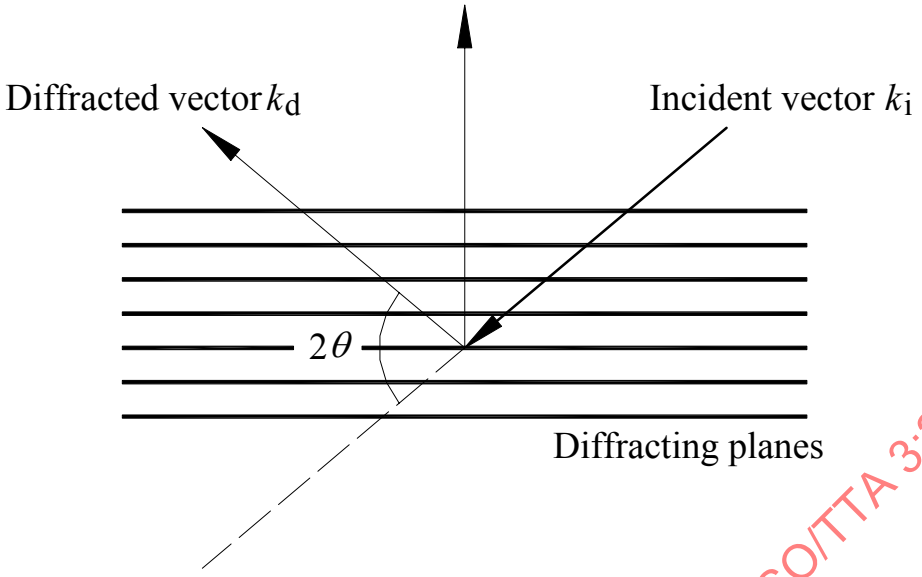


Figure 1 Schematic illustration of Bragg scattering geometry

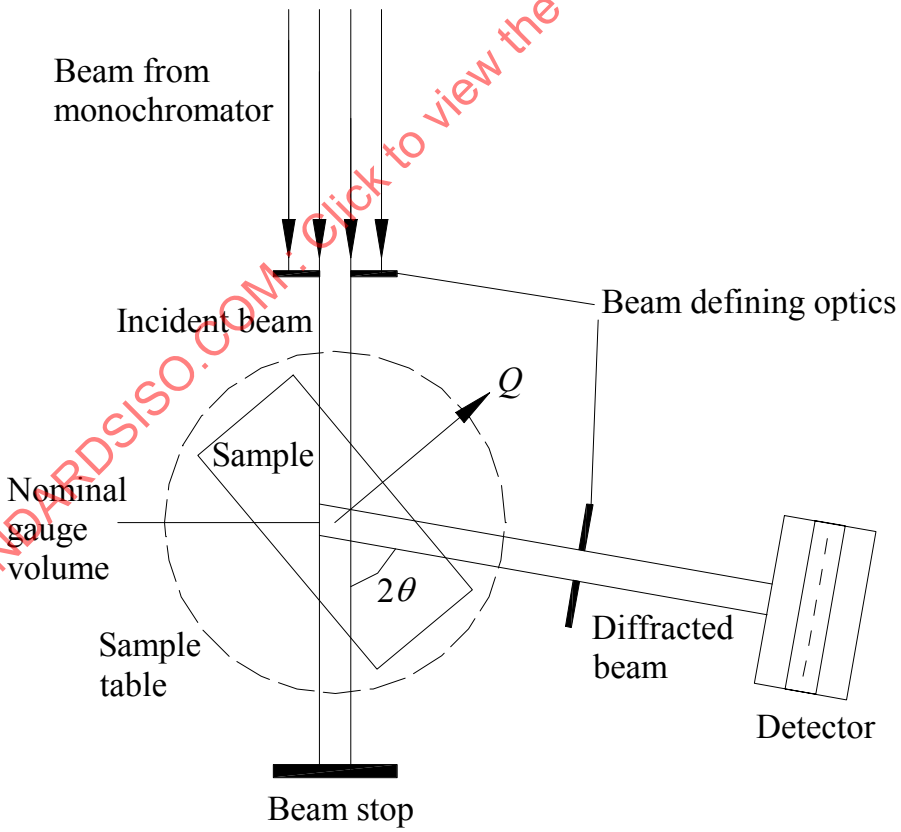


Figure 2 Schematic illustration of a reactor based diffractometer for stress measurement

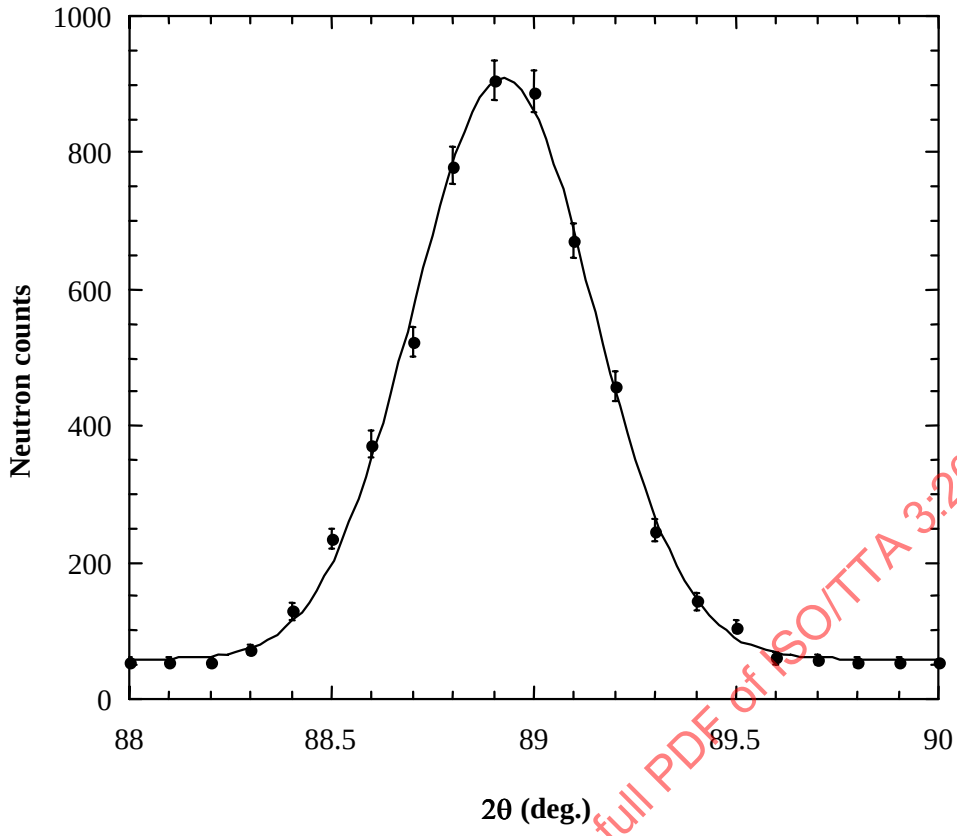


Figure 3 Typical Bragg peak from a reactor based diffractometer fitted with a Gaussian distribution

