

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

ICS 19.100

National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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

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

Zerstörungsfreie Prüfung - Röntgendiffraktometrisches
Prüfverfahren zur Ermittlung der Eigenspannungen

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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of an identical text or by endorsement, at the latest by February 2009, and conflicting national standards shall be withdrawn at the latest by February 2009.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.

The topic "Non destructive testing – X-ray diffraction from polycrystalline and amorphous material" is considered in the present document and several other European Standards, namely:

- 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.

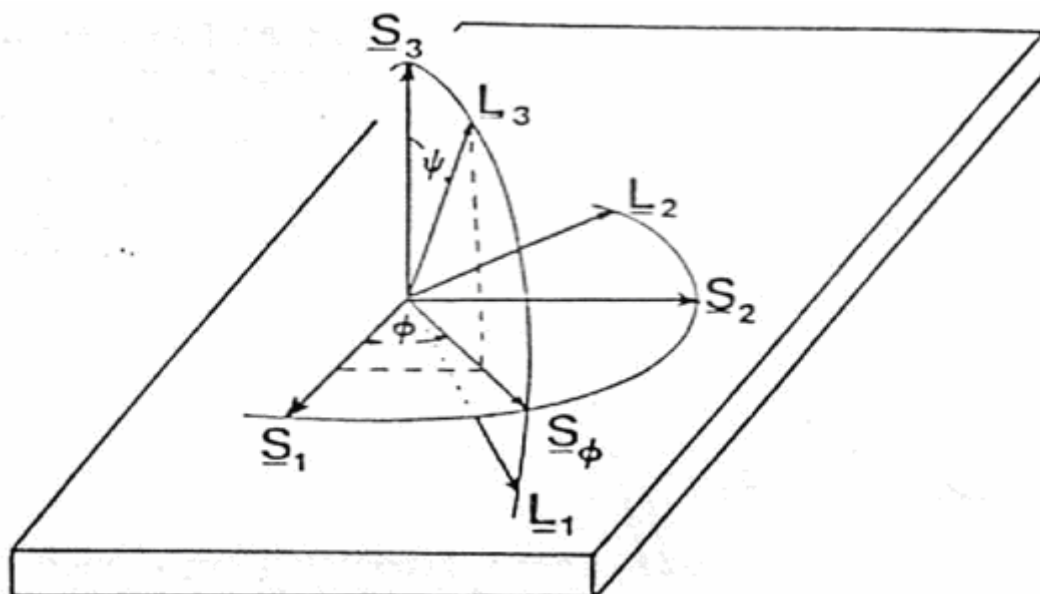
— $\frac{1}{2} S2\{hkl\}, S1\{hkl\}$	Elasticity constants of the family of lattice planes $\{hkl\}$.
— σ_{ii}	Normal stress components ($i = 1,2,3$).
— τ_{ij}	Shear stress components ($i \neq j ; i,j = 1,2,3$).
— Z	Distance to the surface of the specimen.
— z	X-ray penetration depth.
— LP	The Lorentz 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).
— σ_{cert}	Certified normal stress value of the ILQ stress-reference specimen.
— τ_{cert}	Certified shear stress value of the ILQ stress-reference specimen.
— σ_{ref}	Normal stress value of the LQ specimen.
— τ_{ref}	Shear stress real value for the LQ specimen.
— L_{ref}	Average width of the diffraction lines for the LQ specimen.
— $\sigma_{determined}$	Determined Normal stress value of the stress-reference specimen.
— $\tau_{determined}$	Shear stress value determined for the stress-reference specimen.
— $L_{determined}$	The average width of the diffraction line determined for the stress-reference specimen.
— $u(\sigma)$	Standard uncertainty in the normal stress.
— $u(\tau)$	Standard uncertainty in the shear stress.
— $r\sigma_{cert}, r\tau_{cert}, rL_{cert}$	Repeatability of the normal stress, shear stress, and line width respectively of the certified ILQ stress-reference specimen.
— $r\sigma_{ref}, r\tau_{ref}, rL_{ref}$	Repeatability of the normal stress, shear stress, and line width respectively of the LQ stress-reference specimen.
— $R\sigma_{cert}, R\tau_{cert}$	Reproducibility of the normal stress and shear stress.
— λ	Wavelength of the X-rays used.
— $Tr(\sigma)$	Trace of the stress tensor: $Tr(\sigma) = \sum \sigma_{ii}$.
— 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:

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

The stress components σ_ϕ and τ_ϕ are defined respectively as the normal stress and the shear stress in the S_ϕ 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 $\{hkl\}$;
$\sigma_{11}, \sigma_{22}, \sigma_{33}$	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

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

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, $\sigma_{33}=0$ 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:

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

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:

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

where:

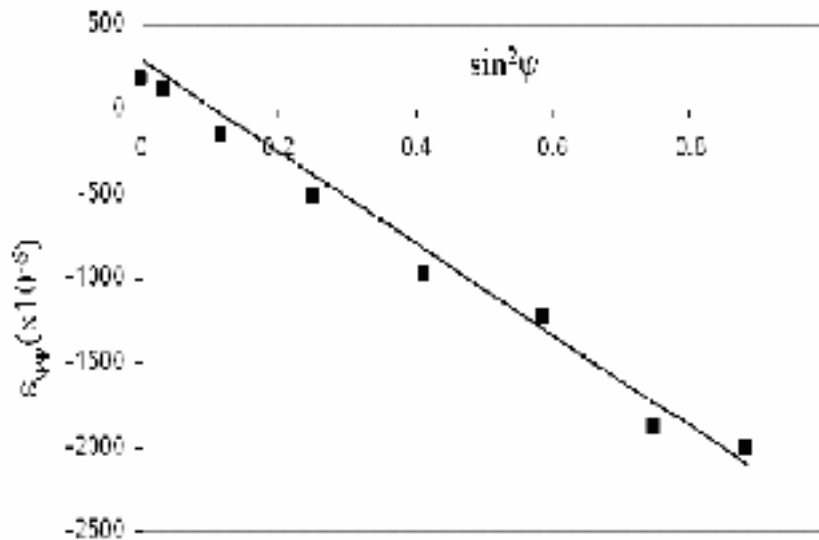
$$\text{Tr}(\sigma) = (\sigma_{11} + \sigma_{22}).$$

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:

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



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 $\sigma_{\phi} = -400$ MPa, $\tau_{\phi} = 0$. The X-ray elasticity constant of the material is:

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

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 $T_p(\sigma)$ should not be used for further calculations.

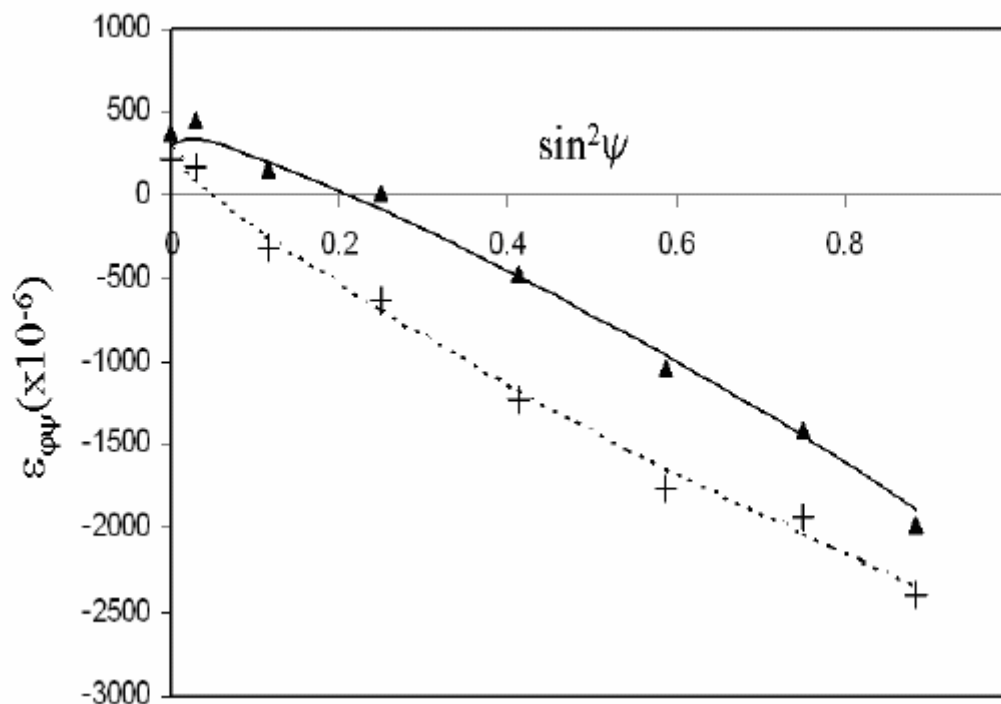
4.3 Triaxial stress analysis

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 $\psi > 0$ and $\psi < 0$ (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:

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

where

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



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
- +
 positive ψ values
- ▲
 negative ψ values

Figure 3 — Example of $\varepsilon_{\phi\psi}$ versus $\sin^2\psi$ plot at constant ϕ in case of triaxial stress (ψ splitting).

In Figure 3 the material undergoes a stress state with $\sigma_\phi = -400$ MPa, $\tau_\phi = -50$ MPa. The X-ray elasticity constant of the material is $\frac{1}{2} S_2\{hkl\} = 6.8 \times 10^{-6} \text{ MPa}^{-1}$. The lines correspond to the least square fitting by equation (5).

At a fixed ϕ angle, the σ_ϕ and τ_ϕ values are obtained by the least squares fitting of the strain data with equation (5). By measuring at least three different ϕ directions and at least three ψ angles the stress tensor can be derived (see Clause 7.4).

5 Specimen

5.1 Material characteristics

5.1.1 General

To measure and calculate the residual stress the following parameters are required:

- crystallographic data of the material;
- X-ray elasticity constants of the material.

When the values of the X-ray elasticity constants have not been determined experimentally (see Clause 10), it is recommended to use X-ray elasticity constants calculated by models taking into account single crystal elastic anisotropy and the coupling conditions between the crystallites (see Clause 8.3.1.2). Incorrect values of the X-ray elasticity constants ($\frac{1}{2} S_2^{\{hkl\}}$ and $S_1^{\{hkl\}}$) may cause significant systematic errors in the stresses calculated.

