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Introduction

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National Committee	Clause/ subclause	Paragraph/ Figure/ Table	Type of comment (General/ technical/editorial)	COMMENTS	Proposed change	OBSERVATIONS OF THE SECRETARIAT
	3.1	1st definition	Editorial	Definition is ambiguous and needs clarifying.	Amend to read '... so that the mains connector to which no connection ...'	
	6.4	§ 2	Technical	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	

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DRAFT
prEN 15305

July 2005

ICS

English version

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

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 138.

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Foreword

This document (prEN 15305:2005) has been prepared by Technical Committee CEN/TC 138 "Non-destructive testing", the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

Introduction

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

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

1 Scope

This European standard describes the 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 5.1.1);
- Highly textured materials;
- Coarse grain material (see 5.1.3);
- 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 document 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.

Safety aspects related to the use of X-ray equipment are not considered in this document. During the measurements the adherence to relevant safety procedures as imposed by law are the responsibilities of the user.

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

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

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

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

ISO 5725-2, *Application of statistics - Accuracy (precision and reliability) of the results and methods of measurement – Part 2: Basis for the determination of the repeatability and the reproduction from a standard method.*

3 Terms, symbols and definitions

For the purposes of this standard, the following terms, symbols and definitions apply

3.1 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 planes.
- ψ The angle between the normal of the specimen and the normal of the diffracting 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 crystalline 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.
  - (L_1, L_2, L_3) Laboratory coordinate system.
  - ${}^1/_2 S_2^{\{hkl\}}$, $S_1^{\{hkl\}}$ 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.

— $r_{\sigma cert}$, $r_{\tau\ cert}$, Repeatability of the normal stress, shear stress, and line width respectively of the certified ILQ stress- r_{Lcert} reference specimen

— $r_{\sigma ref}$, $r_{\tau ref}$, Repeatability of the normal stress, shear stress, and line width respectively of the LQ stress-reference r_{Lref} 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 : $Tr(\sigma) = \Sigma\sigma_{ii}$.

— 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

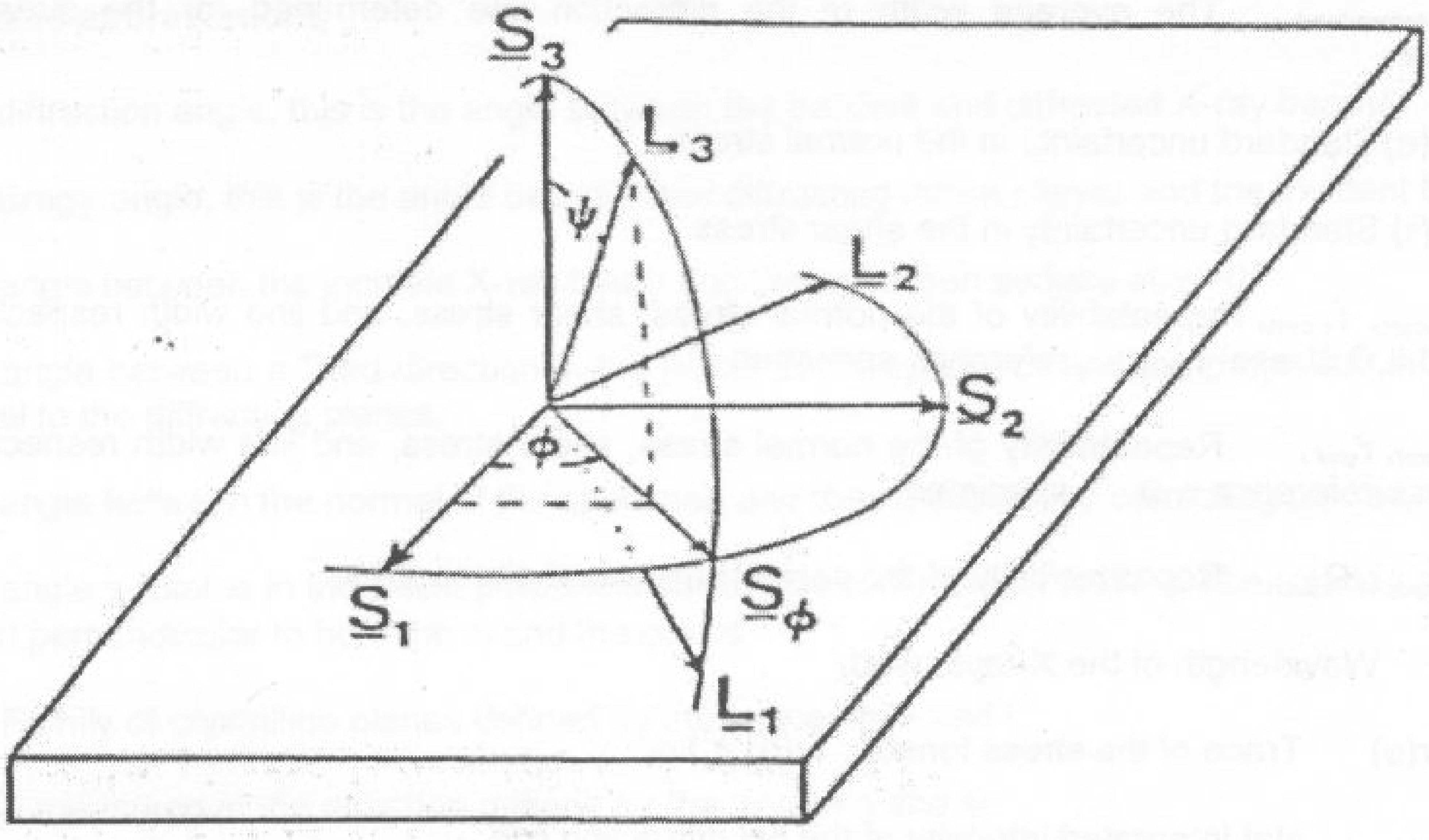
3.2 Terms and definitions

3.2.1 Residual stress

Self-equilibrating internal stresses existing in a free body which has no external forces or constraints acting on its boundary.

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	Normal axe to the specimen surface
L_1, L_2, L_3	Laboratory coordinate system; L_3 is normal to the diffracting{hkl} lattice planes and it is the bisector of the incident and diffracted beams
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 planes
ψ	The angle between the normal of the specimen and the normal of the diffracting planes
$S\phi$	The direction where the $\sigma\phi$ and $\tau\phi$ stresses are measured

Figure 1 — Orthogonal coordinate systems.

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 Fig.1) is:

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

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 Fig. 1):

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

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

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

$\varepsilon_{\phi\psi}^{\{hkl\}}$ is the strain in the direction defined by the angles ϕ and ψ for the family of lattice planes {hkl};

$S_1^{\{hkl\}}$ and $\frac{1}{2}S_2^{\{hkl\}}$	are the X-ray elasticity constants for the family of lattice planes {hkl};
σ_{11}, σ_{22}, σ_{33}	are normal stress components in the directions S_1, S_2 and S_3;
τ_{12}	is the shear stress within the plane defined by S_1 and S_2;
τ_{13}	is the shear stress within the plane defined by S_1 and S_3;
τ_{23}	is the shear stress within the plane defined by S_2 and S_3.
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 planes.
ψ	the angle between the normal of the specimen and the normal of the diffracting planes.
σ_ϕ	is the normal stress value in a direction defined by the angle ϕ
σ_{11}, σ_{22}	are normal stress components in the directions S_1, S_2;
τ_ϕ	is the shear stress value in a direction defined by the angle ϕ

$d_{\phi\psi}$ is the spacing of the family of lattice planes {hkl} in the direction defined by ϕ and ψ and d_0 is the strain-free lattice spacing of the same family of lattice planes {hkl}, thus the strain $\varepsilon_{\phi\psi}$ is given by the formula:

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

or alternatively by the approximate formulae:

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

or

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

where:

$d_{\phi\psi}$	is the spacing of the family of lattice planes {hkl} in the direction defined by ϕ and ψ
d_0	is the strain-free lattice spacing of the same family of lattice planes {hkl}
θ_0	is the Bragg angle associated to d_0
$\theta_{\phi\psi}$	the Bragg angle defined in the direction ϕ and ψ according to Bragg Law (see EN13925-1, pag.6)

The formula (2c) is far too approximate (errors in the order of a few percent are introduced), thus it is deprecated 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 B). A practical estimation of d_0 is given by the *d* value measured at $\sin^2\psi = 0.4$. 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, the equation (1) can be simplified:

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

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

For the usual methods (ω and χ method, see clause 6.1) 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 E).

