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 $\pm$ 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 ± 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
θ, ϕ, ψ, ω	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