Prior knowledge of the specimen history and its microstructure can indicate if problems might occur as listed in Clause 1 (see Clause 12).

5.1.2 Shape, dimensions and weight

For any specimen, a suitable flat region should be chosen for residual stress measurement. The shape and size of the specimen is not critical, but it shall fit onto the specimen stage when required.

The specimen is subjected to various tilts during the measurement and therefore needs to be firmly attached to the specimen stage. Care has to be taken on maximum allowable weight for the goniometer and on how the specimen is to be mounted and held onto the goniometer. The specimen can be clamped to the stage only if this does not lead to additional stresses being induced into the specimen.

The necessary flatness of the specimen depends on the irradiated area. It is recommended that the local radius of curvature of the specimen shall be large enough (see Clause 12) to allow for an irradiation as longer as possible.

5.1.3 Specimen composition/homogeneity

Care shall be taken to choose an irradiated volume with homogeneous composition when possible. Since the penetration depth of the X-rays as well as irradiated area depend on ψ tilt, consideration shall be given to compositional changes that may be present within the surface and the depth (see Clause 6.3).

In multiphase materials the residual stress, i.e. the *overall* residual stress, is determined from the contributions of the stresses present in all specific phases using:

$$\sigma^{overall} = \sum_{phases} x_i \sigma_i \quad (6)$$

where

$\sigma^{overall}$ is the residual stress of the specimen;

x_i is the volume fraction of the i phase;

σ_i is the stress in the i phase obtained from the $\{hkl\}$ lattice planes of that phase.

It is therefore mandatory that the phases are known from which the diffraction lines originate.

5.1.4 Grain size and diffracting domains

The grain size in the irradiated volume can also affect the residual stress value. In many crystalline materials grain sizes are in the range 10-100 μ m. As grains often consist of many diffracting domains, such grain size values are usually acceptable for X-ray stress measurements. For larger grain sizes, it is likely that only a few diffracting domains contribute to the diffraction line. This can lead to large variations in the peak shape and intensity with ϕ , ψ directions. In addition, the presence of micro/intergranular strains may also affect the results. In some cases it is possible to reduce this effect by oscillating the specimen (for details see Clause 7), because this increases the number of diffracting grains.

5.1.5 Specimen X-ray transparency

In some materials the penetration depth of X-rays can be large enough to lead to errors in stress measurements due to the offset of the irradiated volume with respect to the surface (see Annex E). In addition, the effect of stress gradients and significant stress σ_{33} will be more pronounced.

5.1.6 Coatings and thin layers

Residual stresses in coatings can be determined provided that the diffraction lines associated with the coating itself can be identified and isolated from the diffraction lines associated with the substrate.

Measurement in thin layers may lead to the following problems:

- low diffracted intensities and/or insufficient grain statistics;
- additional diffraction phenomena from multilayers;
- overlap with a diffraction line from the substrate;
- steep stress gradient;
- strong texture.

(See also Clause 12.)

Finally, the values of the elasticity constants for the coating may be significantly different from the 'bulk' values.

5.2 Preparation of specimen

5.2.1 Surface preparation

Surface preparation should be avoided. However, if the surface is oxidized, painted or varnished, it can be cleaned by electro polishing or by using chemicals to preserve the stress field as much as possible. Be careful with chemicals which may weaken the grain boundaries or which may preferentially etch one of the phases present as it can lead to local stress relaxation.

5.2.2 Stress depth profiling

5.2.2.1 General

The stress can be determined as a function of depth by successive cycles of electro polishing and stress analysis. In some cases also a variation of the penetration depth of the X-ray, e.g. by using different wavelengths or by tilting of the specimen, can furnish depth profile stress data.

5.2.2.2 Removal of surface layers

Any mechanical or electro discharge machining (EDM) method to remove surface layers induces residual stresses, altering the stress field of the surface. Thus, such methods shall be avoided. Chemical attack or electro polishing is suggested to remove layers without introducing new stresses on the surface. Both chemical attack and electro polishing may cause relaxation of the residual stresses, e.g. due to the removal of stress in the surface layer, changes of surface roughness, or grain boundary attack. When necessary, thick layers can be removed using a combined machining or grinding procedure, followed by electro polishing to remove the layer strained by the machining or grinding. If the volume of the removed material is large compared to the total volume of the specimen, stress redistribution effects shall be considered in the calculation of the original stress field. Simple cases are treated in [2].

5.2.2.3 Evaluation of the thickness reached after removal of the material

The thickness of the layer removed shall be determined. It is suggested that the shape and the roughness of the surface are verified because problems may arise from non-flat and/or rough surface (see Clause 12).

5.2.3 Large specimen or complex geometry

Generally it is recommended to avoid the sectioning of samples. If sectioning of the sample is necessary then this should be carried out with care to avoid changes in the existing residual stresses. The measured region shall, if possible, be far enough from the edge of the specimen to avoid any effect of relaxation of residual stress perpendicular to the edge. It is recommended that the distance to the edge is at least equal to the thickness of the specimen.

In addition, one should avoid overall relaxations which may occur, e.g., when cutting pipes in the axial direction. If relaxations are expected, methods such as strain gauging [3] should be used to monitor any changes during or after cutting.