Note that the elasticity constants of the {hkl} planes may be significantly different from that 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 (3) that the dependence of $\varepsilon_{\phi\psi}$ on $\sin^2\psi$ is linear:

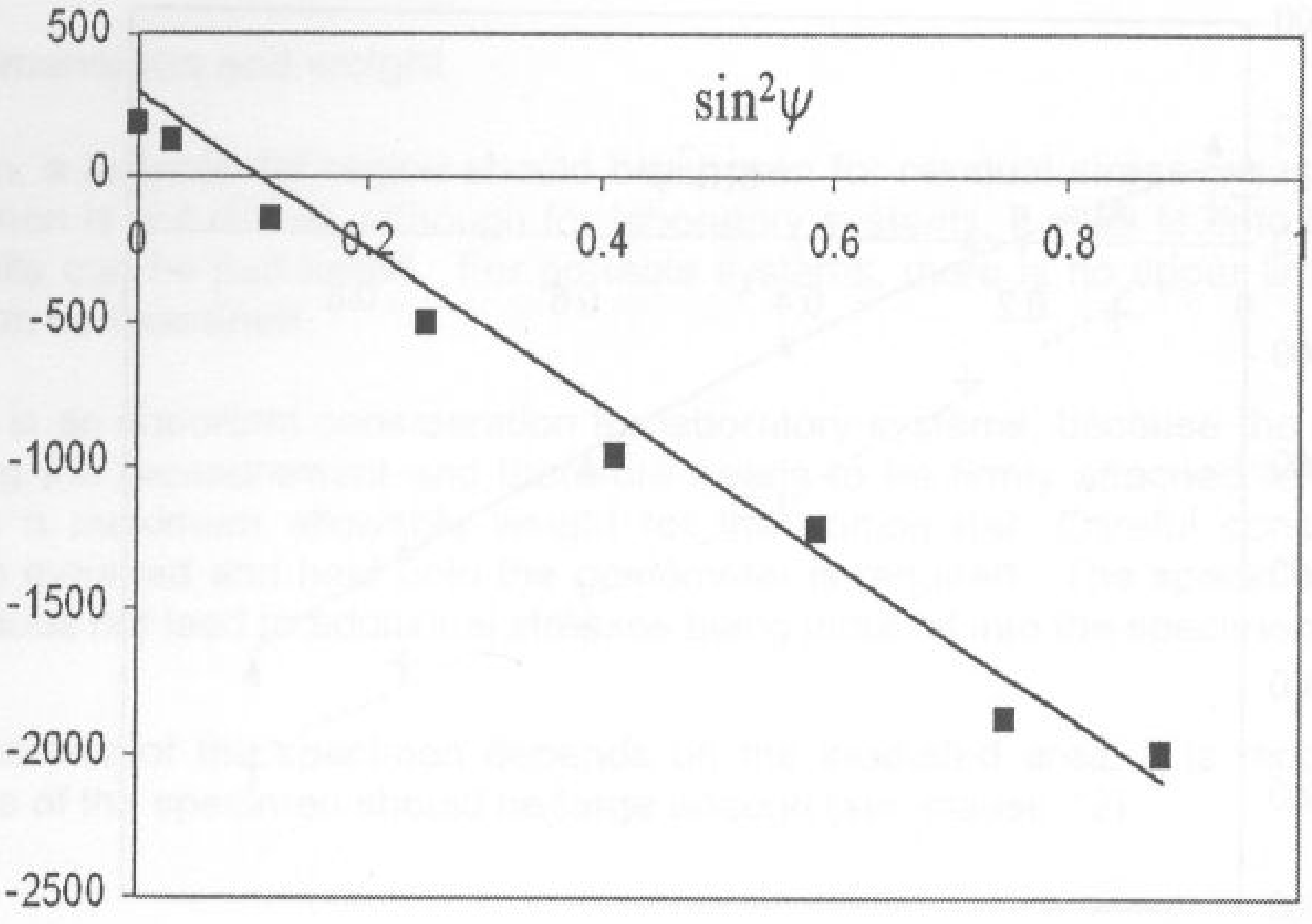
$\varepsilon_{\phi\psi}^{\{hkl\}} = 1/2\ S_2^{\{hkl\}}.\sigma_\phi \sin^2\psi + S_1^{\{hkl\}}.\mathrm{Tr}(\sigma)$ **(4)**

where:

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

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:

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



Key

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

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

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

In figure 2 the material undergoes a stress state with sF = -400 MPa, tF = 0. The X-ray elasticity constant of the material is ½ S2{hkl}= 6.8 10-6 MPa-1. The negative and positive ψ values are superposed and denoted by squares. The lines correspond to the least square fitting by equation (4).

Due to the insufficient accuracy of d_0, the stresses obtained from Tr(σ) should not be used for further calculations.

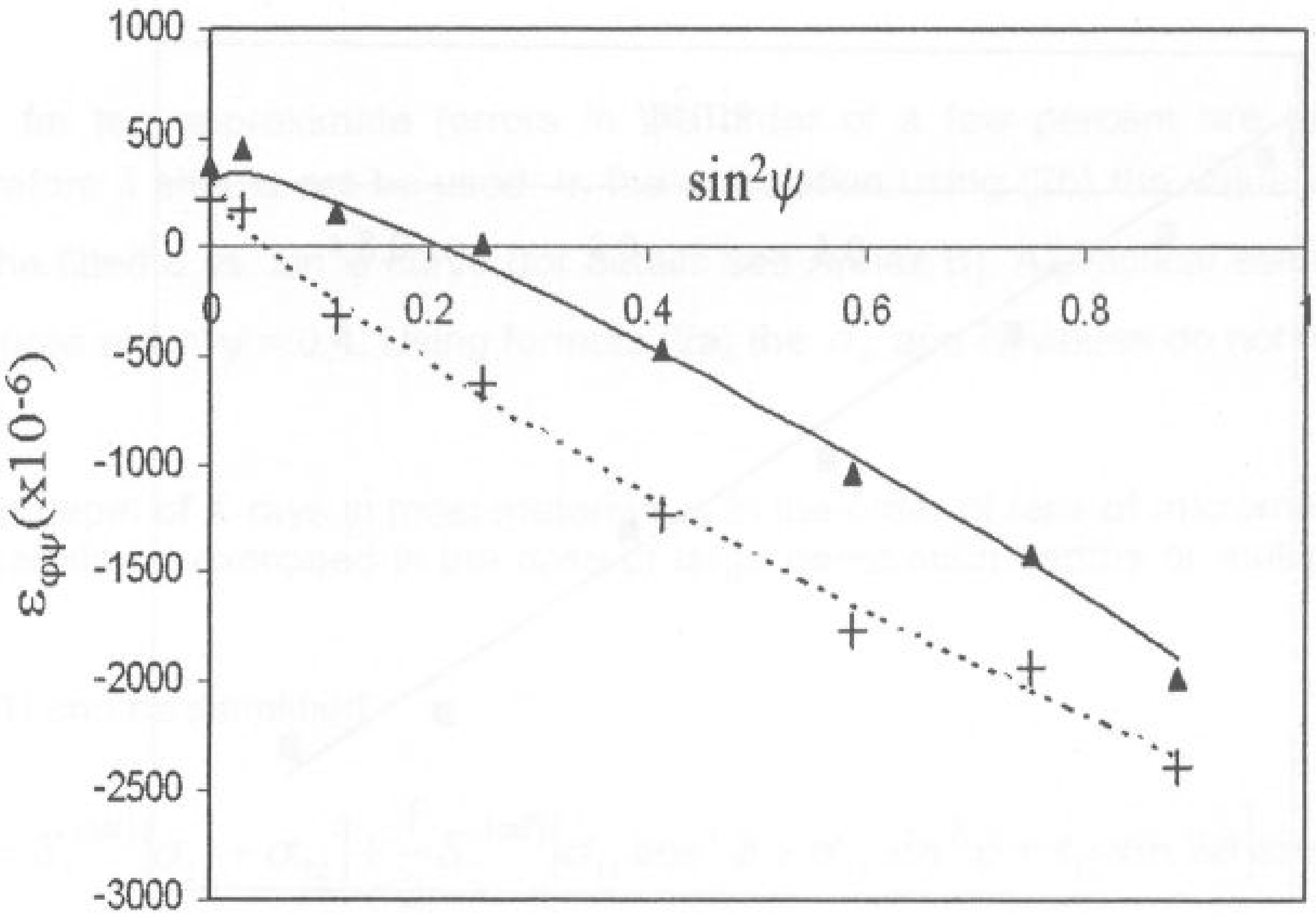
4.3 Triaxial stress analysis

If shear stresses acting in the planes perpendicular to the specimen surface are present ($\tau_{13} \neq 0$ or $\tau_{23} \neq 0$) then the plot of $\varepsilon_{\phi\psi}$ vs. $\sin^2\psi$ is an ellipse, showing the so-called "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 (4) becomes:

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

where

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



Key

$\varepsilon_{\phi\psi}$	strain measured in the direction defined by the angles ϕ and ψ
ψ	The angle between the normal of the specimen and the normal of the diffracting planes
+	positive ψ values
▲	negative ψ values

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

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 ½ $S_2\{hkl\}$ = 6.8 10^{-6} 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 fitting the strain data in equation (5). By measuring at least three different ϕ directions the stress tensor can be derived (see clause 7.4) and at least three ψ angles.

5 Specimen

5.1 Material characteristics

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

— the crystallographic data of the material;

— the X-ray elasticity constants.

A possible source of significant systematic error is the use of incorrect values of the X-ray elasticity constants (1/2 $S_2^{\{hkl\}}$ and $S_1^{\{hkl\}}$) in the stress calculations, because the calculated residual stress is proportional to the values of the X-ray elasticity constants, which may differ by as much as 40% from the bulk values. Thus, whenever possible, the X-ray elasticity constants should be experimentally determined (see clause 10).

When values have not been determined experimentally, 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).

Prior knowledge of the specimen history and its microstructure can indicate if problems might occur as listed in clause 1.

For details see clause 12.

5.1.1 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, although for laboratory systems, it shall fit onto the specimen stage such that the various tilts can be performed. For portable systems, there is no upper limit in theory to the size of specimens that can be examined.

Specimen weight is an important consideration for laboratory systems, because the specimen is subjected to various tilts during the measurement and therefore needs to be firmly attached to the specimen stage and because there is a maximum allowable weight for the goniometer. Careful consideration as to how the specimen is to be mounted and held onto the goniometer is required. 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 should be large enough (see clause 12).

5.1.2 Specimen composition/homogeneity

The phase composition and volume fractions should be constant within the irradiated volume. 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 6.2).

In multiphase materials the measured residual stress is determined from the diffraction line of a specific phase.

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

where

$\sigma^{overall}$ residual stress of overall specimen

x_i is the volume fraction of the i phase

σ_i the stress in the i phase

It is therefore mandatory that the phase from which the diffraction line originates be known.

