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Neporušitveno preskušanje - Preskusna metoda analize zaostalih napetosti z uklonom rentgenskih žarkov

Non-destructive Testing - Test Method for Residual Stress analysis by X-ray Diffraction

Zerstörungsfreie Prüfung - Röntgendiffraktometrisches Prüfverfahren zur Ermittlung der Eigenspannungen

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

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Foreword

This document (EN 15305:2008) has been prepared by Technical Committee CEN/TC 138 "Non-destructive testing", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2009, and conflicting national standards shall be withdrawn at the latest by February 2009.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This European Standard about "Non destructive testing - X-ray diffraction from polycrystalline and amorphous material" is composed of:

— EN 13925-1, *General principles*;

— EN 13925-2, *Procedures*;

— EN 13925-3, *Instruments*;

— EN 1330-11, *Non-destructive testing - Terminology - Terms used in X-ray diffraction from polycrystalline and amorphous materials*

In order to explain the relationship between the topics described in the different standards, a diagram illustrating typical operation involved in XRPD is given in Annex A.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

Residual strains in crystalline materials may be determined by X-ray diffraction analysis. Assuming linear elastic distortions, the related residual stresses are calculated.

In this document the principles of the measure procedure and the analysis technique are described.

1 Scope

This European Standard describes the test method for the determination of macroscopic residual or applied stresses non-destructively by X-ray diffraction analysis in the near-surface region of a polycrystalline specimen or component.

All materials with a sufficient degree of crystallinity can be analysed, but limitations may arise in the following cases (brief indications are given in Clause 12):

— Stress gradients;

— Lattice constants gradient ;

— Surface roughness;

— Non-flat surfaces (see 5.1.2);

— Highly textured materials;

— Coarse grained material (see 5.1.4);

— Multiphase materials;

— Overlapping diffraction lines;

— Broad diffraction lines.

The specific procedures developed for the determination of residual stresses in the cases listed above are not included in this document.

The method described is based on the angular dispersive technique with reflection geometry as defined by EN 13925-1.

The recommendations in this document are meant for stress analysis where only the diffraction line shift is determined.

This European Standard does not cover methods for residual stress analyses based on synchrotron X-ray radiation and it does not exhaustively consider all possible areas of application.

Radiation Protection. Exposure of any part of the human body to X-rays can be injurious to health. It is therefore essential that whenever X-ray equipment is used, adequate precautions should be taken to protect the operator and any other person in the vicinity. Recommended practice for radiation protection as well as limits for the levels of X-radiation exposure are those established by national legislation in each country. If there are no official regulations or recommendations in a country, the latest recommendations of the International Commission on Radiological Protection should be applied.

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 13925-1:2003, *Non-destructive testing – X-ray diffraction from polycrystalline and amorphous material – Part 1: General principles*

EN 13925-2:2003, *Non-destructive testing – X-ray diffraction from polycrystalline and amorphous materials – Part 2: Procedures.*

EN 13925-3:2005, *Non-destructive testing – X-ray diffraction from polycrystalline and amorphous materials – Part 3: Instruments*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms, definitions and symbols

For the purposes of this document, the following term, definition and symbols apply

3.1 Terms and definitions

3.1.1
Residual stress
self-equilibrating internal stresses existing in a free body which has no external forces or constraints acting on its boundary

3.2 Symbols and abbreviations

— 2θ The diffraction angle; this is the angle between the incident and diffracted X-ray beams.

— θ The Bragg angle; this is the angle between the diffracting lattice planes and the incident beam.

— ω The angle between the incident X-ray beam and the specimen surface at $\chi = 0$.

— ϕ The angle between a fixed direction in the plane of the specimen and the projection in that plane of the normal to the diffracting lattice planes.

— ψ The angle between the normal of the specimen and the normal of the diffracting lattice planes.

— χ The angle χ rotates in the plane perpendicular to that containing ω and 2θ; the rotation axis of χ is orientated perpendicular to both the ω and the φ axis.