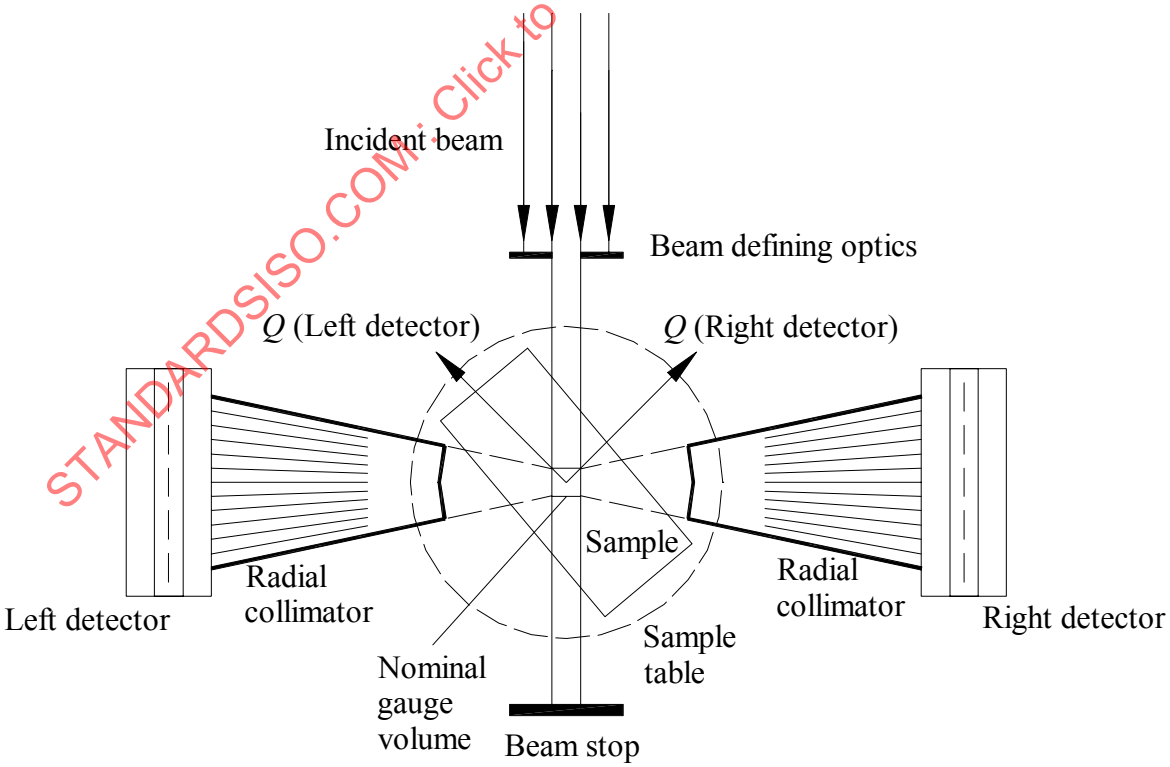


Figure 4 Schematic illustration of a spallation source diffractometer for stress measurement

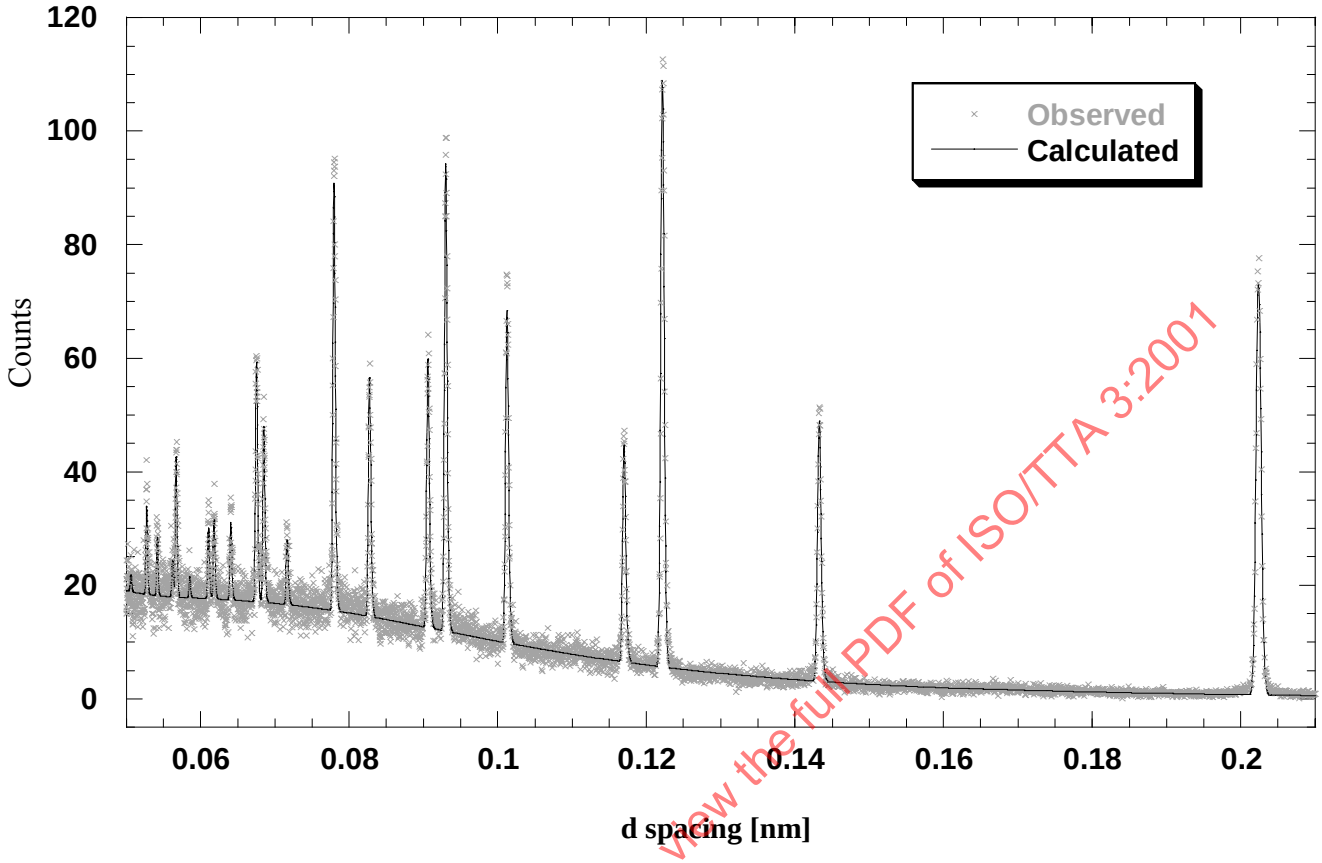


Figure 5 Typical diffraction spectrum from a pulsed spallation source showing the Rietveld (calculated) fit to measured data

4 Calibration procedure

A number of steps shall be carried out prior to experimental measurements being made on a sample.

4.1 Calibration of diffractometer

It is necessary to align and calibrate the diffractometer. At a monochromatic source, the detector angular response should be calibrated. At a time-of-flight source, the combined flight path and detector angular response should be calibrated. In both cases this is done using a typically stress-free sample with a known and well defined lattice parameter, typically silicon, ceria or alumina. These materials are chosen because they scatter neutrons well, and produce sharp diffraction peaks since they have small intrinsic sample peak widths. At a time-of-flight source it is also necessary to calibrate the detector efficiency as a function of wavelength. One way of doing this is to use a sample which predominantly scatters neutrons by a diffuse process such as vanadium. Quantification of the uncertainty in strain measurement is important. Repeated measurement on a reference sample is a method of quantifying the uncertainty in strain measurement.

4.2 Choice of diffracting plane

For the determination of macroscopic elastic strains, consideration shall be given to intergranular anisotropy. Due to the anisotropy of individual crystallites in a material the different lattice reflections exhibit a range of responses to a macroscopic stress field. In the macroscopically elastic regime these effects are linear. This means that any lattice reflection can be chosen for macroscale stress determination within this region. The stiffness of the plane (i.e. the diffraction elastic constant for the particular lattice plane) will *not* be the single crystal stiffness, but some polycrystalline average (see also clause 7). In essentially randomly textured materials the Kröner approach is often used [2]. A good approximation to the Kröner value is frequently the average of the Reuss [3] and Voigt [4] values. A description of these approaches is given in [5]. In non-randomly textured materials, a self-consistent approach to stiffness determination can be used, see for example [6].