5.1.3 Grain size and diffracting domains

The grain size in the irradiated volume can also affect the residual stress value found. In many crystalline materials grain sizes are in the range 10-100μm. As grains often consist of many diffracting domains, these values are usually suitable 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 overcome this problem by oscillating the specimen (for details see clause 7).

5.1.4 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 D). In addition, the effect of stress gradients and significant stress σ_{33} will be more pronounced.

5.1.5 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

The stress can be determined as a function of depth by subsequent electro polishing and stress analyses. 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.

5.2.2.1 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 should be avoided. Chemical attack or electro polishing is suggested to remove layers without introducing new stresses on the surface. However chemical attack or electro polishing may cause relaxation of the residual stresses due to increased 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].

5.2.2.2 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 should, 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.

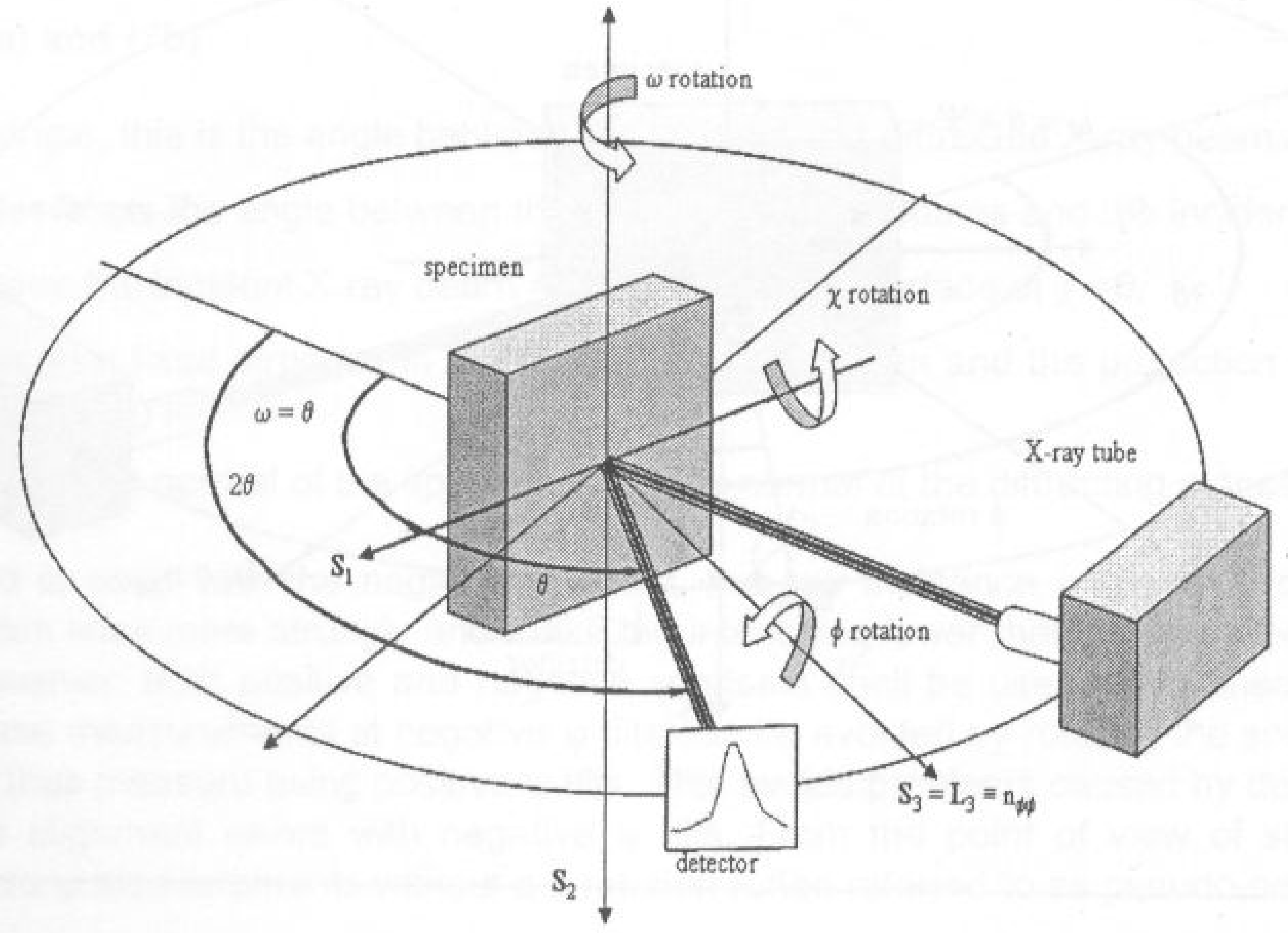
In addition, one should avoid overall relaxations which may occur, e.g., when cutting pipes in the axial direction. If relaxations are expected, methods such as strain gauging [3] should be used to monitor any changes during or after cutting.

6 Equipment

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

6.1 Choice of equipment

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	The Bragg angle, this is the angle between the diffracting lattice planes and the incident beam
2θ	The diffraction angle, this is the angle between the incident and diffracted X-ray beams
ω rotation	rotation along the ω axe
ϕ rotation	rotation along the ϕ axe
χ rotation	rotation along the χ axe

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

The ψ tilt can be performed in two classical geometries (called ω and χ method) shown in Figure 5-6, other geometries can also be used.

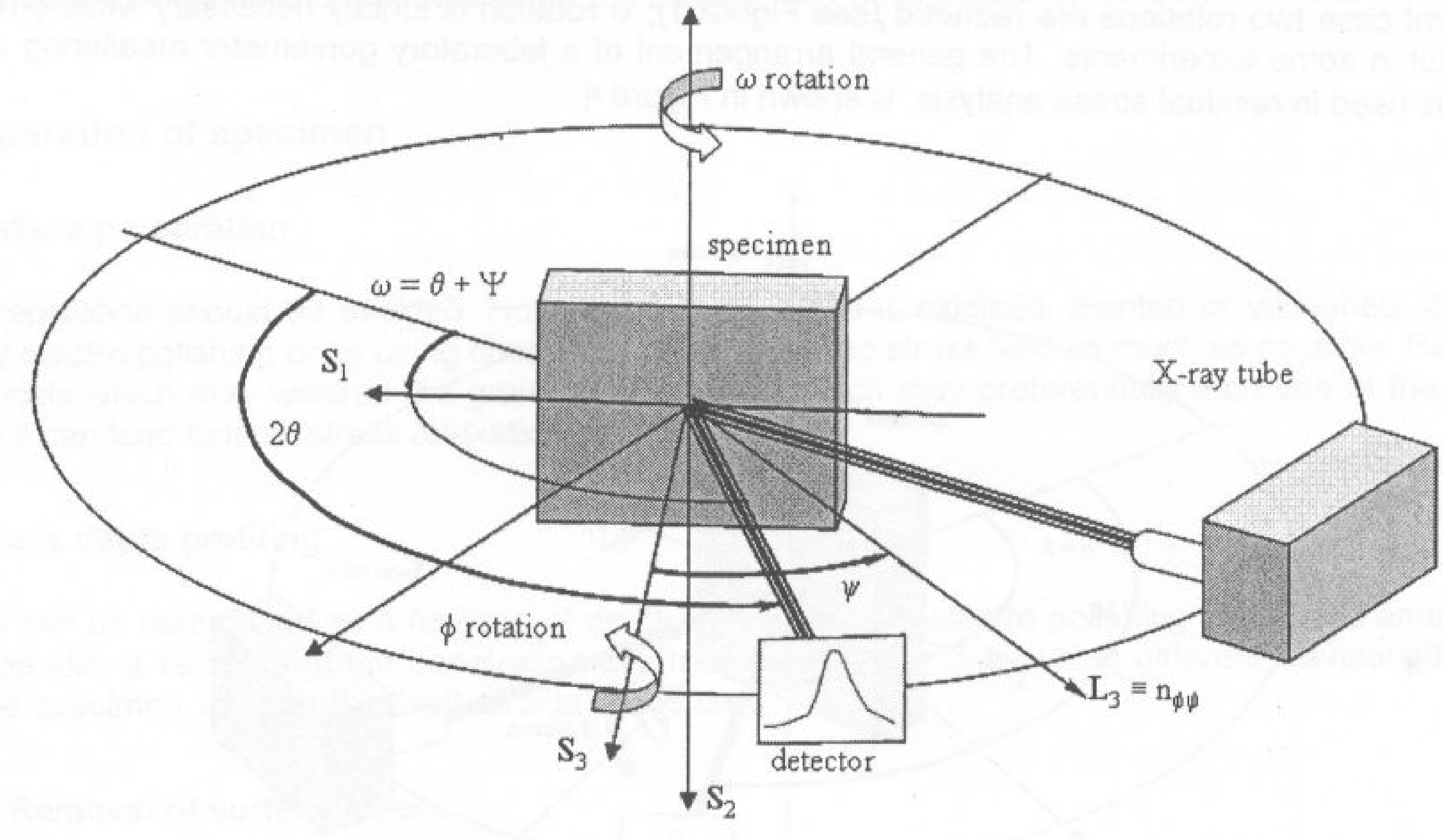
Laboratory goniometers achieve a good precision in the measurement, permitting generally an extensive choice of 2θ angles; they are often used in other applications of X-ray diffraction (texture, profile analysis, phase analysis, (see EN 13925-1)). However, generally only non-cumbersome specimens can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

In the case of a portable system, the ϕ and ψ rotations are performed by moving the source and the detector with respect to the surface area to be investigated. Therefore the object investigated can remain in a fixed position implying almost no limit on its size and weight.

6.1.1 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 de-coupled 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 S_ϕ direction lies in the plane of diffraction (see also Annex E).