— {hkl Family of crystal lattice planes defined by the indices h, k and l.

— $\varepsilon_{\phi\psi}$ Strain measured in the direction defined by the angles ϕ and ψ.

— d_0 Interplanar distance (d spacing) of a strain free specimen.

— $d_{\phi\psi}$ Interplanar distance (d spacing) of strained material in the direction of measurement defined by the angles ϕ and ψ.

— (S_1, S_2, S_3) Specimen coordinate system.

— (L1, L2, L3) Laboratory coordinate system.

- $\frac{1}{2}$ S2{hkl}, S1{hkl} Elasticity constants of the family of lattice planes {hkl.
- σii — Normal stress components (i = 1,2,3).
- τij — Shear stress components (i ≠ j ; i,j = 1,2,3).
- Z — Distance to the surface of the specimen.
- z — X-ray penetration depth.
- LP — The Lorentz Polarization factor.
- A — The Absorption factor.
- ILQ — Inter-Laboratory Qualified (used in connection with stress-reference specimen).
- LQ — Laboratory Qualified (used in connection with stress-reference specimen).
- σcert — Certified normal stress value of the ILQ stress-reference specimen.
- τcert — Certified shear stress value of the ILQ stress-reference specimen.
- σref — Normal stress value of the LQ specimen.
- τref — Shear stress real value for the LQ specimen.
- Lref — Average width of the diffraction lines for the LQ specimen.
- σdetermined — Determined Normal stress value of the stress-reference specimen.
- τdetermined — Shear stress value determined for the stress-reference specimen.
- Ldetermined — The average width of the diffraction line determined for the stress-reference specimen.
- u(□) — Standard uncertainty in the normal stress.
- u(τ) — Standard uncertainty in the shear stress.
- rσcert, rτ cert, — Repeatability of the normal stress, shear stress, and line width respectively of the certified ILQ stress- rLcert reference specimen.
- rσref, rτref , — Repeatability of the normal stress, shear stress, and line width respectively of the LQ stress-reference rLref specimen.
- Rσcert, Rτcert — Reproducibility of the normal stress and shear stress.
- λ — Wavelength of the X-rays used.
- Tr(σ) — Trace of the stress tensor: Tr(σ) = $\Sigma\sigma_{ii}$.
- I_{hkl} — Net integrated intensity of the hkl diffraction line.
- XECs — X-ray elasticity constants.
- s_r and s_R — Standard deviations of the repeatability and reproducibility.

— β Integral breadth.

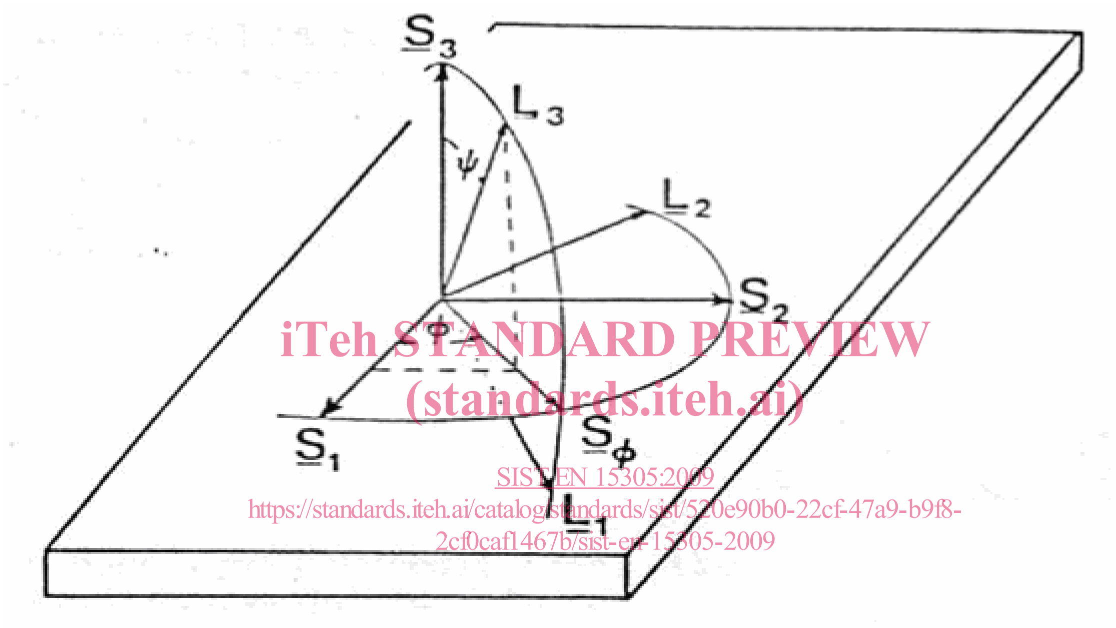
— σ_ϕ Normal stress value in a direction defined by the angle ϕ.

