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

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

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Foreword

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Introduction

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

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

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

1 Scope

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

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

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

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

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

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

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

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

2 Normative references

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

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

3 Terms and definitions

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

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

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

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

3.1
neutron absorption
neutron capture by an atomic nucleus

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

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

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

3.4
attenuation
reduction of the neutron beam intensity

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

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

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

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

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

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

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

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

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

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

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

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

3.13
diffraction
scattering arising from coherent interference phenomena

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

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

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

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

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

3.18
gauge volume
volume from which information is obtained

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

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

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

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

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

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

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

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

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

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

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

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

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

Note 1 to entry: See 7.5.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

4 Symbols and abbreviated terms

4.1 Symbols and units

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

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