6 Equipment

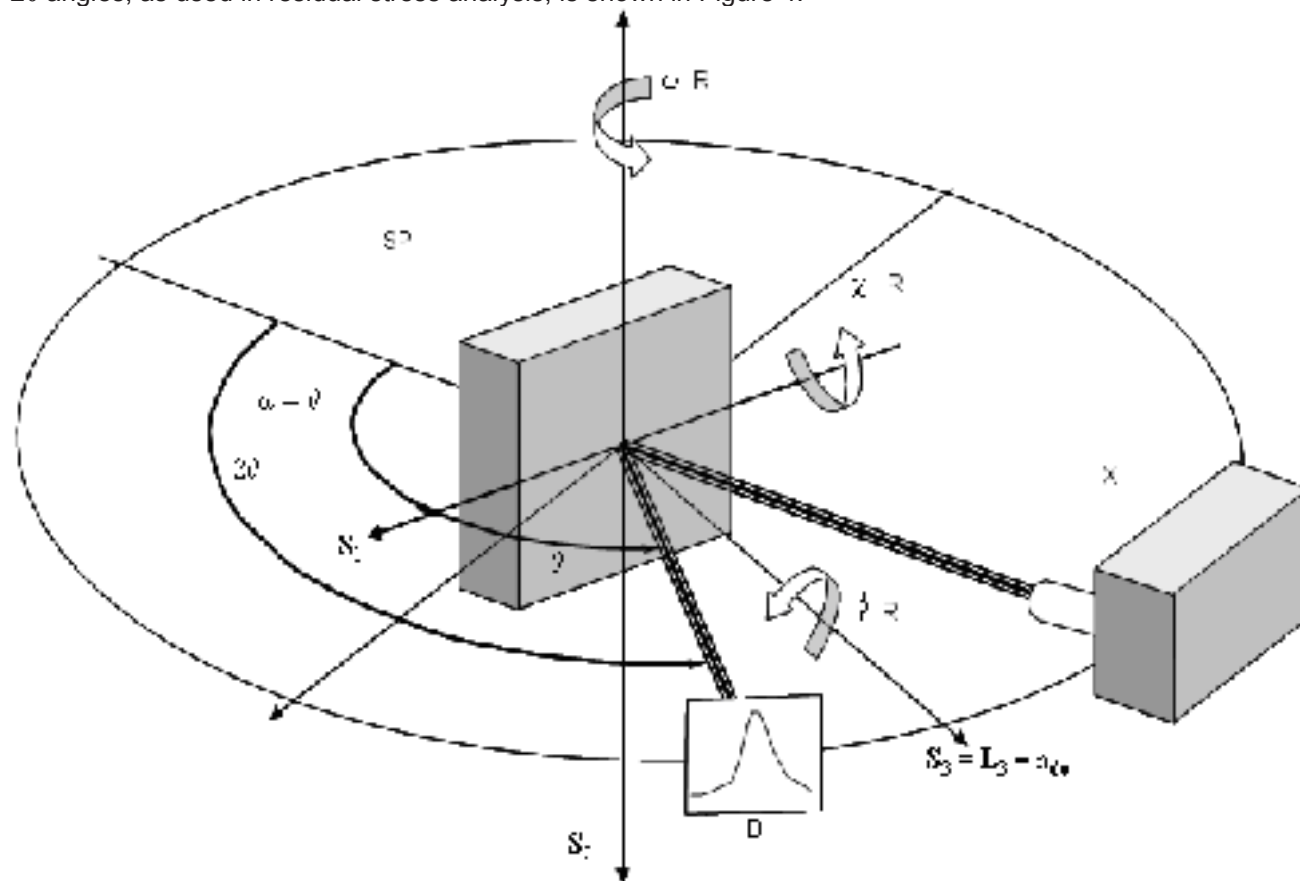
6.1 General

A general description of the equipment used for residual stress analysis can be found in EN 13925-3. In this chapter some specific aspects of the equipment that relate to the analysis of residual stresses are discussed.

6.2 Choice of equipment

6.2.1 General

In the general case two rotations are required (see Figure 1); ψ rotation is strictly necessary while ϕ -rotation can be useful in some experiments. The general arrangement of a laboratory goniometer measuring at high 2θ angles, as used in residual stress analysis, is shown in Figure 4.



Key

S_1, S_2, S_3	specimen coordinate system
θ	Bragg angle, this is the angle between the diffracting lattice planes and the incident beam
2θ	diffraction angle, this is the angle between the incident and diffracted X-ray beams
ωR	rotation about the ω axis
ϕR	rotation about the ϕ axis
χR	rotation about the χ axis
SP	specimen
D	detector
X	X-ray tube

Figure 4 — Goniometer at $\Psi = 0$ for χ and ω method ($\omega = \theta$, $\chi = 0$)