— τ_ϕ Shear stress value in a direction defined by the angle ϕ.

NOTE Elasticity constant is also referred to as elastic constants.

4 Principles

4.1 General principles of the measurement



Key

S_1, S_2 axes in the plane of the specimen; S_1 is defined by the operator

S_3 axis normal to the specimen surface

L_1, L_2, L_3 laboratory coordinate system; L_3 is normal to the diffracting lattice planes {hkl} and it is the bisector of the angle between incident and diffracted beams

ϕ angle between a fixed direction in the plane of the specimen and the projection in that plane of the normal to the diffracting lattice planes

ψ angle between the normal of the specimen and the normal of the diffracting lattice planes

S_ϕ direction in which the stresses σ_ϕ and τ_ϕ are measured

Figure 1 — Orthogonal coordinate systems relevant to XRD stress determination

On the basis of elasticity theory, for a macroscopically isotropic crystalline material the formula to express the strain in the direction defined by the angles ϕ and ψ (see Figure1) is:

$$\varepsilon_{\phi\psi}^{\{hkl\}} = S_1^{\{hkl\}}[\sigma_{11} + \sigma_{22} + \sigma_{33}] + \frac{1}{2}S_2^{\{hkl\}}\sigma_{33}\cos^2\psi + \frac{1}{2}S_2^{\{hkl\}}[\sigma_{11}\cos^2\phi + \sigma_{22}\sin^2\phi + \tau_{12}\sin 2\phi]\sin^2\psi + + \frac{1}{2}S_2^{\{hkl\}}[\tau_{13}\cos\phi + \tau_{23}\sin\phi]\sin 2\psi \quad (1a)$$

The stress components σ_ϕ and τ_ϕ are defined respectively as the normal stress and the shear stress in the $S\phi$ direction (see Figure 1):

$$\sigma_\phi = [\sigma_{11}\cos^2\phi + \sigma_{22}\sin^2\phi + \tau_{12}\sin 2\phi] \quad (1b)$$

$$\tau_\phi = [\tau_{13}\cos\phi + \tau_{23}\sin\phi] \quad (1c)$$

where the symbols of the formulae (1a), (1b), and (1c) are

$\varepsilon_{\phi\psi}^{\{hkl\}}$	strain in the direction defined by the angles ϕ and ψ for the family of lattice planes {hkl};
$S_1^{\{hkl\}}$ and $\frac{1}{2}S_2^{\{hkl\}}$	X-ray elasticity constants for the family of lattice planes {hkl};
σ_{11}, σ_{22}, σ_{33}	normal stress components in the directions S_1, S_2 and S_3 (cf. Figure 1);
τ_{12}	shear stress within the plane defined by S_1 and S_2;
τ_{13}	shear stress within the plane defined by S_1 and S_3;
τ_{23}	shear stress within the plane defined by S_2 and S_3;
ϕ	angle between a fixed direction in the plane of the specimen and the projection in that plane of the normal to the diffracting lattice planes;
ψ	angle between the normal of the specimen and the normal of the diffracting lattice planes;
σ_ϕ	normal stress component in a direction defined by the angle ϕ;
σ_{11}, σ_{22}	normal stress components in the directions S_1, S_2;
τ_ϕ	shear stress value in a direction defined by the angle ϕ.