In the plastic regime, a non-linear elastic strain to stress response can occur with certain diffraction peaks introducing more anisotropy between the behaviour of different crystallographic lattice planes. When determining macroscale stresses it may be desirable to minimise such effects, in others cases they may be the subject of study. Therefore for various classes of symmetry materials, advice is given as to the most appropriate lattice reflections to choose to provide small intergranular strains[1], and therefore good representations of the macroscale stress field. In general a linear elastic strain response with macroscopic applied stress is desired for the measurement plane when macrostresses are to be determined. If a suitable plane is not known, or a new material is being examined, this linearity can be determined by *in situ*

[1] Intergranular strain is defined as the difference in the strain obtained from a particular hkil diffraction peak with that strain due solely to the plane specific elastic modulus (diffraction elastic constant). In a material deforming purely elastically, intergranular strains are thus zero [10, 11].

measurements on a tension (or compression) sample at a selection of applied loads which extend into the plastic regime. Examples of the test method that can be applied are described in the references given in Table 1.

Table 1 Behaviour of diffraction planes for different symmetry materials, with specific examples

Material	**Recommended planes** – *small intergranular strains*	**Problematic planes** – *large intergranular strains*
fcc (Ni [7], Fe [8], Cu [9])	111, 311, 422	200
fcc (Al [10], [11])	111, 311, 422, 220	200
bcc (Fe [11])	110, 211	200
hcp (zircaloy [13], Ti [12])	Pyramidal (10-12, 10-13)	basal (0002) and prism (10-10, 1-210)
hcp (Be [14])	2nd order pyramidal (20-21, 11-22)	basal, prism and 1st order pyramidal (10-12, 10-13)

In the case of highly textured samples, the choice of plane may be different. Plastic deformation caused by highly tri-axial stress histories will also affect intergranular strains. Where intergranular strains are felt to be of concern, consideration should be given to carrying out measurement on several diffraction peaks.

4.3 Full pattern analysis

At a time-of-flight instrument entire diffraction patterns can be obtained. It is therefore typical to carry out a Rietveld [1] analysis on the entire pattern, to obtain a smaller uncertainty in lattice parameter for a given neutron count time. For cubic materials the lattice parameter obtained from such fits has been shown to be independent of intergranular effects [15] to several percent plastic strain, i.e. it is linear as a function of stress. The modulus appropriate for such a fit is the macroscopic Young's modulus. For non-cubic materials it is necessary to identify a suitable linear parameter e.g. in some hexagonal materials it has been found that an appropriately linear parameter is $(2\ \varepsilon_a + \varepsilon_c)/3$, where '$\varepsilon_a$' and '$\varepsilon_c$' are the strains determined in the '*a*' and '*c*' lattice parameters respectively [14]. It is also possible to carry out single peak analysis in the same way as occurs at monochromatic neutron sources.

4.4 Positioning procedures

The initial diffractometer calibration procedure requires the positioning of the centroid of the nominal neutron measurement gauge volume (see 4.5) at a known position, which is defined as the reference point. This reference position is normally along the centre of rotation of the positioning table.

Accurate positioning is required, as described in clause 6. The level of accuracy required depends to some extent on the type of measurement 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 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 neutron scans (see clause 6). All three methods are capable of determining the position of a sample edge relative to the neutron beam to an uncertainty of 0,1 mm.

4.5 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 for both the incident and diffracted neutrons [Figure 6 a)]. The centroid of the NGV is the geometric centre of this volume, and is usually coincident with the reference point (see 4.4).

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 incident beam intensity profile [Figure 6 b)]. The IGV can be defined by experiment or by simulation calculations, and will typically be expressed in terms of the neutron intensity distribution. Traditional methods of measurement of the instrumental gauge volume involve scanning a small probe e.g. a thin (0,25 mm) wire[2] through the instrumental gauge volume. The dimensions can also be defined in terms of a FWHM; whatever practice is adopted should be specified. The difference between the instrumental and nominal gauge volumes may be particularly evident when only small volumes are being sampled. The centroid of the IGV is the centre of this volume, weighted for the beam intensity profile. For most instruments it is coincident with the centroid of the NGV. Note that the IGV and NGV are only properties of the diffractometer itself.

Finally, the sampled gauge volume (SGV), sometimes called the sampling gauge volume is that part of the instrumental gauge volume from which measurements are obtained in an actual experiment (see Figure 7). It is the volume over which the strain measurement is averaged. It is affected by:

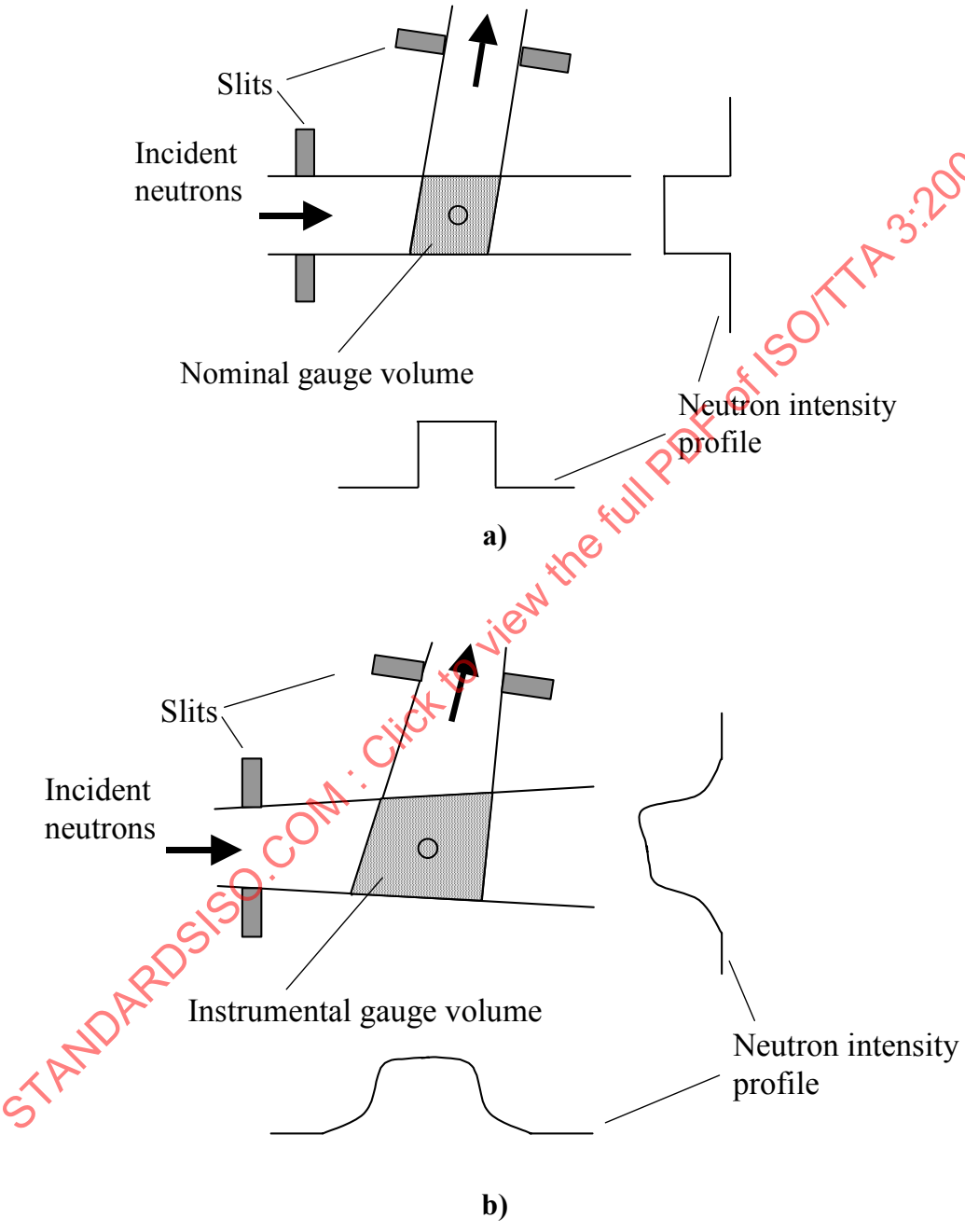
- partial filling of the instrumental gauge volume,
- attenuation of neutrons within a specimen,
- the wavelength distribution of neutrons across the incident beam.