Key

S_1, S_2, S_3	Specimen coordinate system
θ	The Bragg angle, this is the angle between the diffracting lattice planes and the incident beam
2θ	The diffraction angle, this is the angle between the incident and diffracted X-ray beams
ω rotation	rotation along the ω axe
ϕ rotation	rotation along the ϕ axe

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

Positive and Negative Offsets

Figure 7a shows a specimen with a positive ψ-offset, for the ω-method, where ψ has been added to θ. Figure 7b shows a negative ψ-offset where ψ has been subtracted from θ.

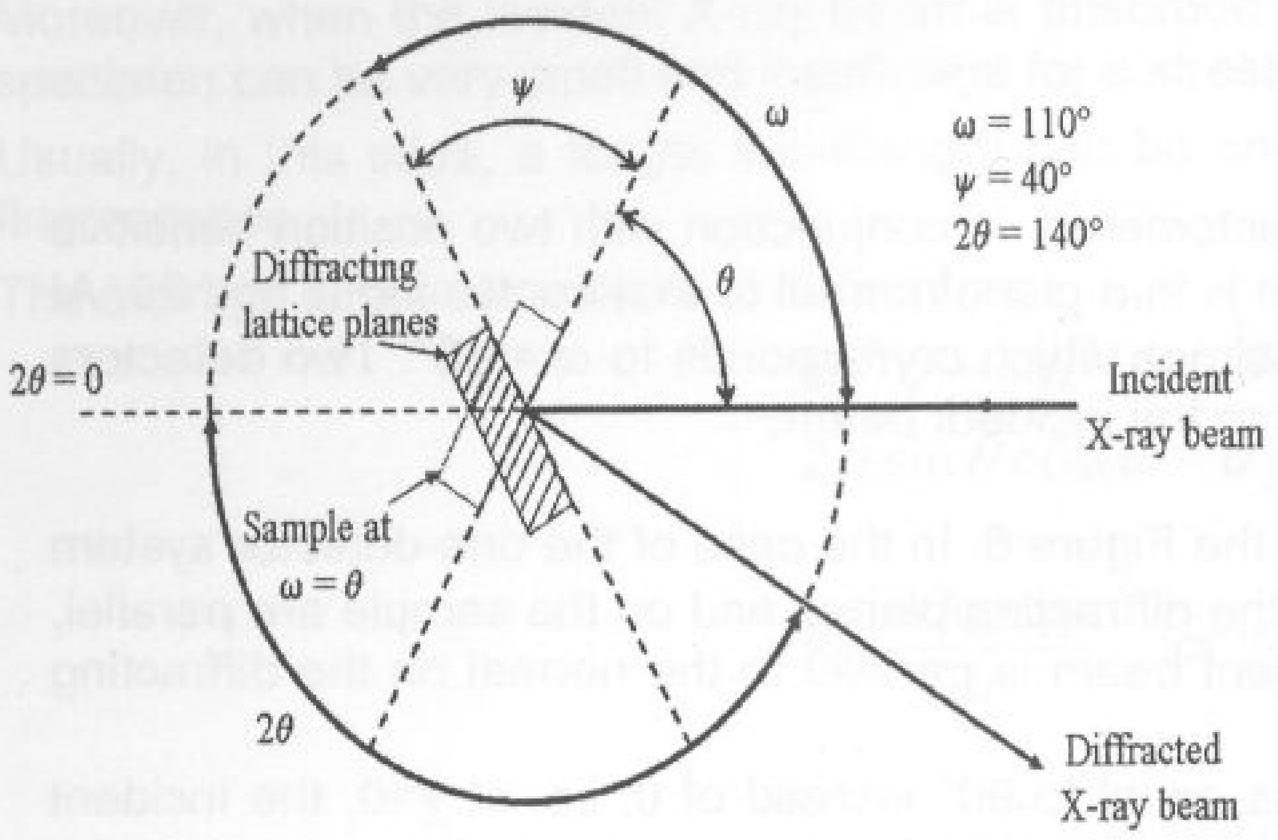


Figure 7a — Positive ψ-offset ($\omega = \theta + \psi$)

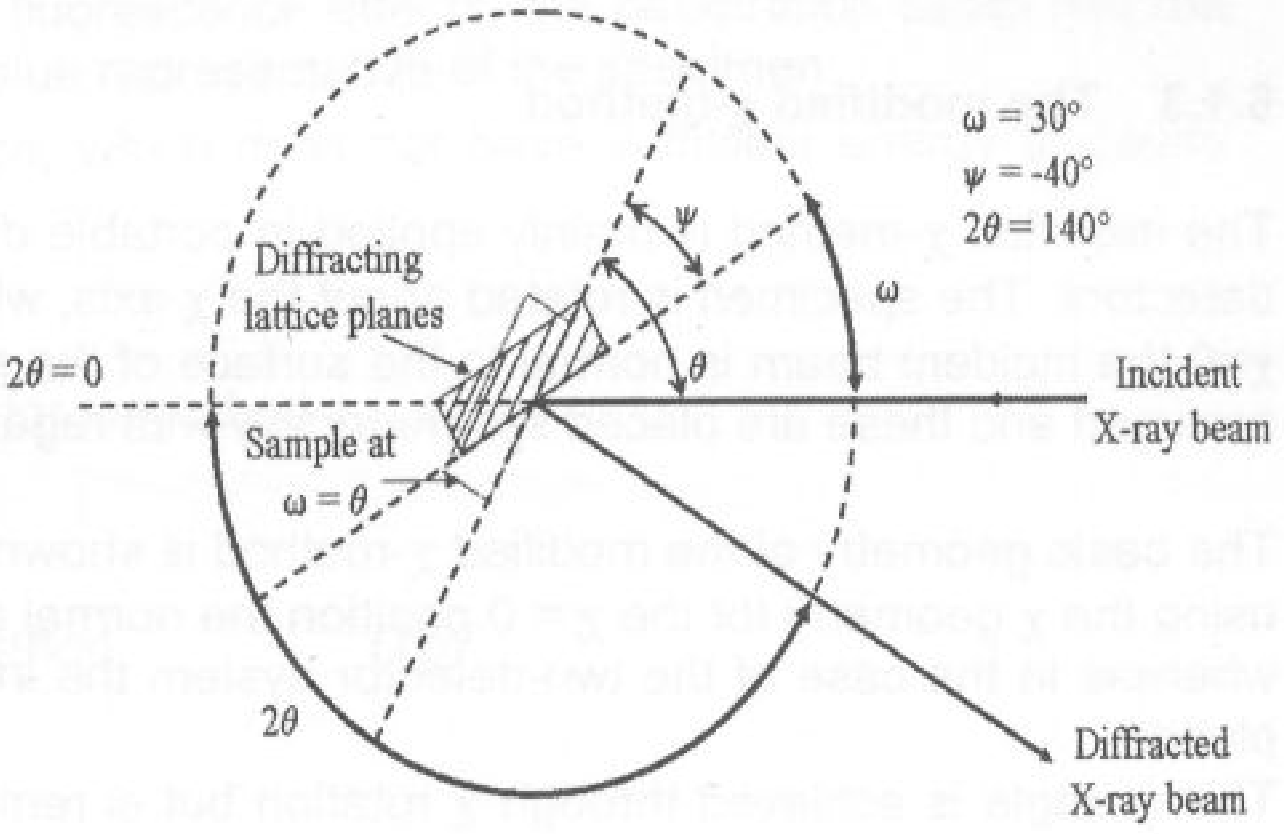


Figure 7b — Negative ψ-offset ($\omega = \theta + \psi$, with $\psi<0$)

Key for Figures (7a) and (7b)

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 planes.

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

When the ω-method is used with the negative ψ-offset, the low incidence angle and "defocusing" effects, broaden the diffraction lines more strongly and make the intensities lower than those collected with 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 thus measure 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.

6.1.2 The χ-method

Figure 6 shows the χ-method, which is also called the side inclination method. Here the specimen is rotated about the χ axis, which lies in the equatorial plane.

Note that the $S\phi$ 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 around ϕ axis of 180° and applying positive ψ.

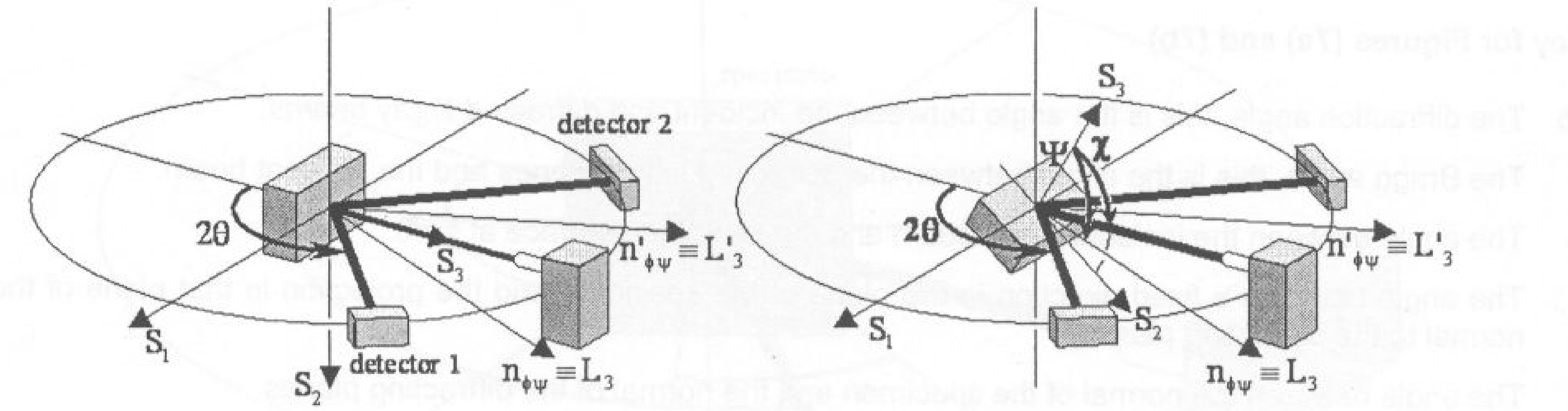
The advantage in using the χ-method is that defocusing effects are the same for positive and negative ψ. Generally the χ-method leads to smaller errors related to the specimen centring than for the ω-method.

