
Neporušitveno preskušanje – Uklon rentgenskih žarkov na polikristalnih in amorfni materialih – 2. del: Postopki

Non-destructive testing - X-ray diffraction from polycrystalline and amorphous materials - Part 2: Procedures

Zerstörungsfreie Prüfung - Röntgendiffraktometrie von polykristallinen und amorphen Materialien - Teil 2: Verfahrensabläufe

Essais non destructifs - Diffraction des rayons X appliquée aux matériaux polycristallins et amorphes - Partie 2: Procédures

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Ta slovenski standard je istoveten z: EN 13925-2:2003

ICS:

19.100 Neporušitveno preskušanje Non-destructive testing

SIST EN 13925-2:2004

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 13925-2

March 2003

ICS 19.100

English version

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

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This European Standard was approved by CEN on 29 November 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

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Foreword

This document (EN 13925-2:2003) 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of an identical text or by endorsement, at the latest by September 2003, and conflicting national standards shall be withdrawn at the latest by September 2003.

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

- EN 13925-1 General principles;
- EN 13925-2 Procedures;
- prEN 13925-3 Instruments;
- prEN 13925-4 Reference materials.

In order to explain the relationships between the topics described in the different standards, a diagram illustrating typical operations involved in XRPD analysis is given in annex A.

Annexes A to E are informative.

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

Introduction

X-ray powder diffraction (XRPD) is a powerful Non Destructive Testing (NDT) method for determining a range of physical and chemical characteristics of materials. These include the type and quantities of phases present, the crystallographic unit cell and structure, crystallographic texture, macrostress, crystallite size and microstrain, and the electron radial distribution function.

This standard aims to describe the general aspects of the XRPD technique and its applications but not to define a specific or detailed standard for each field of application or type of analysis.

The main purposes of the standard are therefore:

- to provide practical guidance, unified concepts and terminology for use of the XRPD technique in the area of Non Destructive Testing with general information about its capabilities and limitations of relevance to laboratories working at different levels of sophistication, from routine testing to research;
- to provide a basis for Quality Assurance in XRPD laboratories allowing performance testing and monitoring of instruments as well as the comparison of results from different instruments;
- to provide a general basis (without imposing specifications) for further specific NDT product standards and related Quality Assurance applications, with aspects common to most fields of application.

In order to make the standard immediately usable in a wide range of laboratories and applications, diffractometers with Bragg-Brentano geometry are considered in more details than the diffractometers using other geometries.

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.

1 Scope

This European Standard specifies the basic procedures applied in the X-ray Powder Diffraction (XRPD) method. Many of these procedures are common to most types of diffractometer used and types of analysis mentioned in EN 13925-1. In the interests of clarity and immediate usability more details are given for procedures using instruments with Bragg-Brentano geometry and application to phase identification. Aspects of specimen preparation and data quality assessment are included, but the standard remains non-exhaustive. It is anticipated that particular standards will address specific fields of application in more details.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 13925-1:2003, *Non-destructive testing — X-ray diffraction from polycrystalline and amorphous materials — Part 1: General principles*.

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

3 Terms and definitions

For the purposes of this European Standard, the general terms and definitions concerning X-ray powder diffraction¹⁾ apply.

4 Specimen preparation

The sample treatment and specimen preparation shall be adapted to the nature of the sample and the type of analysis in order to optimise the quality of the data to be collected [1], [2]. As explained in clause 5 of EN 13925-1:2003, the term 'powder' when used in crystallography, does not strictly correspond to the common usage.

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4.1 General preparation

4.1.1 Lateral specimen size

When theta-compensating variable apertures are used, the surface area of the specimen irradiated by the beam can be kept constant (but not the volume from which diffraction is measured). The specimen shall always intercept the whole incident beam to avoid a loss of diffracted intensity. This can be checked, for example, by initially investigating the range of angles to be measured, after replacement of the specimen with a fluorescent screen of the same dimensions. Alternatively, the length of the specimen surface irradiated by the X-ray beam can be calculated using the equation:

$$\text{Irradiated length (mm)} = R \alpha / \sin \theta \quad (1)$$

where

R is the radius of the goniometer, in millimetres;

α is the divergence angle of the beam, in radians;

θ is half the diffraction angle 2θ , in degrees or radians.

In practice, with fixed aperture slits, the incident beam at low 2θ angles often spreads beyond the specimen surface. The corresponding diffracted intensities can be approximately corrected by comparing them with data recorded in the same angular domain but using a fixed slit of smaller aperture.

¹⁾ a European draft standard (WI 00138078 "Non-destructive testing – X-ray powder diffraction – Terminology") is in preparation

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When a small amount of a powder is to be examined, the diffracted intensity can be maximised by preparing it to form a thin layer with a large area intercepting the incident X-ray beam.