If the IGV is filled by a non-absorbing material the SGV and IGV are equivalent. The centroid of the SGV is the centre of the gauge weighted by the above effects. When the IGV absorption is strong, or if the IGV is not fully filled, the centroid of the SGV will be at a different position to that of the IGV as shown in Figure 7.

The SGV and its centroid should be determined for each measurement. The position at which the average strain in this volume is obtained is the centroid of the SGV. It is important that the measured strain is reported at this position. The effect of the

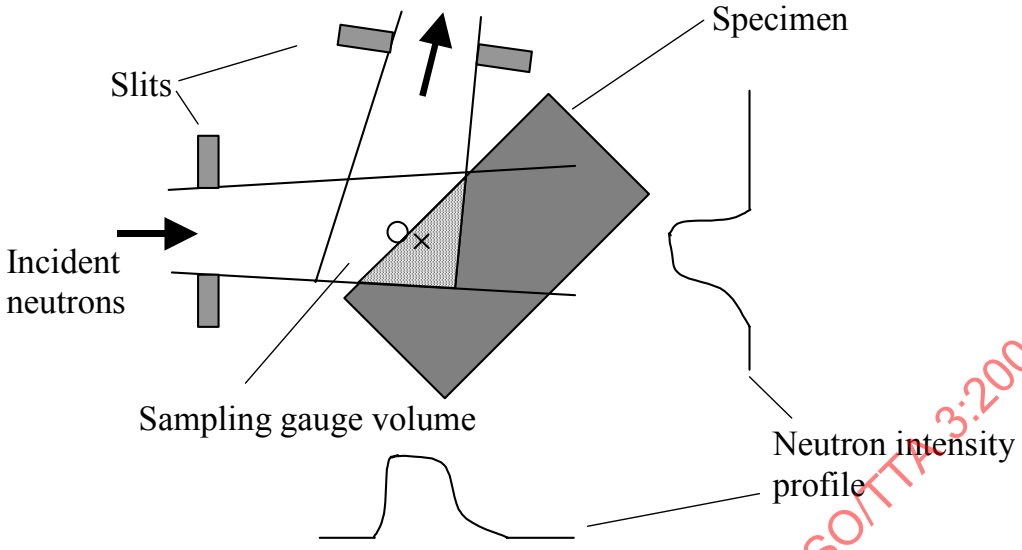
[2] This wire may be of nylon in situations where the diffracted beam is well collimated. In configurations using a single slit in front of a position sensitive detector arrangement, the wire shall be one producing intensity by elastic scattering (Bragg scattering). In this case a stainless steel or copper wire may be used. It is also possible to scan a thin metal sheet through the beam, with its normal parallel to the diffraction vector. The dimensions of the probe object shall be sufficiently small, otherwise attenuation corrections will be required. Plotting of the integrated scattered intensity as a function of position gives a measure of the instrument gauge volume. Such a map will also illustrate the level of beam uniformity across the incident beam width.

weighting will be most significant close to surfaces in highly absorbing materials. If the difference in the IGV and SGV centroid positions is significant in comparison to the size of the IGV, it may be necessary to make corrections to the lattice parameter obtained. The appropriate correction can be determined by experimental or theoretical means.



NOTE The centroid of each volume is indicated by the 'O'. Intensity profiles along the central cross sections are also shown.

Figure 6 Schematic showing plan views of the nominal and instrumental gauge volumes for a) parallel neutron beams and b) divergent beams



NOTE The 'O' indicates the centroid of the instrumental gauge volume, the 'X' the centroid of the sampling gauge volume; these are displaced relative to each other.

Figure 7 The sampling gauge volume for incomplete filling of the instrumental gauge volume, in the case of a high absorption material

4.6 Determination of a strain-free or reference lattice spacing

Since diffraction measurements give the lattice spacing, in order to determine strains it is necessary to have a reference lattice parameter, relative to which the strains can be determined. In some cases it may be possible to determine a strain-free lattice spacing d_0 (the lattice spacing in which the material experiences no strain). In other cases only a reference lattice spacing d_{ref} (the lattice spacing to which other measurements will be compared) will be possible or necessary. It should be noted that absolute 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.

The lattice parameter is sensitive to a number of effects, apart from macroscopic load and instrumental aberrations, and these shall be taken into account in the measurement of reference values. 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/time known to contain negligible stress.
- measurement on a powder, which is representative of the material being examined. This is 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 variations through a weldment to be obtained.

- 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, or when measuring individual stresses in multi-phase materials. It is recommended however that experimental methods are used where possible, and that equilibrium is employed mainly as a check for consistency.
- 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 should be taken with the preparation of 'stress-free' material to avoid the introduction of residual stresses during manufacture.

5 Materials characterisation

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 the possibilities are cited below. In some instances, it may be appropriate to carry out preliminary diffraction measurements to establish the scope of the investigation.

5.1 Composition

5.1.1 Metals, alloys and ceramics

Standard alloy or material designations that indicate chemical composition and processing shall be cited. This information is required to estimate beam attenuation, diffracted beam intensity, background intensity, and possible issues with radioactive activation.

5.1.2 Composites

The matrix and reinforcement phases, indicating the alloy/composition of the matrix and the amount, form, and orientation of the reinforcement, shall be cited.

5.2 Thermal/mechanical history

Processing used to shape, form or join the specimen, including heat treatment, shall be cited. In the case of parts removed from service, the previous operating conditions should be given.

5.3 Phases and crystal structures

The phases in the alloys, ceramics, and composites shall be cited. The crystallographic form of phases used in the measurements shall be specified, if multiple forms exist.

5.4 Homogeneity

If information is known about any spatial variation in composition or phase distribution, it shall be cited. This may affect confidence in making measurements at a particular location in a specimen or component and whether taking the results to be representative of the specimen or component as a whole is valid. In particular, inhomogeneities in the structure and composition can lead to variations in the stress-free lattice spacing, as a function of position in the specimen (see 4.6).

5.5 Sizes and shapes

5.5.1 Grains

The number of grains in the gauge volume is important in determining the quality of the diffraction pattern. A grain size is considered too large when it leads to point-to-point fluctuations in diffraction peak intensity that is greater than the counting statistics; i.e. an insufficient number of grains are being sampled[3]. The grain size in relation to the gauge volume employed and the stress distributions measured shall be given.

5.5.2 Second phases and reinforcement

The shape, size, and orientation of second phases or composite reinforcements shall be cited. Large particle or reinforcement dimensions can result in point-to-point fluctuations in diffraction peak intensity and non-random orientation of second phases or reinforcements can lead to texture effects, as discussed below.

5.6 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 cited and, if possible, characterised.

---

[3] An effective way to determine if the grain size is sufficiently small is to repeatedly record the diffraction peak of interest, with 1-2° rotations of the sample about an axis that is normal to the plane of diffraction (the θ or ω axis) between recordings. The desired outcome is a diffraction peak intensity variation that is within counting statistical uncertainty. Grains that are too large will produce large intensity variations, i.e. variations that exceed those expected from counting statistics. Texture, on the other hand, causes a gradual change of peak intensity over small ranges of sample rotation.

6 Recording and measurement procedures

Informative supplementary comments to all parts of this section are provided in annex A.

6.1 Recording recommendations

The following information shall be recorded, and data archived, and be available, so as to enable conformity with the standard to be verified.

6.1.1 General Information – Instrument

Instrument related information:
a) project title, individual responsible, affiliation, date;
b) neutron source and location, name and type of instrument;
c) fixed source and instrument parameters that have relevance to strain measurements;
d) adjustable instrument parameters that have relevance to strain measurements;
e) nominal and instrumental gauge volume parameters;
f) Bragg peak parameters;
g) temperature ± variation.