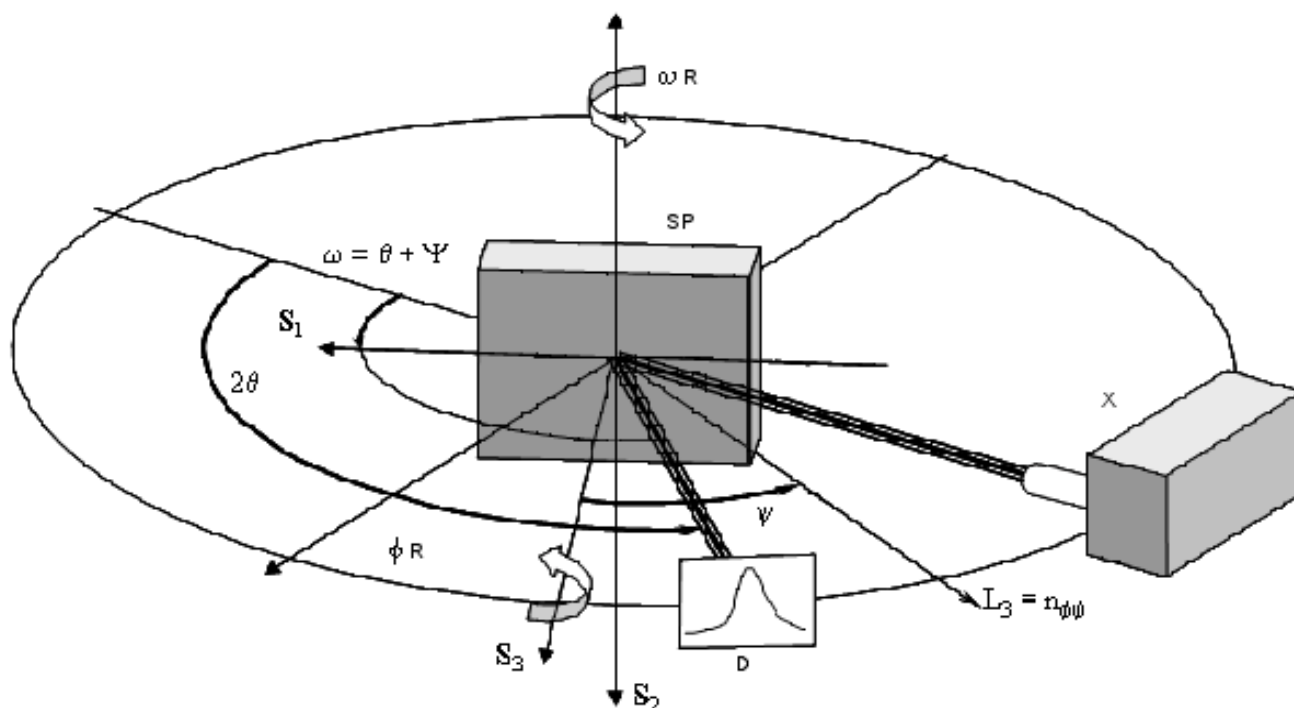
The ψ tilt can be performed in various geometries. Two of them, called ω and χ method, are shown in Figures 5 and 6.

Laboratory goniometers achieve a good precision in the measurement, permitting generally an extensive choice of 2θ angles; they are often used in other applications of X-ray diffraction (texture, profile analysis, phase analysis, (see EN 13925-1)). However, generally only non-cumbersome specimens can be analysed.

6.2.2 The ω -method

In this method (also called iso-inclination method) the specimen is rotated (tilted) about the ω axis. Both ω and 2θ are in the same plane. To obtain ω values, ψ values are algebraically added to θ . Absolute values of ψ are added to θ for positive ψ or subtracted for negative ψ . Most conventional powder diffractometers, with a decoupled ω drive (where ω and 2θ axes are able to move independently) can make measurements using this method. The geometry is shown schematically in Figure 5.

Note that the σ_ϕ direction lies in the plane of diffraction (see also Annex E).



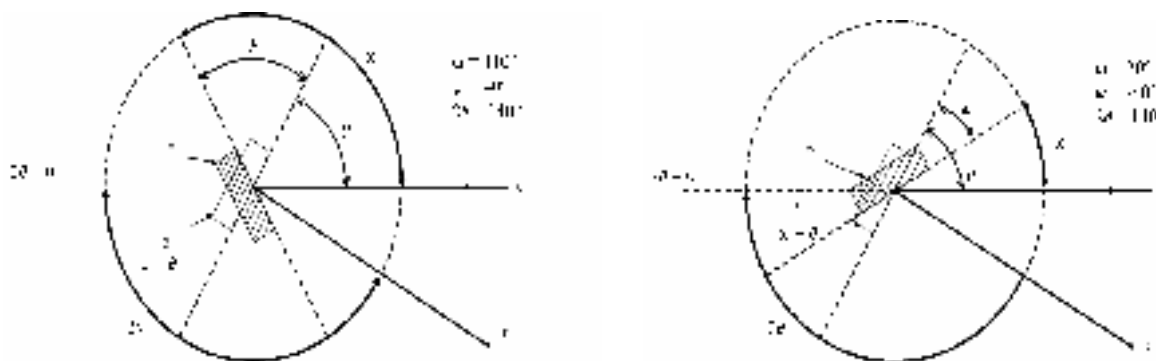
Key

S_1, S_2, S_3	specimen coordinate system
θ	Bragg angle; this is the angle between the diffracting lattice planes and the incident beam
2θ	diffraction angle, this is the angle between the incident and diffracted X-ray beams
ωR	rotation about the ω axis
ϕR	rotation about the ϕ axis
X	X-ray tube
SP	Specimen
D	detector

Figure 5 — The ω -method: ψ angle is achieved through ω rotation where $\phi = 0$ and $\Psi = \omega - \theta = -45^\circ$ (χ remains equal to zero)

Positive and Negative Offsets

Figure 6a shows a specimen with a positive ψ -offset for the ω -method, where ψ has been added to θ . Figure 6b shows a negative ψ -offset where ψ has been subtracted from θ .