This method is sometimes confusingly referred to as ψ method.

6.1.3 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 χ=0 the incident beam is normal to the surface of the specimen which corresponds to ω = 90°. Two detectors are used and these are placed symmetrically with regards to the incident beam.

The basic geometry of the modified χ-method is shown in the Figure 6. In the case of the one-detector system using the χ geometry for the χ = 0 position the normal on the diffracting 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 planes.
The ψ angle is achieved through χ rotation but ω remains equal to 90° instead of θ, i.e. at χ=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 χ=50°(right), the measurement is performed for ϕ=90°(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	is normal to the diffracting {hkl} lattice planes
2θ	The diffraction angle, this is the angle between the incident and diffracted X-ray beams.
ψ	The angle between the normal of the specimen and the normal of the diffracting 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

Figure 8 — The modified χ-method

6.1.4 Other geometries

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

6.2 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 Kα_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:

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

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

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

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

z X-ray penetration depth

θ The Bragg angle

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

χ The angle χ rotates in the plane perpendicular to that containing ω and 2θ.

μ The linear absorption coefficient.

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

Examples of diffraction conditions (wavelength, filter, {hkl} planes, Bragg angle) for common materials are reported in Table 1.

Alloy	Crystalline system	Anode	K_β filter	{hkl}	2θ(°) 1)	Multiplicity of the planes	Average information depth of X-rays in µm at ψ=0 1)2)
Nickel alloys	c	Mn	Cr	{311}	From 152 to 162	24	4.9
Ferritic steels	c	Cr	V	{211}	156	24	5.8
Cast iron (matrix)							
Austenitic steels	c	Mn	Cr	{311}	152	24	7.2
Aluminium alloys	c	Cr	V	{311}	140	24	11.5
Aluminium alloys	c	Cu	Ni	{422}	137	24	35.5
Cobalt alloys	c	Mn	Cr	{311}	From 153 to 159	24	5.6
Copper alloys	c	Mn	Cr	{311}	149	24	4.2
Titanium alloys	h	Cu	Ni	{213}	142	24	5.0
Molybdenum alloys	c	Fe	Mn	{310}	153	24	1.6
Zirconium alloys	h	Fe	Mn	{213}	147	24	2.8
Tungsten alloys	c	Co	Fe	{222}	156	8	1.0
Alumina - alpha	r	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	c	Cu	Ni	{844}	146	24	38.5
		V	Ti	{440}	128	12	8.8

1) Indicative values.

2) 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.

Table 1 — Diffraction conditions for common materials

The radiation source 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 that 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].

6.3 Choice of the detector

Detectors are different in type, size and shape (see 4.3 of prEN13925-3)

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 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.

6.4 Performance of the equipment

6.4.1 Alignment

Primarily, the alignment shall be in accordance with the requirements of prEN 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 6.5 and D3.1) 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.

6.4.2 Performance of the goniometer

Additionally to the demands of the current document, the performance of the goniometer shall follow the requirements of prEN 13925-3. (clause 5).

6.5 Qualification and verification of the equipment

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.

NOTE: Work is in progress on how to define and where to manufacture and buy certified specimens [5].

6.5.1 Qualification

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

6.5.1.1 Qualification of an instrument with 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.

Criteria of qualification.
The equipment is certified if the stress free specimen gives the following result:

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

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

where

$^1/_2S_2^{\{\mathrm{hkl}\}}$ 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 $1/2S_2^{\{\mathrm{hkl}\}}$ used should be that of the analysed material, not that of the powder of the stress-free specimen.

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

When available, the qualification shall be performed on an ILQ stress-reference specimen (see 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 a ILQ stress-reference specimen shall be performed in the following steps:

— Choose the number n (n > 4 is recommended) of measurement to be done on the specimen in repeatability conditions [see clause 11.3.1.2] for the qualification (or the verification). The choice of n depends on the quality policy of the laboratory.

— Calculation of the critical difference, CD, for the normal stress and for the shear stress:

$$\mathrm{CD}_\sigma = \frac{1}{\sqrt{2}}\sqrt{\mathrm{R}_\sigma^{\ 2} - \mathrm{r}_\sigma^{\ 2}\left(\frac{\mathrm{n}-1}{\mathrm{n}}\right)} \quad \text{and} \quad \mathrm{CD}_\tau = \frac{1}{\sqrt{2}}\sqrt{\mathrm{R}_\tau^{\ 2} - \mathrm{r}_\tau^{\ 2}\left(\frac{\mathrm{n}-1}{\mathrm{n}}\right)} \tag{9}$$

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

$$\overline{\sigma} = \frac{1}{n}\sum_{i=1}^{n}\sigma_i \quad \text{and} \quad \overline{\tau} = \frac{1}{n}\sum_{i=1}^{n}\tau_i \tag{10}$$

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

$$|\sigma_{ref} - \overline{\sigma}| \le CD_\sigma \quad \text{and} \quad |\tau_{ref} - \overline{\tau}| \le CD_\tau \tag{11}$$

where for formulae (9), (10), (11)

CD σ	normal stress Critical Difference
CD τ	share stress Critical Difference
R_σ, R_τ	are the reproducibility values
r_σ, r_τ	are the repeatability values (see also clause 11.3.2)
$\overline{\sigma}$	average normal stress on n measurements
σ_i	normal stress of the i measurements
$\overline{\tau}$	average share stress on n measurements
τ_i	average stress of the i measurements
σ_{ref}	Normal stress value of the LQ specimen.
τ_{ref}	Shear stress real value for the LQ specimen.

6.5.2 Verification of the performance of the qualified equipment

The verification shall be performed using both a stress-free specimen and a stress-reference specimen (ILQ or LQ).

6.5.2.1 Verification of the performance of an instrument with a stress free specimen

Verification with a stress free specimen.
Proceed as in 6.5.1.1
Note: the choice of n can be different for a qualification and verification.

6.5.2.2 Verification of the performance of an instrument with a stress-reference specimen

If a LQ stress-reference specimen is used, the three following criteria shall be verified:

— the determined stress shculd satisfy:

— $$\left|\sigma_{\mathrm{ref}} - \sigma_{\mathrm{determined}}\right| \le \frac{r\sigma}{\sqrt{2}} \qquad \textbf{(12)}$$

— the determined shear stress should satisfy:

— $$\left|\tau_{\mathrm{ref}} - \tau_{\mathrm{determined}}\right| \le \frac{r_\tau}{\sqrt{2}} \qquad \textbf{(13)}$$

— the determined width of the line should satisfy:

— $$\left|L_{\mathrm{ref}} - L_{\mathrm{determined}}\right| \le \frac{r_L}{\sqrt{2}} \qquad \textbf{(14)}$$

Where for formulae (12), (13), (14)

σ_{ref}	Normal stress value of the LQ specimen.
$\sigma_{determined}$	Determined Normal stress value of the stress-reference specimen.
τ_{ref}	Shear stress real value for the LQ specimen.
$\tau_{determined}$	Shear stress value determined for the stress-reference specimen.
L_{ref}	Average width of the diffraction lines for the LQ specimen.
$L_{determined}$	The average width of the diffraction line determined for the stress-reference specimen.
r_σ, r_τ, r_L	the repeatabilities obtained by the laboratory on the internal specimen (see clause 11.3.1.2).

If an ILQ stress-reference specimen is used, proceed as in 6.5.1.2.

7 Experimental Method

7.1 General

The following steps should be followed when performing X-ray residual stress measurements:

- Verification of the alignment of the diffractometer (see clause 6.5) and, where appropriate, the calibration of the detector.
- Positioning of the specimen (see clause 7.2).
- Choice of diffraction conditions (see clause 7.3).
- Choice of measurement conditions and data collection (see clause7.4).
- Visual check of the diffraction lines (see clause 8.4.2).
- Data treatment (see clause 8).
- Reporting (see clause 9).

7.2 Specimen positioning

The relationship between the specimen and the incident X-ray beam/tilting direction defines the residual stress measurement direction.
The specimen to be measured shall be placed at the centre of rotation of the goniometer (see clause 6.4.1), the specimen should be oriented so that the stress being measured is in the direction of interest (see Fig. 1).

The irradiated surface area should have low curvature and a negligible stress gradient in the plane of the surface (see clause 12). The position of the irradiated area should be defined precisely. The area can be kept constant by masking a part of the specimen e.g. using wax. However, at high ψ angles, the thickness of the mask may induce a "shadow zone", thus the ψ angle range should be limited.

The centre of the investigated area should be in the centre of rotation of the goniometer (see clause 6.4.1) within 10-100μm depending on the setup of the goniometer. An incorrect positioning with respect to the ψ origin will lead to an inaccurate shear stress. The position of the specimen surface should be invariant to any angular movement in ϕ or ψ. The positioning procedure can be checked with the help of reference specimens during a verification operation as described in clause 6.5.

7.3 Diffraction conditions

The phase considered and the indexing of the diffraction line to be used shall be known.

The diffracting planes, the wavelengths and the instrumental conditions shall be chosen in order to have:

— high diffraction angle;

— no overlapping diffraction lines;

— good background definition;

— adequate average information depth.

Commonly used conditions are given in Table 1 (clause 6.2)

The microstructural effects (grain size, texture, multiphase material, etc.) or stress gradients can make it difficult or even impossible to analyse the diffraction line. In these cases, stress values obtained by measurements made on different phases or different crystallographic planes may be different (see clause 12). In textured specimens often measurements on reflections with a high multiplicity are beneficial. If the specimen has a large grain size it may also help to select the reflection with a high multiplicity and/or a wavelength with a deeper penetration depth.