6.1.2 General Information – Specimen

Specimen related information:
a) specimen material;
b) specimen shape and dimensions;
c) fiduciary marks or reference locations and specimen co-ordinates.

6.1.3 Specific information required for each strain measurement

Information related to specific measurements:
a) specimen orientation relative to the scattering vector Q ± uncertainty;
b) specimen and gauge volume positions relative to the reference point ± uncertainty;
c) strain ± uncertainty;
d) d-spacing ± uncertainty (if an absolute calibration is required).

For monochromatic instruments:
e) strained peak position 2θ ± uncertainty;
f) unstrained reference peak position $2\theta_0$ ± uncertainty;
g) peak shift $\Delta 2\theta$ ± uncertainty;
h) relevant peak parameters ± uncertainty.

For time-of-flight instruments:
i) strained time of flight t ± uncertainty;
j) unstrained reference time of flight t_0 ± uncertainty;
k) time of flight shift Δt ± uncertainty;
l) relevant peak parameters ± uncertainty.

6.2 Definition of co-ordinates

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

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

6.3 Positioning

The positions of a specimen and the gauge volumes shall each be defined relative to the instrument 'reference point' (see 4.4). The reference point position shall be defined as accurately as is practicable.

6.4 Measurement directions

If the principal strain directions are not known, measurements in a minimum of six independent orientations are required for full definition. If the three principal directions are known, three sets of measurements, usually one along each of the principal directions, are sufficient to define the strain/stress state.

6.5 Number and location of measuring positions

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

6.6 The gauge volumes

Gauge volumes are defined by collimating optics in the incident and diffracted beams, and the directions and divergences of those beams. The choice of gauge volume dimensions should 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.

6.7 Near-surface precautions

Corrections for sampling gauge volume centroid position shall be made when scanning through surfaces (or interfaces). Where appropriate, corrections for instrumental aberrations and attenuation should also be made.

6.8 Temperature

The specimen temperature shall be kept stable within a restricted range chosen so that changes in lattice dimensions due to temperature variations are small relative to the uncertainty specified for the strain measurement.

7 Calculation of stress

With neutron diffraction elastic strains are determined and stress 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. The only major alteration is the use of specific diffraction elastic constants rather than the overall aggregate average. Hence, for materials without preferred orientation the average materials constants in the generalized Hooke's law are simply exchanged for the appropriate diffraction elastic constants (E_{hkl}, ν_{hkl}). In this case the compliance constants S_1 and S_2 are related to the diffraction elastic constants through,

$$S_1 = \frac{1}{E_{hkl}}$$

$$\frac{S_2}{2} = \frac{(1+\nu_{hkl})}{E_{hkl}} \qquad (6)$$

These can be measured from simple uniaxial calibration experiments or they can be calculated [2-4].

The procedure for calculating stresses in isotropic materials is described in 7.1 to 7.3. For anisotropic or highly textured materials the choice of elastic constants required is described in 7.4.

7.1 Normal stress determination

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:

$$\sigma_{xx} = \frac{E_{hkl}}{(1+\nu_{hkl})(1-2\nu_{hkl})}\left[(1-\nu_{hkl})\varepsilon_{xx} + \nu_{hkl}(\varepsilon_{yy}+\varepsilon_{zz})\right] \qquad (7)$$

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

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

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

For plane stress conditions when one of these stresses (say σ_{zz}) is zero these equations reduce to,

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

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

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 equations (7) and (8), respectively, and σ_{zz} becomes,

$$\sigma_{zz} = \nu\left(\sigma_{xx} + \sigma_{yy}\right) \tag{12}$$

Calculations of stress are required for all locations where strain measurements have been made.

7.2 Stress state determinations

When the principal directions are not known strain measurements in at least 6 orientations are needed to identify the complete strain state over a selected gauge volume representative of a given location. For this case it is recommended that measurements are made along coordinate axes x, y and z at the point and along a separate set of coordinate axes x′, y′ and z′ at the same point. This second set of axes should be rotated as far as possible away from the first to optimise the sensitivity of the stress calculations. The normal stresses in each set of coordinate axes can then be calculated from equations (7) to (9) and transformation equations employed to determine the corresponding shear stresses and the magnitudes and directions of the principal stresses if required.

7.3 The $\sin^2\psi$ method

An alternative formulation, often used in X-ray diffraction investigations of stresses and strains [5], is based on a definition of the direction in which the strain is measured through the two angles ϕ and ψ as shown in Figure 8. Using this coordinate system, for the case when the z direction is one of the principal axes of deformation, i.e., the shear stresses $\sigma_{xz} = \sigma_{yz} = 0$, the expression relating the measured elastic strain $\varepsilon_{\phi\psi}$ as a function of the stress components in the sample coordinate system is given by,

$$\varepsilon_{\phi\psi} = \left(\frac{1+\nu_{hkl}}{E_{hkl}}\right)\left(\sigma_{\phi} - \sigma_{zz}\right)\sin^2\psi - \frac{\nu_{hkl}}{E_{hkl}}\left[\sigma_{xx} + \sigma_{yy}\right] + \left(\frac{1}{E_{hkl}}\right)\sigma_{zz} \tag{13}$$

from which the stress difference $\sigma_\phi - \sigma_{zz}$ can be deduced, as it is directly related to the slope of the measured strain data $\varepsilon_{\phi\psi}$ versus $\sin^2\psi$ plot.

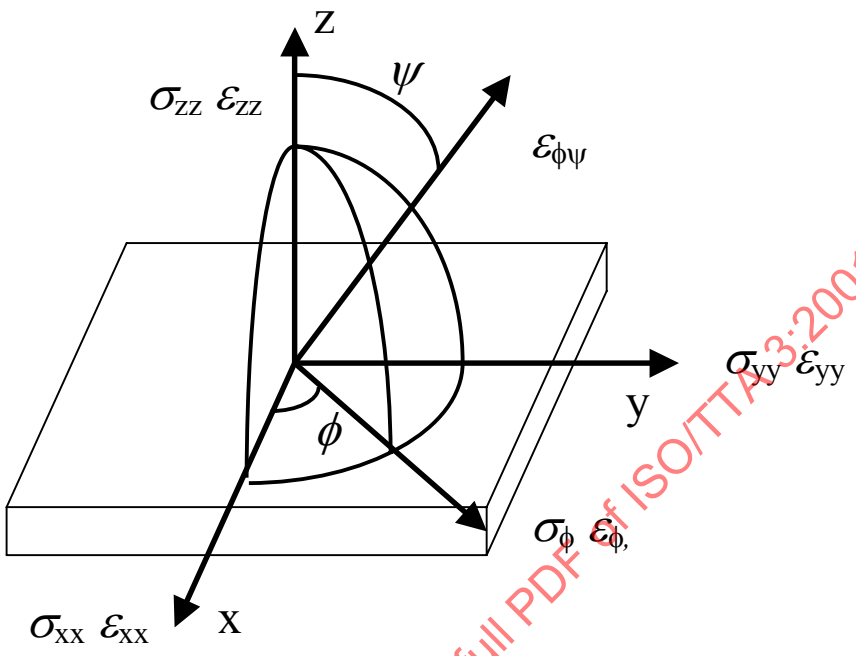


Figure 8 Coordinates for $\sin^2\psi$ method of determining stress state

7.4 Choice of elastic constants

Ideally, experimental values should be available, otherwise estimates should be obtained based on appropriate models. In the case of strong texture, modification of these estimates may be needed.

7.5 Data analysis

The position of a Bragg peak is determined by fitting the experimental data with a mathematical function, which corresponds to the peak shape of the diffracted spectrum, including a background.

7.5.1 Peak fitting function

At reactor sources, a monochromatic beam is normally used and the peak position is normally determined by fitting a Gaussian to the data.