The strain $\varepsilon_{\phi\psi}$ may be expressed in terms of lattice spacings according to the formula:

$$\varepsilon_{\phi\psi}^{\{hkl\}} = \ln\left(\frac{d_{\phi\psi}}{d_0}\right) = \ln\left(\frac{\sin\theta_0}{\sin\theta_{\phi\psi}}\right) \quad (2a)$$

or alternatively by the approximate formulae:

$$\varepsilon_{\phi\psi}^{\{hkl\}} \cong \left(\frac{d_{\phi\psi} - d_0}{d_0}\right) \quad (2b)$$

or

$$\varepsilon_{\phi\psi}^{\{hkl\}} \cong -\cot(\theta_0)\Delta\theta_{\phi\psi} \qquad (2c)$$

where

$d_{\phi\psi}$ spacing of the family of lattice planes {hkl} with their normal in the direction defined by ϕ and ψ;

d_0 strain-free lattice spacing of the same family of lattice planes {hkl};

θ_0 Bragg angle associated to d_0;

$\theta_{\phi\psi}$ Bragg angle associated to $d_{\phi\psi}$.

The formula (2c) is approximate and therefore it should not be used. In the calculation using (2b) the value d_0 can be estimated by interpolation on the fitted d vs. $\sin^2\psi$ curve (for details see Annex C). Using formula (2a) the d_0 and θ_0 values do not need to be accurately known.

Since the penetration depth of X-rays in most materials is in the order of tens of micrometers, σ_{33}=0 can often be assumed. Care should be exercised in the case of large penetration depths or multiphase materials (see Clause 12).

Thus, equation (1) can be simplified:

$$\varepsilon_{\phi\psi}^{\{hkl\}} = S_1^{\{hkl\}}[\sigma_{11}+\sigma_{22}] + \frac{1}{2}S_2^{\{hkl\}}[\sigma_{11}\cos^2\phi + \sigma_{22}\sin^2\phi + \tau_{12}\sin 2\phi]\sin^2\psi + \frac{1}{2}S_2^{\{hkl\}}[\tau_{13}\cos\phi + \tau_{23}\sin\phi]\sin 2\psi \qquad (3)$$

where the symbols are as for formulae (1a), (1b), (1c).

For the usual methods (ω and χ method, see Clause 6.2) the rotation angle ϕ is equal to the rotation applied to the specimen around the surface normal. Other methods exist in which the relations between the angles ϕ, ψ and the specimen rotations are more complex (see Annex F).

Note that the elasticity constants of the {hkl} lattice planes may be significantly different from those of the macroscopic bulk values (see Clause 10).

4.2 Biaxial stress analysis

From X-ray diffraction experiments on polycrystalline materials $\varepsilon_{\psi\phi}$ values at different ψ and ϕ angles are obtained. If the stress state is biaxial ($\tau_{13} = \tau_{23} = \sigma_{33} = 0$), then it follows from equation (3) that the dependence of $\varepsilon_{\phi\psi}$ on $\sin^2\psi$ is linear:

$$\varepsilon_{\phi\psi}^{\{hkl\}} = \frac{1}{2}\,S_2^{\{hkl\}}.\sigma_\phi \sin^2\psi + S_1^{\{hkl\}}.Tr(\sigma) \qquad (4a)$$