Key

- 2θ diffraction angle; this is the angle between the incident and diffracted X-ray beams
- θ Bragg angle; this is the angle between the diffracting lattice planes and the incident beam
- ω angle between the incident X-ray beam and the specimen surface at $\chi = 0$
- 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
- A diffracting lattice planes
- B sample at
- C incident x-ray beam
- D diffracted x-ray beam

Figure 6a — Positive ψ -offset ($\omega = \theta + \psi$, with $\psi > 0$) Figure 6b — Negative ψ -offset ($\omega = \theta + \psi$, with $\psi < 0$)

When the ω -method is used with the negative ψ -offset, the decreased incidence angle increases the “defocusing” effects which increase the broadening of the diffraction lines and decrease the intensities more than an equal but positive ψ at the same 2θ . However, both positive and negative ψ -offsets shall be used when shear stresses shall be evaluated. In this case measurements at negative ψ tilts can be avoided by rotating the specimen (around the ϕ axis) by 180° and then measure the stress using positive ψ tilts. This avoids problems caused by defocusing and/or the higher sensitivity to alignment errors with negative ψ tilts. From the point of view of stress analysis, it is equivalent to negative ψ measurements without a ϕ rotation (often referred to as pseudo-negative tilting).

For focusing optics, measurements applying negative ψ are more susceptible to misalignments than measurements applying positive ψ .

6.2.3 The χ -method

Note that the σ_ϕ direction is perpendicular to the plane of diffraction (see also Annex E).

Mechanically the χ -method is more complex and, for some diffractometers, it requires the incorporation of additional equipment (such as an Eulerian cradle). Negative ψ can also be reached by rotation about ϕ axis of 180° and applying positive ψ .

The advantage in using the χ -method is that defocusing effects are the same for positive and negative ψ . Generally the χ -method leads to smaller errors related to the specimen centring than for the ω -method.

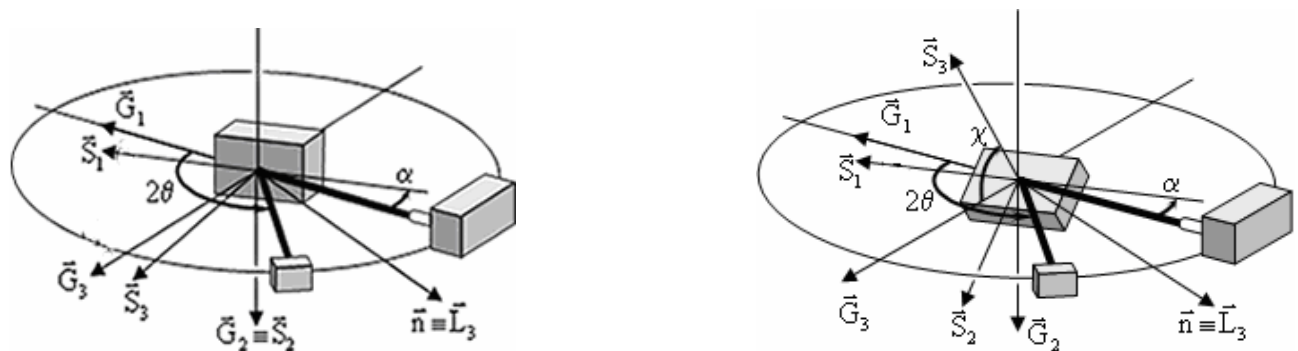
This method is sometimes confusingly referred to as the ψ method.

6.2.4 The modified χ -method

The modified χ -method is mainly applied in portable diffractometers in conjunction with two position sensitive detectors. The specimen is rotated about the χ axis, which is in a plane normal to that containing ω and 2θ . At $\chi = 0$ the incident beam is normal to the surface of the specimen which corresponds to $\omega = 90^\circ$. Two detectors are used and these are placed symmetrically with regard to the incident beam.

In the case of the one-detector system using the χ geometry for the $\chi = 0$ position the normal on the diffracting lattice planes and on the sample are parallel, whereas in the case of the two-detector system the incident beam is parallel to the normal on the diffracting lattice planes.

The ψ angle is achieved through χ rotation but ω remains equal to 90° instead of θ , i.e. at $\chi = 0$, the incident beam is normal to the surface of the specimen. Thus, $\cos \psi = \cos \chi \sin \theta$. Starting position (left) and position for $\chi = 50^\circ$ (right), the measurement is performed for $\phi = 90^\circ$ (the strains obtained from the two detectors shall be averaged and the appropriate corrections shall be performed).