At spallation sources, the peak shape is intrinsically asymmetric. The peak fitting function is normally a convolution of an exponential decay function into a Gaussian.

7.5.2 Background function

The background function used in the fits depends on the instrumental set-up and the types of neutron source. If the background function is not a constant, the parameters of the function should be calibrated and should be fixed in the fits.

Because the gradient of a sloping background and the peak position are correlated during curve fitting, care shall be taken with sloping backgrounds. Unless the background can be determined independently of the peak profile, a fixed gradient should be used.

7.5.3 Peak to background ratio

If the ratio of the peak count (background subtracted) to the background is less than twice that of the fitted peak position, it may be difficult to separate the peak position from effects caused by fitting the background.

7.5.4 Overlapping or asymmetrical peaks

Reflections with peak profiles containing overlapping asymmetric peaks should be avoided wherever possible. Asymmetry in a peak profile may arise due to sample dependent effects such as inhomogeneities and stacking faults, as well as for instrumental reasons. Where the origin and behaviour of this asymmetry is not understood, the use of asymmetric peaks should be avoided for strain measurements. In the study of multiphase materials, overlapping peaks are sometimes unavoidable. Double peak fitting strategies can be used for the analysis, but these can lead to large numbers of fit parameters and a large increase in their uncertainties. In such cases, the following strategies are often employed.

7.5.5 Truncated peak fit

The overlapping peak effect is eliminated by performing a single peak fit to an incomplete peak profile with the side of the tail that is influenced by the interfering peak truncated.

The optimum "cut-off" point on the contaminated side for a truncated peak fit is 0,5 FWHM from the peak position. The feasibility of this method depends on the peak separation, width and intensity ratios of the two peaks. Table 2 provides a guideline for the case where a truncated peak fit is valid.

Table 2 Guideline limit of applicability for a truncated peak fit. *H*, FWHM and 2θ denote the peak amplitude, width and peak position

H_2/H_1	$FWHM_2$ /$FWHM_1$	$(2\theta_2-2\theta_1)$ / $FWHM_1$	H_2/H_1	$FWHM_2$ /$FWHM_1$	$(2\theta_2-2\theta_1)$ / $FWHM_1$
0,1	0,1	>0,5	0,3	0,1	>0,5
0,1	0,5	>0,5	0,3	0,5	>0,5
0,1	1,0	>0,5	0,3	1,0	>0,5
0,5	0,1	>0,5	1,0	0,1	>0,7
0,5	0,5	>1,0	1,0	0,5	>1,0
0,5	1,0	>1,5	1,0	1,0	>1,5
Subscripts 1 and 2 refer to the peak of interest and the interfering peak respectively.					

7.5.6 Double peak fit

A double peak profile is fitted to the whole diffracted spectrum. In some cases, some of the fitted parameters can be fixed or related to each other. For instance, if the volume fraction of a two phase material is known, the intensity ratio of the two peaks can be calculated. A double peak fit is more likely to succeed if it is valid to apply constraints to some of the parameters.

8 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 9 lists the quantities to be reported for a stress or strain measurement. For all of the quantities the uncertainties should be reported, most noticeably strain and measurement position. It is recommended that such uncertainties are determined and reported in accordance with the ISO 'Guide to the expression of uncertainty in measurement' [16,17]. An abbreviated summary of the nomenclature and method of calculating the combined standard uncertainty (u_c) of a measurement is given in annex B.

9 Reporting

The basic reporting philosophy is that sufficient information be presented to enable the measurements and subsequent data processing to be reproduced. In this way the information necessary to understand, evaluate, and further interpret the results will be provided. A rigid reporting format is not put forth as there is great variability in materials, available information, and objectives of studies.

9.1 Strain and/or stress values

The strain or stress values resulting from the measurements, and their associated uncertainty estimates, shall be reported, as follows:

- the strain and/or stress components and the values determined;
- the locations at which measurements were made, i.e. the weighted centroids of the instrumental or sample gauge volumes;
- the size and shape of the instrumental or sample gauge volume;
- the uncertainty estimates shall delineate the types of uncertainties included;

9.2 Strain-free or reference lattice spacing

The values 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.

9.3 Conversion of strain to stress

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

9.4 Elastic constants

If the measured strains are converted to stresses, the elastic constants used shall be provided.

9.5 Neutron source and instrument

The following information shall be provided:

- neutron source,
- instrument at source,
- wavelength and monochromator description (reactor) or wavelength range (pulsed source).

9.6 General measurement procedures

The following aspects of the physical measurement 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 is achieved;
- uncertainties in positioning;
- diffraction peak fitting function and procedure used.

9.7 Materials characterisation

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

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

Annex A
(informative)

Recording and measurement procedures

If the spatial variation of strain is required, three parameters shall be determined with appropriate accuracy:

a) strain, from the shift of the peak $\Delta 2\theta$ or the change in the time of flight Δt;
b) the direction of the strain measurement;
c) the position within the specimen at which the measurement is made.

This annex gives details of parameters that should be measured and recorded. It also presents the procedures that are used by experienced practitioners to comply with this TTA.

A.1 Recording requirements

The following types of instrument, specimen and measurement information should normally be recorded to ensure conformance with the standard.

A.1.1 General information – Instrument

Parameters for monochromatic instruments:

a) monochromating crystal/reflection, type of detector, monochromator to reference point distance (DED), detector to reference point distance;
b) absolute or nominal wavelength and calibration procedure;
c) neutron optical parameters such as incident slit width (ISW), detector slit width (DSW), incident slit height (ISH), detector slit height (DSH), incident slit to reference point distance (ISD), detector slit to reference point distance (DSD);
d) vertical and horizontal gauge intensity profile;
e) Bragg peak parameters; number of points in peak, angular increment or bin size between points in peak;
f) temperature ± variation.

Parameters for time-of-flight instruments:

a) moderator to reference point distance, detector to reference point distance, type of detector, angular range of detector;
b) wavelength range, calibration procedure;
c) neutron optical parameters such as incident slit width (ISW), incident slit height (ISH), incident slit to reference point distance (ISD), mechanism and characteristics of mechanisms for defining diffracted beam volume (e.g. radial collimator);
d) vertical and horizontal gauge intensity profile;
e) number of Bragg peaks used in Rietveld refinement or *d*-spacing range used in refinement. Size of bin in time-of-flight.
f) temperature ± variation.

With both types of instruments, alternative beam defining optics other than slits may be used. In these cases the equivalent values for (ISW), (DSW), (ISH), (DSH), (ISD) and (DSD) shall be quoted.

A.1.2 General information – Specimen

Specimen material:
a) chemical composition, crystal structure, grain size and texture, neutron attenuation length;

Specimen shape and dimensions;
a) diagram of the specimen showing dimensions, fiducial marks or reference locations and specimen co-ordinates.

A.1.3 Specific information required for each strain measurement

Relevant peak parameters for monochromatic instruments, or time-of-flight instruments carrying out single peak fits:
a) FWHM ± uncertainty;
b) peak height H ± uncertainty;
c) background B ± uncertainty;
d) peak height/background ratio H/B;
e) number of neutrons counted in peak N_n.

Relevant peak parameters for time-of-flight instruments using Rietveld refinement:
a) peak shape used, and relevant description parameters including,
   i. width, as a function of wavelength, and
   ii. peak shape anisotropy
b) background fit used;
c) level of description of strain anisotropy in Rietveld refinement;
d) model of texture used in Rietveld refinement.

A.2 Definition of co-ordinates

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

A.2.1 Specimens with elements of symmetry

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

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

b) Cylindrical specimens: a combination of rectangular and orthogonal co-ordinates aligned along and perpendicular to the axis in the axial (a), radial (r) and hoop (θ) directions.

NOTE In this case the axial direction is unique for all locations within the specimen. The radial and hoop directions are respectively along and perpendicular to the particular diameter on which the points are located and consequently rotate around the axis for points located on other diameters.

