SLOVENSKI STANDARD
SIST-TS CEN/TS 18094:2025

01-april-2025

Neporušitvene preiskave - Preskusne metode za ugotavljanje preostalih napetosti s sinhrotronskim uklonom rentgenskih žarkov

Non-destructive testing - Test method for determining residual stresses by synchrotron x-ray diffraction

Zerstörungsfreie Prüfung - Prüfverfahren zur Bestimmung von Eigenspannungen mittels Synchrotron-Röntgendiffraktometrie

Essais non-destructifs - Méthode d'essai pour l'analyse des contraintes résiduelles par diffraction des rayons X au synchrotron

Ta slovenski standard je istoveten z: **CEN/TS 18094:2024**

ICS:

19.100	Neporušitveno preskušanje	Non-destructive testing

SIST-TS CEN/TS 18094:2025 **en,fr,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 18094

December 2024

ICS 19.100

English Version

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Ref. No. CEN/TS 18094:2024 E

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European foreword

This document (CEN/TS 18094:2024) has been prepared by Technical Committee CEN/TC 138 “Non-destructive testing”, the secretariat of which is held by AFNOR.

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Introduction

This document has been developed with support from the EASI-STRESS project which has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 953219.

Much of the content of this document has been supplied by the partners from EASI-STRESS either by converting information from EASI-STRESS deliverables or through direct engagement with individuals to draft and review different parts of this document.

The engagement of experts in CEN/TC 138/WG 10 not affiliated with EASI-STRESS who also made essential contributions to this document has been equally important. A heartfelt gratitude is extended to these individuals whose exceptional dedication and voluntary contributions have significantly enriched the work.

The EASI-STRESS project (active from January 2021 until June 2024) aimed to increase industrial trust in synchrotron X-ray and neutron diffraction-based residual stress characterization techniques by validation and benchmarking, developing standard operating procedures and creating this document. The industrial aim was to revolutionize industrial residual stress management in metals, potentially enabling up to 15 % material savings from reducing over-dimensioning of components due to lack of knowledge of residual stress levels.

In order to improve industrial accessibility, the project also focused on professionalising the measurement service by harmonizing data formats and developing software tools for data analysis and establishing an industrial service function for residual stress measurement.

EASI-STRESS has also been used as practical case study on adopting advanced characterization principles according to the CHADA (CHAracterization Data).

Another source of inspiration comes from the VAMAS initiative (Versailles Project on Advanced Materials and Standards), which ultimately led to the establishment of the standard for neutron residual stress measurement, EN ISO 21432:2020.

The following EASI-STRESS deliverables are available online at the European Commission CORDIS website for EASI-STRESS[1] and have been used for the drafting of this document:

— D2.1 Benchmark samples and relevant information for their manufacture

— D2.2 Development of best practice in correlation of modelled and measured stress data. This includes details to consider during modelling and experiments and reporting formats.

— D2.3 Round-robin results from laboratory techniques and synchrotron and neutron facilities.

— D3.1 Report on technical specifications as identified in collaboration with the industrial users and at the interface with WP2, WP4 and WP5.

— D3.2 Report on SOPs (Standard Operating Procedures) for instruments dedicated to bulk analysis and to near-surface analysis.

— D4.2 Technical report with mathematical formalisms (equations), dedicated technical drawings and diagrams that describes coordinate systems, variables, workflows for data processing, and that includes the description of the experimental parameters to be included in FE-modelling software.

[1] : https://cordis.europa.eu/project/id/953219/results

1 Scope

This document describes the test method for determining residual stresses in polycrystalline materials by the synchrotron X-ray diffraction method. The method can be applied to both homogeneous and inhomogeneous materials including those containing distinct phases.

Information on how to carry out residual stress measurements by the synchrotron X-ray diffraction technique is provided as:

— 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,

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

— the selection of the stress-free reference (sample) facilitating the residual strain calculation, and

— the methods available for deriving residual stresses from the measured strain data.

Procedures are presented for calibrating synchrotron X-ray diffraction instruments, enabling:

— accurately positioning and aligning test pieces;

— precisely defining the volume of material sampled for the individual measurements;

and also for:

— making measurements;

— carrying out procedures for analysing the results;

— determining their uncertainties.