Key

S_1, S_2, S_3	specimen coordinate system
L_3	normal to the diffracting $\{hkl\}$ lattice planes
2θ	diffraction angle; this is the angle between the incident and diffracted X-ray beams
ψ	angle between the normal of the specimen and the normal of the diffracting lattice planes
χ	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 axes

Figure 7 — The modified χ -method

6.2.5 Other geometries

The geometries presented in Figures 5 and 6 are the more common geometries. Other geometries are possible, but not considered in detail in this document. A general approach describing some geometries is given in Annex F.

6.3 Choice of radiation

The choice of X-ray tube anode and therefore the wavelength of the incident X-ray beam is critical for the determination of residual stress.

If the $K\alpha_1$ radiation in the incident beam causes fluorescence from the specimen, the wavelength is not suitable, because the fluorescence produces a very high background and consequently poor peak-to-background ratio. This can be dramatically improved by using e.g. a diffracted beam monochromator, which removes the fluorescent radiation before the detector, or by using an electronic energy discriminating detector.

Moreover, when the incident X-ray beam is absorbed by fluorescence effects, the penetration depth into the specimen can be very small and insufficient for a stress value representative of the specimen.

Usually, in this case, a longer wavelength can be chosen, which does not have sufficient energy to cause fluorescence.

The average information depth is defined as:

$$z = \frac{\sin^2 \theta - \sin^2(\omega - \theta)}{2\mu \sin \theta \cos(\omega - \theta)} \quad \text{for } \omega\text{-method} \quad (7a)$$

$$z = \frac{\sin \theta \cos \chi}{2\mu} \quad \text{for } \chi\text{-method} \quad (7b)$$

$$z = \frac{\cos \chi (1 - \cot^2 \theta)}{2\mu} \quad \text{for modified } \chi\text{-method} \quad (7c)$$

where for formulae (7a), (7b), (7c)

z is the X-ray penetration depth;

θ is the Bragg angle;

ω is the angle between the incident X-ray beam and the specimen surface at $\chi = 0$;

χ is the angle χ rotates in the plane perpendicular to that containing ω and 2θ ;

μ is the linear attenuation coefficient.

For other geometries, adequate formulae shall be used (see Annex F).

Examples of diffraction conditions (e.g. radiation to be used, filter, $\{hkl\}$ lattice planes, Bragg angle) for common materials are reported in Table 1.

Table 1 — Diffraction conditions for common materials

Alloy	Crystal structure	Anode	K _β filter	{hkl}	2θ(°) ^a	Multiplicity of the lattice planes	Average information depth of X-rays in μm at: ψ = 0 ^{a b}
Nickel alloys	cubic	Mn	Cr	{311}	152 to 162	24	4.9
Ferritic steels	cubic	Cr	V	{211}	156	24	5.8
Cast iron (matrix)							
Austenitic steels	cubic	Mn	Cr	{311}	152	24	7.2
Aluminium alloys	cubic	Cr	V	{311}	140	24	11.5
Aluminium alloys	cubic	Cu	Ni	{422}	137	24	35.5
Cobalt alloys	cubic	Mn	Cr	{311}	153 to 159	24	5.6
Copper alloys	cubic	Mn	Cr	{311}	149	24	4.2
Titanium alloys	hexagonal	Cu	Ni	{213}	142	24	5.0
Molybdenum alloys	cubic	Fe	Mn	{310}	153	24	1.6
Zirconium alloys	hexagonal	Fe	Mn	{213}	147	24	2.8
Tungsten alloys	cubic	Co	Fe	{222}	156	8	1.0
Alumina - alpha	rhomboedric	Cu	Ni	{146}	136	12	37.4
				{4.0.10}	145	6	38.5
		Fe	Mn	{2.1.10}	152	12	20.0
Alumina - gamma	cubic	Cu	Ni	{844}	146	24	38.5
		V	Ti	{440}	128	12	8.8

^a Indicative values.

^b Average information depth is defined as the depth from which 67% of the diffracted intensity has been absorbed. It is the depth at which the stress is evaluated if the stress gradient along the depth is linear.

The X-radiation should be selected to give a reflection at a Bragg angle greater than 130° 2θ. However, though not ideal, it is possible to use reflections which are as low as 120° 2θ. Using reflections with 2θ angles of less than 120° is not recommended because of the low sensitivity of strain measurement and in the case of focusing optics also the high sensitivity to misalignment.

The diffraction line should not be too close to the high 2θ limit of the instrument: if possible, the whole diffraction line down to the background at both sides of the peak shall be recorded.

Depending on the set up of the diffractometer and the treatment of data, large errors can arise if these conditions are not fulfilled [4].

Residual stresses calculated by using different wavelengths may have different values because of the different penetration depth of the X-ray beam into the specimen. This is more an issue where steep stress gradients are present (see Clause 12).

6.4 Choice of the detector

Detectors are different in type, size and shape (see 4.3 of EN 13925-3:2005)

The choice of spot, linear or area detector is important to reduce the measurement time and it may influence some aspects of the setting geometry.

Spatial resolution of a linear or area detector is generally not an issue in stress analysis.

A good energy resolution helps to reduce the background and thus to obtain good line position repeatability.

Saturation of the detector shall be avoided because it distorts the shape of the diffraction lines.