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.

EXAMPLES

Railway rails: longitudinal, transverse and vertical.
Rods and wires: axial, radial and hoop.
Regular polygonal cross-sections: triangular, square, hexagonal etc.; axial, normal to faces, parallel to faces.

A.2.2 Distorted specimens

Many engineered components are made from originally rectangular or circular sections that have been distorted during manufacture or in service, often as a result of residual stresses. It may be appropriate to employ one co-ordinate system throughout or several systems that are suitable for a series of local scans. In these latter cases locally appropriate co-ordinate directions in distorted regions are normal to surfaces and in the most symmetrical in-plane directions.

A.2.3 The general case

In the general case of an irregular specimen for which the triaxial stress state is required, co-ordinates should be along the three most symmetrical, preferably orthogonal directions.

A.3 Positioning

The specimen and the gauge volumes should be positioned relative to the 'reference point' of the instrument. The reference point is usually a point on the central axis of the instrument about which the detector is rotated or upon which it is focussed if it is fixed in position.

NOTE An approved standard base-plate and sample mounting system designed for the accurate and efficient mounting of locating devices and samples is described in Annex C.

A.3.1 The reference point

The reference point position should be defined as accurately as is practicable, preferably to within 10 % of the minimum dimension of the gauge volume that is to be used. The position of the reference point may be determined by optical or mechanical means using the rotation of a small-diameter cylinder or wire to define the axis.

A.3.2 The gauge volume

The geometrical centre of the instrumental gauge volume should be positioned at the reference point. 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 neutrons scattered from the small-diameter cylinder or wire used to define the plane axis or from an alternative cylindrical crystalline or incoherent scatterer.

NOTE Positioning efficiency is generally optimised if the cylinder has a diameter equal to or less than the gauge volume width.

For slit-defined systems the intensity profile may be recorded for the incident beam with the cylindrical scatterer positioned on the axis as the incident slit is translated across the incident beam. The detector should preferably be at a near 90° diffraction

angle and the detector slit should be wide open. The incident slit should then be centred at the position corresponding to the central maximum of the intensity profile, allowance being made where necessary for beam attenuation. Alternatively the scatterer may be translated across the fixed incident slit and the slit then re-positioned so that the intensity is at the maximum when the scatterer is on the axis. After the incident beam has been positioned, the detector slit is usually set to the same width as the incident slit and a similar positioning procedure is adopted. The centre of the gauge volume is usually set to near the mid-height of the incident beam; the vertical location may be determined by vertically translating a horizontal rod with its axis along the scattering vector and determining the centre of the scattered intensity profile. Alternatively a horizontal slit may be vertically traversed through the reference point and the centre of the transmitted beam derived from the intensity profile of the straight-through or diffracted beams.

NOTE 2 Instruments should preferably be designed and constructed so that gauge dimensions may be varied automatically and precisely without the need for re-calibration. If the beam positioning repeatability cannot be guaranteed after a slit change, the gauge positioning procedure should be repeated.

A.3.3 The specimen

The location of the specimen should be described relative to the reference point and to the direction of the scattering vector using appropriate co-ordinates. 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 necessary to have a fiducial mark or reference point and directions marked on the specimen. The fiducial 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 $< \pm 0{,}1°$ ($\pm$ 1,7 mrads). The specimen should be aligned to a similar accuracy relative to the translator directions.

NOTE 1 Alignment is particularly important when long translations are required.

NOTE 2 The limit of alignment by unaided eye by a skilled experimenter is $\approx 0{,}5°$ so optical or precise mechanical devices are essential when aligning translators and specimens.

NOTE 3 Gauge volume and specimen positions are each defined with respect to the reference point but scattering is from the part of the specimen that is within the instrumental gauge volume. Any uncertainties in positioning the gauge volume relative to the reference point will produce corresponding systematic uncertainties in specimen relative to gauge volume positions. If the gauge volume is not precisely centred at the reference point, rotation of the specimen about the reference axis will also result in an effective displacement, as well as rotation, of the gauge volume position relative to the specimen. If the instrument design cannot guarantee gauge volume and specimen positioning to the required accuracy using optical or mechanical methods, the position of the gauge volume relative to the specimen surface should be verified using 'wall scans'. Wall scans can provide a peak intensity profile 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 should be noted that it is necessary to repeat wall scans for each measurement orientation, and at a number of locations along a surface, when area scanning or if there is significant translation parallel to that surface. Care should be taken when wall scanning surface-treated, textured and large grained and absorbing materials as the through-surface intensity profile for such materials can be substantially distorted from that expected from simple occupied volume considerations.

A.3.4 Positioning accuracy

The positioning accuracy required will depend upon the specimen being scanned and the strain gradient at the point and direction being measured. Uncertainties in positioning Δx lead to systematic uncertainties in strain $\Delta\varepsilon$ at a point given by $\Delta\varepsilon = (\partial\varepsilon/\partial x)\Delta x$. Positioning accuracy should be adequate to ensure that any systematic uncertainty in strain resulting from a positioning uncertainty should be less than or equal to the maximum statistical uncertainty specified for the measurement resulting from uncertainties in determining the position of the peak.

EXAMPLE

For a specified statistical uncertainty $\pm 100 \times 10^{-6}$ in a region of strain gradient $2\,000 \times 10^{-6}$ per mm the positioning uncertainty should be $\leqslant \pm 50$ µm.

A.4 Measurement directions

A.4.1 Specimens with elements of symmetry

For specimens with significant elements of symmetry, measurements made in the (usually three) symmetry directions can be sufficient in practice, though not absolutely, adequately to describe the stress state.

EXAMPLES

Rectangular specimens: Along orthogonal symmetry directions x, y, z.
Cylindrical specimens: Along axial (a), radial (r) and hoop (θ) directions.
Extended constant cross-section specimens: Parallel to the long axis and along orthogonal axes.

A.4.2 Distorted specimens

For distorted specimens, measurements may be made locally normal to the surface and in the most symmetrical in-plane directions or relative to one uniquely defined co-ordinate system as appropriate.

A.4.3 The general case

For the general case of an irregular specimen for which the triaxial stress state is required, measurements may be made in the three most symmetrical, preferably orthogonal, directions with three more in appropriate other directions well away from the first three directions.

NOTE Consideration should be taken of the consequences of long path lengths and attenuation when choosing additional measurement directions.

A.5 Number and location of 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.

NOTE 1 Sharply varying strain patterns require more data points than smooth patterns with little significant detail.

NOTE 2 For efficient strain mapping it may be advisable 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.

A.5.1 Measurements at one location

If strain data are required at just one specified location, they may be obtained from a single measurement at the specified point but with limited experimental evidence of statistical and positioning uncertainties. If repeated measurements are made at the point they will enable an experimental estimate to be made of the statistical data uncertainty. In regions of steep strain gradient, or near discontinuities in a specimen, interpolation from short line/area/volume scans about the point will enhance or assure the experimental assessment of uncertainties or systematic uncertainties in peak and gauge volume positions.

A.5.2 Location of measuring positions

For initial outline mapping, and when strain gradient variations are small, it is generally preferable to use a regular scanning matrix with dimensions appropriate to the strain gradients expected in each co-ordinate direction. When strain gradient variation is large along any direction, it may be necessary to increase the mapping density in that direction in order to obtain adequate spatial resolution. In these regions the mapping density should be greater but preferably should still remain regular so that any distorting effects that the mapping matrix combined with statistical uncertainties has on the interpolated strain curve are apparent.

A.6 The gauge volume

A.6.1 Gauge volume definition

The nominal gauge volume is the projected volume defined by parallel beams passing through the incident and diffracted beam masks. Because of beam divergence, the actual boundaries of the instrumental gauge volume will not be parallel and will be diffuse so that its limits are not precisely defined.