The prior knowledge of these problems helps with the choice of the best operating conditions to better facilitate the treatment of the data.

For accurate comparisons with previous data/measurements it is useful to check the planes and the wavelengths previously used and, if possible, select the same ones.

For details see clause 12.

7.4 Data collection

The main parameters for data collection are:

<u>Acquisition time and measurement step size</u>:

The counting time required to obtain a sufficiently accurate diffraction pattern (see clause 8.4.2) will vary depending on the tube, the optics, the detector (see prEN 13925-3 clause 4.3), and specimen characteristics.

Sufficient data points should be collected to describe the upper part of the line. The step size should be between FWHM/20 and FWHM/10.

The measurement range should be, if possible, chosen in order to have enough background on each side of the peak. A measurement range larger than four times the FWHM of the peak is usually adequate. Care should be taken if another diffraction line is close to the measured peak: the diffraction range should be limited so that the background subtraction is not perturbed. Sometimes not subtracting the background gives better results than subtracting a poor estimation of the background.

<u>Choice of ϕ and ψ angles</u>:

If there is no shear stress τ_{ϕ}, i.e. in the plane normal to the specimen surface in the investigated ϕ direction, at least 4 - 5 measurements in a range of $\sin^2\psi$ values as large as possible (typically 0.5 or more) and constant $\sin^2\psi$ steps are recommended. Due to defocusing problems or beam overflow there is a practical limit to the maximum ψ value. For example in focusing geometry it is usually taken 15° less than the theoretical limit ($|\psi| = 90°$ for the χ -method and $|\psi| = \theta$ for the ω-method).

If a shear stress normal to the specimen surface in the investigated ϕ direction seems to be present, at least 7 measurements in the $\sin^2\psi$ range are necessary with negative and positive values of ψ in order to analyse the ψ-splitting. More than 9 measurements are recommended.

For the ω-method, pseudo-negative angles (i.e. positive ψ with additional 180° ϕ rotation) are recommended when negative ψ tilts are required for the analysis of ψ-splitting. In this case, true negative ψ angles (i.e. 'glancing'/small incident angle; large deflected angle) should be avoided because of the strong sensitivity to specimen displacement and equatorial beam misalignment (even when these errors are small).

If the principal directions are not known, full stress/tensor determination requires at least three independent ϕ directions. The usual way to choose these directions is 0°, 45° and 90°. However it is advisable to use a larger ϕ range and/or more independent ϕ angles. To obtain the uncertainties for the stress values at least four ϕ values are required. The difference between the ϕ angles should be chosen according to:

$$\Delta\phi = 180°/n$$

where

$\Delta\phi$ The difference between the ϕ angles

n is the number of independent ϕ angles.

For each ϕ at least 7 ψ directions in the $\Delta\sin^2\psi$ range are necessary with negative and positive values of ψ.
In contrast to the determination of stresses in particular directions ϕ, determination of the full stress tensor requires the knowledge of the unstressed lattice spacing value, d_0, with at least the precision needed to calculate the stress normal to the specimen surface ($\Delta\varepsilon$ about 1 part in 10^5).

Oscillations:

This method can be applied to materials that have large domain sizes (such as castings, forged goods, welds, etc.) with the aim of increasing the number of domains that contribute to the diffracted signal. Generally the options are:

- oscillate the specimen around any axis (χ, ψ, ϕ, ω) to increase the number of grains meeting the criterion for diffraction. The oscillations can be very large ± 10° or more [6]. However, oscillations greater then 10° are not recommended. When specimen oscillations are used, the acquisition time per step shall allow an integer number of complete oscillation.
  For linear detectors, when used in the scanning mode, an integration takes place over a range of ω-angles which is equivalent to ω oscillation.
  For area detectors, when the γ-integration is used to generate the diffraction profile, it actually integrates the data collected in a range of various diffraction vectors (see Annex C) resulting in a virtual oscillation.

- the specimen can be translated along S_1 and/or S_2 directions during the measurement in order to cover a larger area of the surface. Translation during a measurement in both S_1 and S_2 directions can give a marked improvement in the diffraction line profile.

X-ray Tube Power

The X-ray tube should in general be operating near its maximum recommended power output, so that the diffraction line can be recorded in the minimum time possible.

7.5 "Normal Stress Measurement Procedure" and "Dedicated Stress Measurement Procedure"

7.5.1 General

Two stress measurement procedures are defined and described that conform to this standard and that yield results (stress values) of which it is allowed to be denoted as "stress measurements according to this standard":

a) the Normal Stress Measurement Procedure that shall be applied to specimens with an unknown stress state and to specimens where the stress state is not assessed or proven;

b) the Dedicated Stress Measurement Procedure that can be applied to series of very similar specimens (routine analyses) e.g. with the purpose to reduce the measurement effort.

The Dedicated Stress Measurement Procedure is introduced to enable qualified and standardized stress measurements for e.g. quality control and stress mapping, and it shall guarantee a proven and tractable one to one relationship between the results obtained with the Normal Procedure and the Dedicated Stress Measurement Procedure.

7.5.2 Normal stress measurement procedure for a single specimen

The normal stress measurement procedure is described in clauses 7.1 to 7.4

7.5.3 Dedicated Stress Measurement Procedure for very similar specimens

Specimens are regarded here as "very similar" if the differences between their stress states (not the stress values), their chemical and phase compositions, their texture, their microstructure are expected to be insignificant for the stress values to be determined.

For a series of such specimens a laboratory can define and describe a Dedicated Stress Measurement Procedure. In order to conform to this standard such a Dedicated Stress Measurement Procedure shall be validated through the execution of the Normal Procedure as described in clause 7.5.1 as well as the Dedicated Stress Measurement Procedure. The validation procedure requires that:

1) the validation measurements shall be performed on several specimens of the series to establish the allowable range of stress values.

2) the equipment shall be aligned and validated/qualified according to clauses 6.4 and 6.5 before starting the validation of the Dedicated Stress Measurement Procedure as well as before starting the measurement on the series of specimens.

3) it proves that the value obtained by the Dedicated Stress Measurement Procedure is appropriate and sufficient for the purpose, i.e. that the reduction of measurement effort is justified.

The procedure shall define (i) quantitatively the acceptable deviations from the expectations of the intermediate results (e.g. the linearity of $\varepsilon_{\phi\psi}$ versus $\sin^2\psi$ plots, the shear stress value, the peak width) that are relevant to the stress determination and (ii) the procedure to be followed if unacceptable deviations are observed.

If the Dedicated Stress Measurement Procedure obeys the above, then the stress results obtained using that procedure are accepted as conforming to this standard.

8 Treatment of the data

8.1 General

In what follows the treatment of the data is described.

Analysis of the diffraction lines and calculation of the stress may be performed using proprietary software supplied with the particular diffractometer being used, or other software. It shall be verified that the software used provides adequate tools for performing the data treatment as described in this clause and for reporting as described in clause 9.

The complete treatment of the measurement data is subdivided into three stages:

i) Treatment of the diffraction data to obtain peak positions;

ii) Stress calculation from peak positions;

iii) Critical assessment of the results.

8.2 Treatment of the diffraction data

8.2.1 General

General treatments of diffraction data and analysis are reported in EN 13925-1 “General Principles”, clause 6 and EN 13925-2 “Procedures” subclause 6.3, Annex D and Annex E.

8.2.2 Intensity corrections

A precise determination of the position of a diffraction line can be obtained after applying the appropriate corrections to the intensities. The following corrections are commonly used:

Divergence slit correction.

If automatic (or variable) divergence slits have been used a separate correction shall also be applied in order to convert the intensities to 'fixed slit' intensities.

Absorption factor A.

The absorption factor quantifies the influences of the absorption of the radiation by the material of the specimen on the intensity profiles of the diffracted beams. It can depend on the θ, χ, and ω angles.

Background removal

Subtraction of the background intensity contribution

Lorentz-polarisation factor LP.

The Lorentz-polarisation factor quantifies the influences of geometrical factors on the intensity profiles of the diffracted beams.

Alpha2 stripping

The pure $K\alpha_1$ diffraction pattern is obtained from a $K\alpha_1+\alpha_2$ diffraction pattern by means of a numerical $K\alpha_2$ stripping procedure.

The details of diffraction data treatment are given in Annex D

8.2.3 Determination of the diffraction line position

Several methods exist for the determination of the diffraction line position. The parameters of the chosen method shall be specified. The most commonly used are:

- Centre of Gravity method
  - Classical Centre of Gravity
  - Sliding Centre of Gravity
  - Threshold Centre of Gravity
  - Centred Centre of Gravity (Centred Centroid)
- Polynomial fit
  - 3-point parabola
  - Multiple-point parabola
  - Higher order polynomial
- Profile function fit
  - Gauss
  - Lorentz also called Cauchy
  - Modified Lorentz (Pearson VII with m=2)
  - Intermediate Lorentz (Pearson VII with m=1.5)
  - Pearson VII
  - Pseudo-Voigt
  - Voigt
  - Pearson IV
- Middle of width at % height method
  - Middle of FWHM (50% height)
  - Middle of 2/3 height (67% height)
- Cross correlation method

Other methods are also possible. Refer to Annex D for details and recommendations.

The chosen method shall ensure good repeatability.

8.2.4 Correction on the diffraction line position

In the case of a low absorbing material, transparency introduces a shift of the diffraction line position (see clause 5.1.4). The data shall be corrected (see Annex D.3.2).

In the case of any remaining misalignments, further corrections can be performed (see Annex D.3.1).