6.5 Performance of the equipment

6.5.1 Alignment

Primarily, the alignment shall be in accordance with the requirements of EN 13925-3. (Clause 7.1)

Additional requirements are:

- The surface of the specimen shall coincide with the axis of rotation of the specimen which depends on whether the ω or χ method is used. For the ω -method the surface of the specimen shall coincide with the ω -axis of the goniometer. For the χ -method it shall coincide with the χ -axis of the cradle.
- If several stress components are measured, the ϕ axis shall be coincident with the centre of the irradiated area.

The incident X-ray beam shall intersect the axes of rotations ϕ and ψ . Displacement of the beam will introduce positional errors during rotation and/or translation of the specimen. For the χ -method the displacement in the axial direction is most important to minimize and for the ω -method it is the displacement in the equatorial plane.

The alignment of the beam can be checked for example using a fluorescent screen, narrow aperture (glass slit) or small reference specimen. The alignment shall be verified before performing the measurement. If the perfect alignment of specimen height and beam is not achieved, the diffraction line positions can be corrected afterwards by using data coming from either a reference specimen (see Clause 6.6 and E.3.1) or a thin layer of fine well crystallised powder deposited on the surface of the specimen itself. A shift of the ψ origin cannot be corrected by this procedure.

The weight of the specimen can affect the alignment of the system. Therefore the alignment shall be checked by using a similar loading condition of the specimen stage.

6.5.2 Performance of the goniometer

Additionally to the demands presented in this document, the performance of the goniometer shall follow the requirements of EN 13925-3. (Clause 5).

6.6 Qualification and verification of the equipment

6.6.1 General

The qualification and verification of the equipment is performed in accordance with EN 13925-3 (Clause 8):

- It is necessary to qualify new equipment or existing equipment after mechanical changes have been made or if any changes to the electronics have occurred. Qualification is performed by measuring stress-free and ILQ stress-reference specimens (see Clause 11.3).
- It is necessary to periodically verify the performance of the equipment. This verification is performed with the stress-free and ILQ or LQ stress-reference specimens (see Clause 11.3).

An ILQ stress-reference specimen may be obtained through round robin tests of at least five laboratories in accordance with ISO 5725-2. If certified specimens are available they should be used as ILQ stress-reference specimen [5].

6.6.2 Qualification

6.6.2.1 General

The qualification shall be performed using both a stress-free specimen and an ILQ stress-reference specimen.

6.6.2.2 Criteria of qualification of a stress free specimen

The measurement of a stress free specimen (see 11.2) allows the evaluation of the errors related to the displacement of the beam and of the specimen.

Powder materials are expected to have zero stress: i.e. 2θ constant for all ψ and ϕ angles. If stress values significantly different from zero are obtained, the system shall be checked, adjusted and then requalified.

The equipment is certified if the stress free specimen gives the following result:

$$|\sigma| \leq \frac{1}{10000} \cdot \frac{1}{1/2S_2^{\{hkl\}}} \quad \text{with} \quad |u(\sigma)| \leq \frac{1}{10000} \cdot \frac{1}{1/2S_2^{\{hkl\}}} \quad (8a)$$

$$|\tau| \leq \frac{1}{20000} \cdot \frac{1}{1/2S_2^{\{hkl\}}} \quad \text{with} \quad |u(\tau)| \leq \frac{1}{20000} \cdot \frac{1}{1/2S_2^{\{hkl\}}} \quad (8b)$$

where

$\frac{1}{2} S_2^{\{hkl\}}$ is the elasticity constants of the family of lattice planes $\{hkl\}$;

$u(\sigma)$ and $u(\tau)$ are the uncertainties as calculated in 8.3.2.

The value of $1/2S_2^{\{hkl\}}$ used should be that of the analysed material, not that of the powder of the stress-free specimen.

6.6.2.3 Qualification of an instrument with an ILQ stress-reference specimen

When available, the qualification shall be performed on an ILQ stress-reference specimen (see Clause 11.3). If the obtained values are significantly different from the reference value, the equipment shall be checked, adjusted and then requalified.

The qualification with an ILQ stress-reference specimen shall be performed in the following steps:

- Choose the number n ($n > 4$) of measurement to be done on the specimen in repeatable conditions (see Clause 11.3.1.3) for the qualification (or the verification). The number n shall be reported.
- Calculation of the critical difference, CD, for the normal stress and for the shear stress:

$$CD_{\sigma} = \frac{1}{\sqrt{2}} \sqrt{R_{\sigma}^2 - r_{\sigma}^2 \left(\frac{n-1}{n} \right)} \quad \text{and} \quad CD_{\tau} = \frac{1}{\sqrt{2}} \sqrt{R_{\tau}^2 - r_{\tau}^2 \left(\frac{n-1}{n} \right)} \quad (9)$$

- Realisation of n measurements σ_i and τ_i ($i = 1$ to n) on the ILQ specimen and calculation of the average values:

$$\bar{\sigma} = \frac{1}{n} \sum_{i=1}^n \sigma_i \quad \text{and} \quad \bar{\tau} = \frac{1}{n} \sum_{i=1}^n \tau_i \quad (10)$$

- The goniometer is considered qualified or verified if the two following conditions on the normal stress and the shear stress are fulfilled:

$$|\sigma_{ref} - \bar{\sigma}| \leq CD_{\sigma} \quad \text{and} \quad |\tau_{ref} - \bar{\tau}| \leq CD_{\tau} \quad (11)$$