Instrumental gauge volume parameters should be obtained from horizontal and vertical intensity profiles measured across the principal gauge volume dimensions. The profiles may be obtained by measuring the diffracted neutron intensity as a fine wire is traversed across the gauge cross section as described in A.3 for positioning the gauge volume. Alternatively the profiles may be obtained from measurement of the intensity variation of neutrons scattered from a powder specimen as a fine slit is traversed across incident and diffracted beams.

NOTE 1 The instrumental gauge volume intensity profile may best be illustrated by means of three 1-dimensional intensity profiles or a 3-dimensional intensity contour map. However, for most practical purposes, the instrumental gauge volume may be described by just three linear dimensions and the diffraction angle. The dimensions quoted should correspond to the widths of the 50 % intensity contours across the incident and diffracted beams. An estimate of the sharpness of the gauge boundary should be provided. The basis of the estimate (the distance over which the intensity drops from 90 % to 10 % say) should be stated.

If masks or slits are used, the radial distance of incident (ISD) and detector (DSD) beam defining masks and slits from the reference point should be as small as is

practicable so that the effects of divergences are minimised, whilst still permitting movement of the specimen without risk of collision when scanning. Wherever possible, the ISD should be such that at least 67 % of the incident neutrons fall within the nominal gauge volume.

NOTE 2 Divergence characteristics of instruments vary but as a general rule when crystal monochromated beams are employed the 67 % criterion requires that ISD $\leqslant$ 100 × ISW or 10 × ISH whichever is the least.

A.6.2 Gauge dimensions

Gauge dimensions should be chosen so as to permit detail to be resolved as necessary. In general, if gauge dimensions exceed the size of or distance between features of significance, the strain patterns are smoothed and detail is lost.

A.6.3 Grain size considerations

Generally, strain determination for polycrystalline specimens assumes that there are a sufficient number of grains within the subset scattering into the detector to enable a satisfactory statistical average diffraction peak to be measured. If only a small number of grains are sampled, significant peak positional uncertainties 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 angular uncertainty due to coupling between position and angular sensitivity of the detector.

NOTE Oscillation 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.6.4 Attenuation considerations

Differential attenuation from different parts of the gauge volume caused by different beam path lengths through the specimen changes the effective intensity scattered from different points in the gauge volume. When the specimen shape and scattering configuration combine to cause differential attenuation across the gauge cross-section, dimensions should be restricted so that the ratio of attenuation from front to back of the gauge volume is $\leqslant e$ (2,718). If gauge volume dimensions exceed this limit, the effective displacement of the position at which the average strain in the gauge volume is measured from the sampling gauge volume centroid should be calculated and used to record the location of the strain data.

NOTE For measurements normal to a flat surface in reflection geometry, the e ratio limit corresponds to the condition that the slit widths $w \leqslant (L/4)\sin 2\theta$, where L is the attenuation length of the material, which for $2\theta = 90°$ reduces to $w \leqslant (L/4)$. For in-plane measurements in through-thickness transmission geometry there is generally no attenuation limit to gauge volume size as the path length is constant for all locations within the gauge.

A.6.5 Counting time considerations

The statistical quality of the data is a function of the number of neutrons counted which is related to the size of the sampling gauge volume and the counting time. To minimise 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.7 Near-surface precautions

A.7.1 The sampling gauge and its position

When scanning through surfaces (or interfaces) the proportion of the instrumental gauge volume occupied by the specimen material (or materials) varies, as does the shape of the occupied portion (or portions) as the surface (or interface) is traversed. Consequentially the sampling gauge volume centre is not at the instrumental gauge volume centre. Geometrical corrections for sampling gauge volume position (or positions) should be made when scanning through surfaces (or interfaces). Where appropriate, additional corrections for attenuation should be made as described in A.6.

NOTE Strain fields at surfaces and interfaces, especially when thermally or mechanically treated, can be steep. This has two consequences; positioning has to be correspondingly accurate (see A.5) and the gauge cross-section has to be correspondingly small (see A.6).

A.7.2 Instrumental surface aberrations

High resolution single-detector instruments are generally angular sensitive with very small position sensitivity. They may be employed for through-surface scanning usually with insignificant or small instrumental aberrations but they are relatively inefficient. Many instruments employ linear or multi-detectors, that have position sensitivity, in order to improve counting efficiencies. Their position sensitivity can generate instrumental aberrations that cause apparent peak shifts that can be large in comparison with strain-related peak shifts. The aberrations arise due to translation of the effective gauge cross-section horizontally across the incident and detector slits when the traversed surface is vertical.

A.7.3 Instrumental surface aberration reduction techniques

Instrumental surface aberrations should be minimised by making front detector slit distances DSD as small as is practicable and the reference point to detector distance relatively large. Aberrations also reduce with slit widths and with peak width.

When specimen surfaces are flat, the aberrations can be substantially eliminated by mounting the specimen surface horizontal and by scanning vertically. However, this may introduce additional attenuation constraints and some loss of spatial resolution.

A.7.4 Computational cancellation of instrumental surface aberration effects

In principle, surface aberration effects can be calculated and hence subtracted from experimentally affected data. However, the effects are sensitive to many parameters that are steeply non-linear very close to surfaces so that without experimental verification, computed corrections should not be relied upon.

Surface aberrations are generally antisymmetric at oppositely oriented surfaces. If two sets of measurements are made through a surface, one with the surface facing the incident beam and the other in the opposite direction, averaging the peak shifts at the same locations for the two data sets will in principle reveal the strains and eliminate the aberrations. Subtracting the two data sets gives the aberrations and eliminates the strain.

NOTE The results are subject to the combined experimental uncertainties, so care should be taken to ensure that the magnitudes of the aberrations are reduced (A.7.3), so as to minimise the statistical uncertainties which can be large. Likewise, spatial positioning should be accurate to minimise strain gradient uncertainties.

A.8 Temperature measurement and recording

Lattice dimensions are affected by temperature as well as by strain. For many engineering materials coefficients of thermal expansion are within the range $10\text{-}20 \times 10^{-6}$ K^{-1}. Specimen temperatures should be kept stable within a restricted range chosen so that lattice changes due to temperature changes are small relative to maximum statistical uncertainty specified for the strain measurement. Temperatures at which measurements are made should be recorded.

Annex B
(informative)

Determination of uncertainties in a measurand

B.1 Introduction

This annex provides a short introduction to the determination of uncertainty, and is based on the ISO 'Guide to the Expression of Uncertainty in Measurement' [16,17].

The stated value of the uncertainty of a measurand (the quantity being measured) is an estimate of our lack of knowledge of the true value of the measurand. There are many possible sources of uncertainty after any corrections for known systematic effects have been made. For example;

a) instrument resolution or discrimination threshold;

b) non-representative sampling;

c) temperature effects on the measurement or other environmental conditions,

d) bias in reading analogue instruments;

e) inexact values of constants and other parameters obtained from external sources;

f) approximations and assumptions incorporated in the measurement method and procedure;

It must be recognised that these sources are not necessarily independent, and that an unrecognised systematic effect cannot be taken into account in the evaluation of the uncertainty of the result - but contributes to its error.

In many cases the estimated variance (u^2) may be calculated from a series of repeated observations. The estimated standard deviation u is then the positive square root of u^2. Where an experimental determination is impracticable u^2 shall be evaluated using available knowledge.

The final value of the uncertainty is obtained from the values of all the uncertainties contributing to the measurement. It is termed the combined standard uncertainty and is denoted by u_c. It is given (for independent uncertainties) by:

$$u_c^2(Y) = \sum_{i=1}^{N} \left(\frac{\partial f}{\partial x_i} \right)^2 u^2(x_i) \qquad \text{(B.1)}$$

where f is the function:

$$Y = f(x_1, x_2, \ldots, x_N) \qquad \text{(B.2)}$$

in the expression for the measurand Y which depends on the number of (N) factors x_i