8.3 Stress calculation

8.3.1 Calculation of strains and stresses

8.3.1.1 Strain calculation

Formulae are given in 4.1.

8.3.1.2 Stress calculation

1) Choice of X-ray elasticity constants (XECs)

The XECs ($S_1^{\{hkl\}}$ and $1/2S_2^{\{hkl\}}$) of the material under analysis should be experimentally obtained (see clause 10). They may also be calculated from mechanical models e.g. Kröner, Mori-Tanaka, Hill models or taken from the literature. The use of the Voigt or Reuss models is not recommended.

The use of macroscopic elasticity constants is not recommended if the X-ray elasticity constants are available

Note that the use of wrong XECs in the calculation of stress will introduce systematic errors proportional to the stress (but may be acceptable for comparative measurements).

The XEC's used and their origin shall be reported.

2) Stress calculations

a) Analysis in a single direction.

A straight line (equation (4)) can be fitted to the data if no shear stress is present. Otherwise an ellipse (equation (3)) shall be fitted.

b) Tensor analysis.

An appropriate fitting procedure shall be used to fit equation (5).

8.3.2 Errors and uncertainties

8.3.2.1 General

Guidelines for the expression of uncertainty can be found in the ISO 1993 Guide to the Expression of Uncertainty in Measurement (Geneva, Switzerland: International Organisation for Standardisation), L. Kirkup A guide to GUM, Eur. J. Phys. 23 (2002) 483–487)

Sources of errors and uncertainties can be:

- Material: microstructure, chemical homogeneity, stress homogeneity, etc.
- Experimental parameters: diffraction parameters (see EN 13925-2), specific choices of ϕ, 2θ, χ and ω angles, etc.
- Assumptions of the chosen mechanical model: biaxial, triaxial, anisotropic model, etc.

In the report information shall be given on the method used to estimate uncertainty.

8.3.2.2 Errors

Errors should be kept as low as possible.

In addition to the issues mentioned in clause 12 at least the following points shall be considered:

- beam misalignments,
- detector calibration (in the case of 1D or 2D position sensitive detectors),
- beam divergence,
- specimen displacement,
- models used for diffraction data treatment,
- mechanical models used for stress calculation.

Some of these errors are smaller at high 2θ angles.

8.3.2.3 Uncertainties

The uncertainty is composed of several contributions. The most commonly considered contributions are:

- Counting statistics: the repeatability of the diffraction line position is mainly influenced by the height of the peak, by the height to background ratio, and the height to noise ratio.
- Dispersion on strain values: it can be calculated from the standard deviation of the strain values by least squares fitting.

Generally, standard deviations shall be given. A confidence interval can also be quoted.

8.4 Critical assessment of the results

8.4.1 General

All the criteria listed in 8.4.2 and 8.4.3 shall be verified. If the criteria mentioned there are not fulfilled, one shall:

1) verify not to be in a limiting case (see clause 12);
2) reprocess the data (see clause 8);
3) perform a new measurement.

8.4.2 Visual inspection

8.4.2.1 For a single diffraction line

Check for overlaps with neighbouring diffraction lines, truncations, irregular shape of the diffraction line and the background.

8.4.2.2 For the complete set of diffraction measurements

The data shall be inspected by following all these steps:

- plot one of the following quantities versus $\sin^2\psi$: $\Delta 2\theta_{\varphi\psi}{}^{\{hkl\}}$, $2\theta_{\varphi\psi}{}^{\{hkl\}}$, $d_{\varphi\psi}{}^{\{hkl\}}$,$\varepsilon_{\varphi\psi}{}^{\{hkl\}}$; Check for non-linear or non-elliptic behaviour (see Figs. 2 and 3).

- the net integrated intensities $I_{\varphi\psi}{}^{\{hkl\}}$ after applying the corrections in Annex D.1 shall be plotted versus $\sin^2\psi$. These intensities are likely to be the same for all ψ-angles recorded for a random, non-textured specimen. Large changes in intensity are indicative of a highly textured material or coarse diffracting domains (see clause 12 and 8.4.3.2).

- plot the line width versus $\sin^2\psi$. Usually an increase is observed due to defocusing effects. Check for sudden changes.

8.4.3 Quantitative inspection

8.4.3.1 For a single diffraction line

Diffraction lines can be considered for further analysis if at least the following conditions are fulfilled:

- 0.8 times the mean of the experimental peak widths ≤ peak width ≤ 1.2 times the mean of the experimental diffraction line widths;
- maximum intensity of the diffraction line ≥ 300 counts above background.

Comparison with previous experiments on the same or a similar material is suggested.

8.4.3.2 For the complete set of diffraction measurements

The complete set of diffraction measurements can be considered for further analysis if at least the following conditions are fulfilled:

- \- for the complete dataset the ratio of the integrated intensity $\frac{\text{Max } I_{\phi\psi}^{\{\text{hkl}\}}}{\text{Min } I_{\phi\psi}^{\{\text{hkl}\}}}$ shall be smaller than 3 to exclude strongly textured materials (see 12.4.1).
- \- the standard deviation of the diffraction line widths shall be less than 10% of the average width.

Criteria on the uncertainty of the normal stress:

a) if $|\sigma| \geq \frac{1}{\left(400 \cdot 1/2S_2^{\{hkl\}}\right)}$ then $|u(\sigma)|$ shall be ≤ $\frac{1}{\left(1600 \cdot 1/2S_2^{\{hkl\}}\right)}$; **(15a)**

b) if $|\sigma| < \frac{1}{\left(400 \cdot 1/2S_2^{\{hkl\}}\right)}$ then $|u(\sigma)|$ shall be ≤ $\frac{1}{\left(5000 \cdot 1/2S_2^{\{hkl\}}\right)}$ or $\leq \frac{1}{4}|\sigma|$ whichever is the largest. **(15b)**

Criteria on the uncertainty of the shear stress:

$|u(\tau)| < \frac{1}{\left(10000 \cdot 1/2S_2^{\{hkl\}}\right)}$ **(16)**

where for formulae (15a), (15b), (16)

$u(\sigma)$	Standard uncertainty in the normal stress
$u(\tau)$	Standard uncertainty in the shear stress
$^1/_2S_2^{\{hkl\}}$	Elasticity constant of the family of lattice planes{hkl}

9 Report

The experimental conditions, analysis procedures and results shall be recorded.

In particular the following information shall be recorded:

- A reference to this document.

— Details of any treatment applied to the specimen prior to stress measurement, which may have altered the condition of the surface. Examples are grinding, polishing (mechanical or electrochemical) and chemical treatments. If a depth profile of stress is being measured using material layer removal, details of the method by which material is removed should be reported.

— Sufficient information about the specimen, e.g. schematic drawing, to locate points and to identify directions of the measurements

— Type of equipment including the type of specimen tilting.

— Technical parameters: current and tension of the X-ray generator, Kβ filter if used, the values of λ, {hkl}, 1/2 $S_2^{\{hkl\}}$ and $S_1^{\{hkl\}}$, the reference specimen used, the irradiated area dimension, 2θ range, 2θ step, step counting time, values of ϕ and ψ.

— Main information about the fitting routine used, or, if not known, the name and version of the software package used to analyse the data (method of diffraction line position determination). Any variable parameters used in the peak fitting routine: e.g., constant or sloping background selected, percentage of the peak used for a centre of gravity calculation, etc.

— Method used for residual stress analyses (e.g. biaxial or triaxial method).

— The results: a) relevant tables and diagrams,

— b) values of the residual stress determined,

— c) type and values of uncertainties for the stress values as defined in 8.3.2 and confidence level, if required.

The report shall at least contain:

— A reference to this document

— Sufficient information about the specimen, e.g. schematic drawing, to locate points and to identify directions of the measurements;

— Information about the conformity of the measurement with respect to the standard;

— X-ray elasticity constants;

— The results:

a) relevant tables and diagrams,

b) values of the residual stress determined,

c) type and values of uncertainties for the stress values as defined in 8.3.2 and confidence level, if required.

The report should also contain:

— The d-$\sin^2\psi$ or ε-$\sin^2\psi$ - curves and one observed profile. If the diffraction lines recorded are atypical (because of texture, for example), it may be appropriate to report all diffraction lines along with the results.

— General information concerning chemical composition, thermal history and any processing performed on the specimen.

— If a depth profile of stress is being generated, the depth of each measurement should be recorded relative to the initial surface position. If depth variation is obtained by changing the incident X-ray wavelength or intensity, the position of the diffracting gauge volume centroid below the surface should be reported, along with an indication of how its position has been calculated.

