TECHNICAL SPECIFICATION

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

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

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Contents

Page

Foreword

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ISO/TS 21432 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 138, *Non-destructive testing*, in collaboration with Technical Committee ISO/TC 135, *Non-destructive testing*, Subcommittee SC 5, *Radiation methods,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Introduction

Neutron diffraction is a non-destructive method that can be employed for determining residual stresses in crystalline materials. It can also be used for establishing applied stresses. The procedure can be employed for determining stresses within the interior of materials and adjacent to surfaces. It requires specimens or engineering components to be transported to a neutron source. Measurements of elastic strain are obtained which are then converted to stress. The purpose of this document is to provide the technical specification for reliably determining stresses that are relevant to engineering applications.

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

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

1 Scope

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

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

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

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

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

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 13925-3, *Non-destructive testing — X-ray diffraction from polycrystalline and amorphous materials — Part 3: Instruments*[1)]

1) To be published.

3 Terms and definitions

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

3.1
absorption
neutron capture by an atomic nucleus

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

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

3.3
anisotropy
dependence of material properties on orientation

3.4
attenuation
reduction of neutron intensity

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

3.5
background
intensity considered not belonging to the diffraction signal

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

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

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

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

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

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

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

3.11
peak position
single value describing the position of a Bragg peak

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

3.12
diffraction
scattering based on interference phenomena

3.13
diffraction elasticity constants
elasticity constants associated with individual (*hkl*) lattice planes for a polycrystalline material

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

3.14
diffraction pattern
distribution of scattered neutrons over the available range of wavelengths or times of flight and/or scattering angles

3.15
full width at half maximum
FWHM
width of the diffraction line at half the maximum height above the background

3.16
full pattern analysis
determination of crystallographic structure and/or microstructure from a measured diffraction pattern of a polycrystalline material

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

3.17
gauge volume
volume from which diffraction data are obtained

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

3.18
lattice parameters
linear and angular dimensions of the crystallographic unit cell

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

3.19
lattice spacing
d-spacing
spacing between adjacent crystallographic lattice planes

3.20
macrostress
type I stress
mean stress in a volume containing a large number of grains

NOTE Also called stress of type I.

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

NOTE There are two classes of microstress:

— the mean deviation from the macrostress determined over a grain or phase dimension (also called type II);

— the mean deviation from the type II stress determined over a volume of several atomic dimensions (also called type III).

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

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

3.24
orientation distribution function
quantitative description of the crystallographic texture

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

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

3.26
reference point
centroid of the instrumental gauge volume

NOTE See 6.5.

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

[VIM: 1993]

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

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

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

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

3.29
incoherent scattering
scattering of neutrons in an uncorrelated way

3.30
single peak analysis
statistical procedure to determine the characteristics of a peak and the background from the measured diffraction data

3.31
texture
preferred orientation of crystallites (crystallographic texture) or reinforcements (morphological texture) within a specimen

3.32
through surface scan
procedure to determine the position of a specimen surface or interface

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

3.33
time-of-flight
time needed by a neutron of a given speed (i.e. energy or wavelength) to cover the distance from a defined starting point to the detector

3.34
uncertainty of measurement
parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand

[VIM: 1993]

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

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

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

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

3.35
wall scan
see-through surface scan

4 Symbols and abbreviated terms

4.1 Symbols

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

E_{hkl}	Elasticity modulus associated with the (hkl) diffracting lattice planes	GPa
g	strain gradient	mm^{-1}
h	Planck's constant	Js
hkl	Indices of a crystallographic lattice plane	
$hkil$	Alternative indices of a crystallographic lattice plane for hexagonal structures	
H	Peak height	—
I	Integrated neutron intensity of a Bragg peak above background	
k_i, k_f	Wave vector of the incident and scattered neutrons	nm^{-1}
L	Path length from neutron source to detector	m
l	Neutron attenuation length	mm
m_n	Neutron mass (1.67×10^{-27} kg)	kg
N_n	Total number of neutrons counted	
Q	Scattering vector ($k_f - k_i$)	nm^{-1}
t	Time of flight of neutrons from source to detectors	
T	Temperature	°C or K
u	Standard uncertainty	—
x,y,z	Axes of the specimen co-ordinate system	
α	Coefficient of thermal expansion	K^{-1}
Δ	Variation of, or change in, the parameter that follows	
ε	Elastic strain	—
ε_{ij}	Components of elastic strain tensor	—
ε_{hkl}	Normal elastic strain associated with the (hkl) diffracting lattice plane	—
λ	Wavelength of neutrons	nm
ν	Poisson's ratio	
ν_{hkl}	Poisson's ratio associated with the (hkl) diffracting lattice plane	
σ	Stress	MPa
σ_{ij}	Components of stress tensor	MPa
σ_Y	Yield stress	MPa
2θ	diffraction angle	degrees
ϕ, ψ, ω	Orientation angles	degrees

4.2 Subscripts

hkl, hkil	Indicate relevance to crystallographic lattice planes
x, y, z	Indicate components along the x-, y-, z-axes of the quantity concerned
ϕ ψ	Indicate the normal component, in the (ϕ ψ) – direction of the quantity concerned
0 (zero)	Indicates strain free value of the quantity concerned
ref	Indicates reference value of the quantity concerned

4.3 Abbreviated terms

PSD Position Sensitive Detector

TOF Time of flight

IGV Instrumental gauge volume

NGV Nominal gauge volume

SGV Sampled gauge volume

5 Summary of method

5.1 Preamble

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

5.2 Outline of principle — Bragg's law

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

$$2d_{hkl}.\sin\theta_{hkl} = \lambda \quad (1)$$

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

5.3 Neutron sources

Neutron diffraction uses neutrons generated by fission or spallation; the former is predominantly employed in steady-state nuclear reactors and the latter in pulsed spallation sources. In both cases the neutrons produced are moderated to bring their energies to the thermal range, i.e. $\lambda \geqslant 0.09$ nm. At reactor sources, a monochromatic beam of neutrons is usually produced by using a crystal monochromator to select a given neutron wavelength from the polychromatic beam. At spallation sources, the neutron beam usually consists of a series of short pulses each containing a spectrum of wavelengths The energy (and therefore wavelength) of each neutron can be determined by measuring the distance it has travelled to the detector and the time it has taken to travel this distance, called the time of flight (TOF). TOF measurements are, therefore, wavelength dependent (sometimes termed energy dispersive), with the entire diffraction pattern being recorded at any particular scattering angle. Short pulses of polychromatic neutrons can also be produced by one or more choppers at continuous sources or from long pulses.

5.4 Strain measurement

When a specimen is illuminated by a monochromatic parallel beam of neutrons of known wavelength, its lattice spacing can be determined from the observed Bragg angle using Bragg's law (1). If the specimen contains no strain, the lattice spacings correspond to the strain free (stress free) values for the material and