The principles of the synchrotron X-ray diffraction technique are described and put into perspective with EN 15305:2008 and EN ISO 21432:2020, which are used to measure stresses in the bulk of a specimen.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

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

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

3.1
absorption
reduction in the intensity of radiation in a medium resulting from energy conversion within the medium

Note 1 to entry: In this document, the term attenuation is also used to describe absorption when the focus is on the beam.

[SOURCE: EN 1330-11:2007, 3.1, modified - Note 1 to entry modified.]

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
angle-dispersive diffraction
beam scattering using constructive interference based on a monochromatic photon beam

Note 1 to entry: The diffraction pattern is collected by scanning the scattering angle.

3.4
anisotropy
dependence of material properties of a sample on the spatial direction

3.5
attenuation
reduction of the X-ray beam intensity

3.6
background
intensity considered not belonging to the *diffraction* (3.12) signal

Note 1 to entry: Background dependence on the scattering angle (3.34) is not uncommon and can have an influence on the *peak position* (3.30) resulting from data analysis.

[SOURCE: EN ISO 21432:2020, 3.5, modified - Note 1 to entry modified by deletion of 'or *time-of-flight* '.]

3.7
beam alignment
intensity scan procedure to determine the position of the gauge volume in the system of the instrument to set the reference point using a special sample (e.g. thin foil, plate, wire)

3.8
beam-defining optics
arrangement of devices used to define the properties of an X-ray beam such as the wavelength and intensity distributions, divergence and shape

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

3.9
Bragg peak
intensity distribution of the beam diffracted by a specific (*hkl*) lattice plane

3.10
data analysis
process to derive the desired information from the measured data

Note 1 to entry: In general, this applies to the fitting of analytical functions to the reduced data in order to derive, e.g. peak positions, FWHM, peak intensity, etc.

3.11
data reduction
conversion of detector signal to angle/energy by integration and peak fit

3.12
diffraction
scattering arising from coherent interference phenomena

3.13
diffraction elastic constants
E_{hkl}
ν_{hkl}
elastic constants associated with *diffraction* (3.12) from individual (*hkl*) lattice planes for a polycrystalline material

3.14
diffraction pattern
intensity distribution of X-rays diffracted from a crystalline material over the available wavelength, *energy-dispersive diffraction* (3.15) and/or *diffraction* (3.12) angle ranges

3.15
energy-dispersive diffraction
beam scattering using constructive interference based on a polychromatic (or white) photon beam covering a broad energy spectrum

Note 1 to entry: The diffraction pattern is collected at a fixed scattering angle using an energy-resolving detector.

3.16
entry scan
procedure to determine the position of a specimen surface or interface with respect to the *reference point* (3.33)

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

3.17
full pattern analysis
determination of the crystallographic structure and/or strain from a measured (multi-peak) *diffraction pattern* (3.14) of a crystalline 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* (3.35).

3.18
full width at half maximum
FWHM
width of the *Bragg peak* (3.9) at half the *peak height* (3.28) above the *background* (3.6)

3.19
gauge volume
intersection between the incident beam and the projection of the aperture slits, i.e. the volume from which information is obtained

3.20
incoherent scatterer
material scattering of X-rays in an uncorrelated way thus giving rise to a strong *background* (3.6) signal and no *Bragg peaks* (3.9) or only some with low amplitude

3.21
lattice parameters
linear and angular dimensions of the crystallographic unit cell

3.22
lattice spacing
***d*-spacing**
lattice plane spacing
distance between adjacent parallel crystallographic lattice planes

3.23
metadata
information necessary to interpret the set-up and conditions of the experiment related to the acquisition of the raw data in order to ensure reproducibility

3.24
monochromatic beam
X-ray beam with narrow band of energies (wavelengths)

3.25
monochromatic instrument
instrument employing a narrow band of X-ray energies (wavelengths)

3.26
orientation distribution function
quantitative description of the crystallographic *texture* (3.36)

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

3.27
peak function
analytical expression to describe the shape of the *Bragg peak* (3.9)

3.28
peak height
maximum number of signal counts of the *Bragg peak* (3.9) above the *background* (3.6)

3.29
peak intensity
integrated intensity
area under the *diffraction* (3.12) peak above the *background* (3.6), normally calculated from the associated fitted parameters of a selected *peak function* (3.27) and a background function

3.30
peak position
single value describing the angular, energy, or lattice spacing position of a *Bragg peak* (3.9) in the measured spectrum

Note 1 to entry: The peak position is normally derived from fitting an analytical function to the measured data.