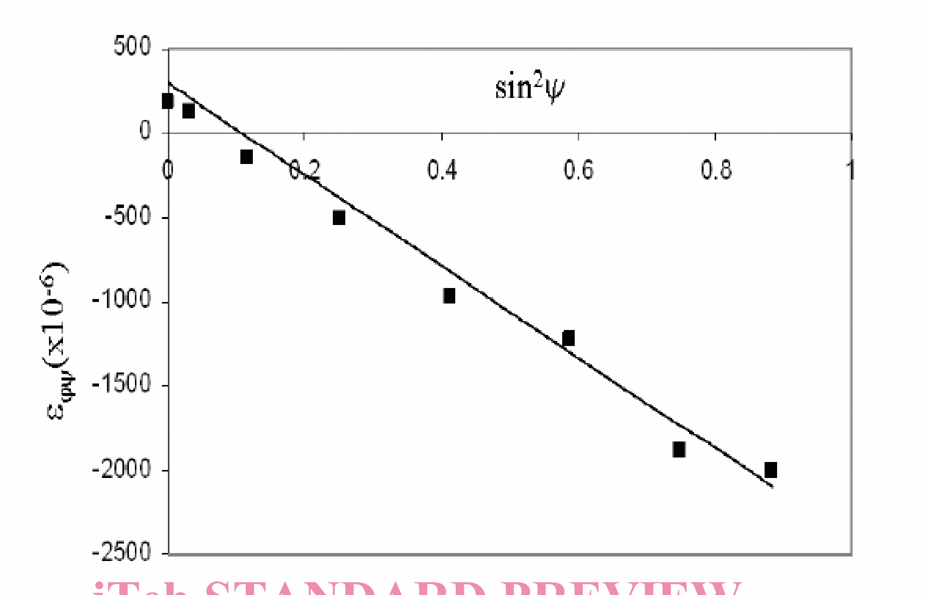
where:

$Tr(\sigma) = (\sigma_{11}+\sigma_{22})$.

For formula (4a) the same symbols hold as for formula (3).

If the stress state is biaxial then experimentally a straight line should be obtained (see Figure 2).
The stress in the ϕ -direction, σ_ϕ, is calculated from the slope of the straight line:

$$\sigma_{\phi} = \frac{\left(\dfrac{\partial \varepsilon_{\phi\psi}^{\{hkl\}}}{\partial \sin^2 \psi}\right)}{\frac{1}{2} S_2^{\{hkl\}}} \qquad (4b)$$



Key

$\varepsilon_{\phi\psi}$ strain measured in the direction defined by the angles ϕ and ψ

ψ angle between the normal of the specimen surface and the normal of the diffracting lattice planes

Figure 2 — Example of $\varepsilon_{\psi\phi}$ versus $\sin^2\psi$ plot at constant ϕ in case of biaxial stress.

In Figure 2 the material undergoes a stress state with σ_ϕ = -400 MPa, τ_ϕ = 0. The X-ray elasticity constant of the material is:

$$\frac{1}{2} S_2\{hkl\} = 6.8 \ 10^{-6} \ \text{MPa}^{-1} \qquad (4c)$$

The σ_ϕ values for negative and positive ψ values are coinciding and denoted by squares. The line corresponds to the least square fitting by equation (4a).

Due to the insufficient accuracy of d_0, the stresses obtained from $T_\rho(\sigma)$ should not be used for further calculations.

4.3 Triaxial stress analysis

If shear stresses acting in the planes perpendicular to the specimen surface are present ($\tau_{13} \neq 0$ and/or $\tau_{23} \neq 0$) then the plot of $\varepsilon_{\phi\psi}$ vs. $\sin^2\psi$ is an ellipse, showing the "ψ-splitting" for ψ>0 and ψ<0 (see Figure 3). If σ_{33} is not equal to zero then the slope of $\sin^2\psi$ plot is proportional to $\sigma_\phi - \sigma_{33}$. In these cases, equation (4a) becomes:

$$\varepsilon_{\phi\psi} = \frac{1}{2} S_2^{\{hkl\}} (\sigma_\phi - \sigma_{33}) \sin^2 \psi + \frac{1}{2} S_2^{\{hkl\}} . \tau_\phi \sin 2\psi + \frac{1}{2} \cdot S_2^{\{hkl\}} . \sigma_{33} + S_1^{\{hkl\}} . \mathrm{Tr}(\sigma) \qquad (5)$$

where

$\mathrm{Tr}(\sigma) = (\sigma_{11} + \sigma_{22} + \sigma_{33})$