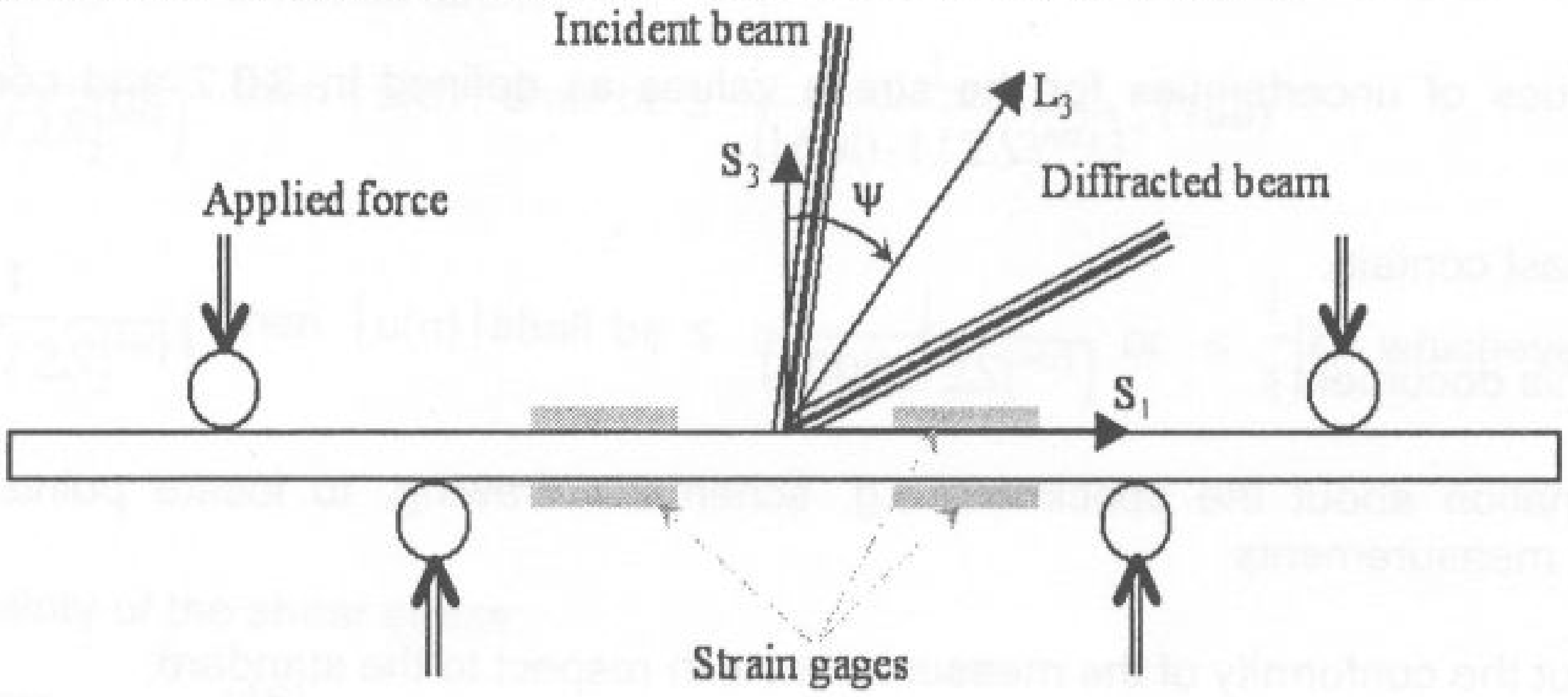
10 Experimental determination of XECs

10.1 Introduction

For the correct calculation of residual stresses the X-ray elasticity constants (XECs) $S_1^{\{hkl\}}$ and 1/2 $S_2^{\{hkl\}}$ shall be used. These constants can be found in the literature, calculated through micro/macro mechanical models or obtained by experiment, as described in this chapter.
The values obtained by experiment are correct for single phase materials. In the case of multiphase materials a mechanical model is required for an interpretation of the results [150].

10.2 Loading device

The measurement of the constants requires a specific device to allow the loading of the specimen inside the diffractometer.
The device shall be designed to apply uniform loads on the irradiated area, taking into account the measurement geometry. During the loading, the diffractometer measurement area of the loaded specimen shall be always in the centre of the goniometer.
The load can be pure tension, shearing or bending. Usually four-points bending is recommended.
In order to know the stress, force measurement devices shall be used or strain gauges shall be applied on the specimen. The loading device shall have been previously calibrated (see 10.3)



Key

S_1, S_3	Specimen coordinate system
L_3	Laboratory coordinate system
ψ	The angle between the normal of the specimen and the normal of the diffracting planes.

Figure 9 — Example of 4 points bending test to determine the X-ray elasticity constant (XEC).

In figure 9 the stress is tensile and the measurement is done in ω method

10.3 Specimen

The specimen used for the determination of the XECs should be made of the same material (chemical composition, microstructure, etc.) as the specimen for residual stress measurements.
It is necessary to accurately control the strain and/or the stress applied. If strain gauges or an extensometer are used, the macroscopic elasticity constants E and ν are required. If strain gauges are used, one or more strain gauges shall be applied on the specimen. At least one strain gauge shall be applied parallel to the specimen longitudinal axis. It is advisable to apply the strain gauges as near as possible to the measuring area.

10.4 Loading device calibration and specimen accommodation

The device (for the specimen instrumented with strain gauges) shall be calibrated with a traction standard testing machine (i.e. a tensile testing machine) or by means of calibrated dead weights. The stresses in the measurement zone are determined as a function of applied load and geometry. The calibration steps are:

- to perform at least two cycles of loading and unloading from zero to 75% of the yield load (load that would cause in the specimen a stress equal to the yield limit), in order to verify that the strain-gauge electrical signal returns to zero at the end of each cycle;
- and then to perform three cycles of loading and unloading between 5% and 75% of the yield load.

10.5 Diffractometer measurements

The device should be placed at the centre of the diffractometer such that the specimen can be positioned according to clause 7.2. Different loads should be applied and for each one the measurement shall be performed with the specimen oriented in the longitudinal direction.
The applied stress or strain in the measured area shall be evaluated for example by strain gauges.
The measurements shall start from high loads, with measurements performed for decreasing loads to avoid effects caused by inelastic deformation of the specimen. The minimum number of loading steps shall be five, regularly distributed between 70% and 5% of the yield load.

10.6 Calculation of XECs

For each step of loading, the average applied stress shall be calculated in order to determine $\varepsilon_{\phi\psi}$:

$$\varepsilon_{\phi\psi} = 1/2\ S_2^{\{hkl\}} (\sigma_{11}{}^{R} - \sigma_{33}{}^{R} + \sigma_{11}{}^{A}) \sin^2 \psi + 1/2\ S_2^{\{hkl\}}.\tau_{13}{}^{R}\ \sin 2\psi + 1/2 \cdot S_2^{\{hkl\}}\ \sigma_{33}{}^{R} + S_1^{\{hkl\}} [\, \mathrm{Tr}(\sigma^{R}) + \sigma_{11}{}^{A} \,] \quad (17)$$

The elliptical curve (see Eq. 5) $\varepsilon_{\phi\psi} = a \cdot \sin^2 \psi + b \cdot \sin(2\psi) + c$ for each load shall be drawn. The slope '*a*', splitting '*b*' and intercept '*c*' are evaluated by the least squares method.

- $a = 1/2\ S_2^{\{hkl\}} (\sigma_{11}{}^{R} - \sigma_{33}{}^{R} + \sigma_{11}{}^{A})$ (18a)
- $b = 1/2\ S_2^{\{hkl\}}.\tau_{13}{}^{R}$, (18b)

and

- $c = 1/2 \cdot S_2^{\{hkl\}}.\sigma_{33}{}^{R} + S_1^{\{hkl\}}. [\, \mathrm{Tr}(\sigma^{R}) + \sigma_{11}{}^{A} \,]$ (18c).

Where for formulae (17), (18a), (18b) and (18c)

$\varepsilon_{\phi\psi}{}^{\{hkl\}}$	is the strain in the direction defined by the angles ϕ and ψ for the family of lattice planes {hkl};
$^1/_2 S_2^{\{hkl\}}$	Elasticity constant of the family of lattice planes{hkl}
$\sigma_{11}{}^{A}$	applied stress,
$\sigma_{11}{}^{R}$, $\sigma_{22}{}^{R}$ and $\sigma_{33}{}^{R}$	residual normal stresses
$\tau_{13}{}^{R}$	residual shear stress,
$\mathrm{Tr}(\sigma^{R})$	trace of residual stresses.

The plot of the slope '*a*' against the applied stress, gives a straight line of slope $1/2\ S_2^{\{hkl\}}$.

The plot of splitting '*b*' values against the applied stress, shall give a horizontal line of slope 0.
The plot of intercept '*c*' values against the applied stress, gives a straight line of slope $S_1^{\{hkl\}}$.
Uncertainties shall be estimated for instance through least squares method and given with the results.

11 Reference specimens

11.1 Introduction

Incorrect alignment of the equipment leads to displacements of the diffraction line.
To verify the alignment the use of a flat specimen without stresses, for example a powder, is suggested.
Measurements on a stress-reference specimen give an indication of errors due to the misalignment of the goniometer or to electronic or mechanical drifts.

11.2 Stress-free reference specimen

The reference powder used shall have a diffraction line with a similar position to that of the specimen under study (see prEN 13925-3).
The powder shall have fine grains and have sufficient intensity of the reflection in all directions. Annealing treatment of the powder is sometimes useful to improve the line definition.

11.2.1 Preparation of the stress-free specimen

The stress-free reference specimen can be prepared by covering a flat, non-crystalline substrate (e.g. a glass plate) with a layer of powder by:

a) sedimentation from a liquid (e.g. propanol-2);

b) smearing grease in a layer as thin as possible, depositing the powder onto it, pressing it gently, and carefully shaking off the surplus;

c) deposition on a double sided adhesive film and pressing it gently, and carefully shaking off the surplus;

d) mixing it with grease, depositing the mixture on the glass plate, and levelling it in order to get a flat surface.

e) Mixing it with greasy, liquid glue (without crystalline components) and solvent, depositing the mixture on the glass plate, and levelling it in order to get a flat surface.

Attention shall be paid to avoiding dissolution of the powder or the substrate, or chemical interaction (such as polymerisation), which can introduce stresses.

For the cases a, b, and c:
- Adhesion of the powder shall be checked by turning the specimen upside-down and checking for falling powder;
- it is advised to use a compound with a high average atomic mass in order to have a sufficiently strong diffraction pattern of the powder and to reduce the intensity diffracted by the substrate through absorption by the powder.

The use of a thin slab of a single-crystal (e.g. a silicon wafer) as a flat substrate is not recommended because of the risk of severe damage to the detector by the very strong diffraction of the substrate at some ϕ, ψ combinations.

Grease and double-sided adhesive film can yield a significant undulating contribution to the background of the pattern of the reference powder. Therefore, the thinner the layer of grease or the double sided adhesive film the better.