When specimens with low attenuation are investigated on diffractometers working in a reflection geometry, it should also be taken into account that, at low diffraction angles, the incident and diffracted X-rays may propagate a considerable distance in a direction nearly parallel to the specimen surface. Thus the optimum lateral sample size might be considerably larger than the area of intersection of the incident X-ray beam with the specimen surface.

4.1.2 Effect of specimen displacement

A specimen surface that is offset with reference to the Bragg-Brentano goniometer 2θ rotation axis, results in a line shift by an angle, in radians of²⁾

$$\Delta(2\theta) = 2\theta_{\text{obs}} - 2\theta_{\text{theo}} = -2\Delta h \cos \theta / R \quad (2)$$

where

$\Delta(2\theta)$ is the shift (in radians) in the theoretical line position to align it with the observed position. It is positive when the specimen surface is displaced towards the source and detector;

$2\theta_{\text{obs}}$ and $2\theta_{\text{theo}}$ are the observed diffraction angle and the diffraction angle calculated from the Bragg law;

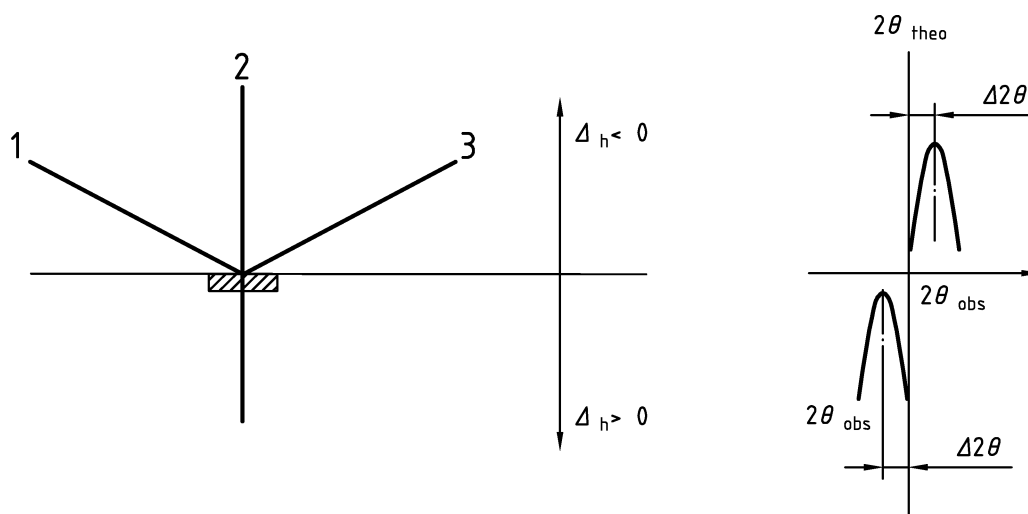
Δh is the specimen surface displacement (in millimetres) measured along the bisector of the angle between the incident beam and the diffracted beam. It is positive if the specimen surface moves away from the X-ray source and the detector.

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R is the radius of the goniometer (in millimetres).

This is illustrated schematically in Figure 1.

²⁾This equation is similar to that given by Wilson [3].



Key

- 1 Source
- 2 Bisector
- 3 Detector

The symbols are defined in equation (2)

Figure 1 — Relationships between the specimen displacement and the diffraction line position

Specimen displacements smaller than 20 μm are difficult to avoid. For example, with a goniometer of 200 mm radius, this offset would result in a maximum angular error of 0,01° (2θ).

Use of an appropriate internal standard allows the detection and correction of this effect simultaneously with that arising from specimen transparency.

4.1.3 Effects of specimen thickness and transparency

When the XRPD method is applied in a reflection geometry it is often preferable to work with specimens of 'infinite' thickness. This means that, for a given mass attenuation and apparent density of the specimen and a given range of diffraction angles, the diffracted intensity from the back of the specimen is negligible.

To ensure that the diffracted intensity is at least 99,9% of the maximum attainable by increasing the specimen thickness, the thickness shall be at least [4]:

$$t = 3.45 \sin \theta / (\mu' \rho') \quad (3)$$

where

t is the thickness, expressed in centimetres;

ρ' is the specimen density, the mass of the specimen divided by its volume including voids expressed in grams per cubic centimetre;

μ' is the weighted sum of the mass attenuation coefficients (often referred to as the mass absorption coefficient) expressed in square centimetres per gram [5]. It is additive for the mass attenuation coefficients of the constituent elements of the material when weighted by their fractional concentration, e.g.:

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$$\mu' = \sum c_i \mu'_i \quad (4)$$

where

μ'_i is the mass attenuation coefficient of the i^{th} element, expressed in square centimetres per gram;

c_i is the fractional concentration by weight of the i^{th} element.