Note 2 to entry: The peak position is the determining quantity to calculate the strain based on this method (see Figure 3).

3.31
polychromatic beam
X-ray beam containing a broad band of energies (wavelengths)

3.32
raw data
measurement data as recorded by the instrument together with the metadata of a measurement prior to any processing for evaluation

3.33
reference point
centroid of the instrumental *gauge volume* (3.19)

3.34
scattering angle
diffraction angle
angle at which diffraction from an incoming X-ray beam in the specimen material creates a Bragg peak

Note 1 to entry: Diffraction angle is defined as θ and scattering angle as 2θ.

3.35
single peak analysis
mathematical procedure to determine the characteristics of a peak and the *background* (3.6) from the measured *diffraction* (3.12) data

3.36
texture
crystallographic texture
preferred orientation
deviation of the crystallite orientation distribution from randomness in a polycrystalline specimen

[SOURCE: EN 1330-11:2007, 3.130]

3.37
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

3.38
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.

3.39
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

4 Symbols and abbreviated terms

4.1 Symbols and units

For the purposes of this document, the following symbols and units apply.

a,b,c	Lengths of the edges of a unit cell, here referred to as lattice parameters	nm
B	Background at the peak position	—
c_{ph}	Speed of light in vacuum	ms^{-1}
d	Lattice plane spacing	nm
E	Macroscopic elastic modulus (Young's modulus)	GPa
E_{hkl}	Elastic modulus associated with the (hkl) diffracting lattice planes	GPa
E_γ	Photon energy	J
E_{0y}	Initial photon energy	J
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	—
q	Absolute value of scattering vector	nm^{-1}
u	Standard uncertainty	—
x,y,z	Axes of the specimen coordinate system	
α	Coefficient of thermal expansion	K^{-1}
λ	Wavelength	nm
Δ	Variation of, or change in, the parameter that follows	
ε	Elastic strain	—
$\hat{\varepsilon}$	Strain tensor	—
ε_{ij}	Components of the elastic strain tensor	—
ε_{hkl}	Normal elastic strain associated with the (hkl) diffracting lattice plane	—
ν	Poisson's ratio	
ν_{hkl}	Poisson's ratio associated with the (hkl) diffracting lattice plane	
$\hat{\sigma}$	Stress tensor	MPa
σ_{ij}	Components of the stress tensor	MPa
σ_Y	Yield stress	MPa

Θ	diffraction angle	Degree
2θ	scattering angle	Degree
τ_{ϕ}	Shear stress value in a direction defined by the angle ϕ	MPa
τ	Attenuation depth	m
ϕ, ψ	Orientation angles	Degree

4.2 Subscripts

For the purposes of this document, the following subscripts apply.

hkl, *hkil*	Indicate relevance to crystallographic (*hkl*) or (*hkil*) lattice planes
x, y, z	Indicate components of the quantity concerned along the x-, y-, z-axes
$\phi\ \psi$	Indicate the normal component, in the ($\phi\ \psi$) − direction of the quantity concerned
0 (zero)	Indicates stress-free value of the quantity concerned
Ref	Indicates reference value of the quantity concerned

4.3 Abbreviations

For the purposes of this document, the following abbreviations apply.

ADXRD	Angle Dispersive X-Ray Diffraction
CSC	Conical Slit Cell
DEC	Diffraction elastic constants
EBSD	Electron backscatter diffraction
EDXRD	Energy-Dispersive X-Ray Diffraction
FEA	Finite element analysis
FWHM	Full Width at Half Maximum
GV	Gauge Volume
MCA	Multi-Channel Analyser
PSD	Position-Sensitive Detector
SGV	Sampled gauge volume
SXRD	Synchrotron X-ray Diffraction
XRD	X-ray Diffraction

5 Summary of the synchrotron XRD measurement method

5.1 General

This document deals with the determination of residual stresses that are needed in engineering analyses. The stresses are determined from X-ray synchrotron diffraction measurements of lattice spacings within engineering components. From changes in these spacings, elastic strains can be derived from which stresses can be calculated. The stress at any point in an engineering body has 9 components, 6 of which are independent (see Figure 1).