For specimens with low attenuation (such as organic compounds) a large fraction of the diffracted intensity appears to originate from a position below the surface resulting in line shifts and changes in line widths and shapes. This effect, referred to as the transparency effect, is large for thick specimens with low attenuation. The line shift (in radians) due to the transparency of a thick specimen is given by the relationship (see footnote 1):

$$\Delta(2\theta) = 2\theta_{\text{obs}} - 2\theta_{\text{theo}} = -\frac{1}{2\mu' \rho' R} \sin 2\theta \quad (5)$$

where

$\Delta(2\theta)$ is the shift (in radians) in the theoretical line position to align it with the observed position;

$2\theta_{\text{obs}}$ and $2\theta_{\text{theo}}$ are the observed diffraction angle and the diffraction angle calculated from the Bragg law;

R is the radius (in centimetres) of the goniometer.

For such materials, the specimen should be as thin as possible (consistent with acceptable diffraction intensities) to give accurate measurement of line position. It is advisable to use a non-diffracting specimen substrate (also called a low background holder), e.g. a plate of mono-crystal silicon cut parallel to the {510} lattice planes. In the case of thin specimens with low attenuation accurate measurements of line positions can be made with focusing diffractometer configurations in either transmission or reflection geometry. Accurate measurements of line positions on thick specimens with low attenuation are preferably made using diffractometers with parallel beam optics. This helps to reduce the effects of specimen thickness.

Use of an appropriate internal standard allows the detection and correction of this effect simultaneously with that arising from specimen displacement.

NOTE "centimetre " and "gram" are the units commonly used in tables of attenuation coefficient and density.

4.2 Block specimens

4.2.1 Surface preparation

The specimen surface shall be sufficiently flat for the purpose of the measurement to be made, e.g. when using Bragg-Brentano geometry, surface roughness can result in displacement, broadening and reduced intensity of diffraction lines. Mechanical, electrolytic or chemical polishing can be carried out to obtain a flat surface or to study an area in depth or free of disturbances arising from the initial preparation.

Mechanical polishing can cause various changes in the material (strain hardening, phase changes, etc.). This altered layer shall be removed by adequate chemical or electrochemical polishing.

4.2.2 Mounting and specimen holder

Block specimens are mounted either directly into the stage of a diffractometer if the size and shape are suitable or mounted into a specimen holder that is itself mounted on the diffractometer stage. Care has to be taken in either method to ensure that the specimen surface is aligned with all the relevant rotational axes of the goniometer, including additional rotations that might be used for applications such as texture or macrostress measurement. In the case of Bragg-Brentano geometry, the specimen surface has to be aligned with the goniometer axis and be symmetrically oriented between the incident and diffracted beams.

4.2.3 Additional precautions

When examining block specimens, the possibility of depth-dependent inhomogeneity has to be recognised. It can result in a diffraction pattern with varying relative contributions from the different components as 2θ is varied. X-ray opaque masking is sometimes used to limit the irradiated surface on large specimens but care has to be taken to ensure that the mask does not contribute to the diffraction pattern. A preferred alternative, where practical, is to mask the X-ray beam to limit the area irradiated or from which diffracted X-ray are detected.

4.3 Powder specimens

4.3.1 Sampling of multi-phase powders

Prior to carrying out an XRPD investigation of an unknown powder, there shall be proper sampling followed by appropriate specimen preparation. In the case of a multi-phase powder, the unknown powder might be inhomogeneous on a microscopic or even a macroscopic scale due to differences in the properties of the individual components such as the density, size and shape of the particles, state of agglomeration, etc. To provide representative and reproducible results of an XRPD analysis, it may be necessary to homogenise an amount of the unknown powder that is much larger than the quantity needed for the specimen size.

In cases where maximum reliability of the XRPD results is required, statistical methods for homogeneity testing shall be applied. For sampling techniques see *e.g.* the BCR guidelines of the European Commission [6].

4.3.2 Milling and sieving

Milling and sieving may be required to increase the number of crystallites in the specimen or to minimise micro-absorption effects between particles of different composition and size. The issue of crystallite size is dealt with in this sub-clause in more details.

The number of crystallites of each individual crystalline phase in the irradiated specimen volume shall be sufficient to assure a desired level of reproducibility for the collected data. This problem is often denoted as "crystallite statistic". For Cu K α radiation and quartz specimens measured with Bragg-Brentano geometry, a maximum crystallite size of 10 μm has been found to achieve reproducibility of diffraction line intensity within 2 % to 3 % [7 (p. 365 ff)].

Based on this figure and the relationship given in the same work, the mean relative deviation, U_m , in diffracted intensity may be roughly estimated by:

$$U_m = 60 (\mu' \rho l^3)^{1/2} \quad (6)$$

where

ρ is the crystal density, expressed in grams per cubic centimetre;

μ' is the mass attenuation coefficient, expressed in square centimetres per gram;

l is the crystallite dimension, expressed in centimetres.

Values of U_m up to about 10%, arising from larger crystallites, often give satisfactory data for phase identification. Smaller values of U_m (and hence smaller crystallite sizes) are necessary for quantitative analysis where a higher level of reproducibility is needed. However excessive milling, giving crystallite dimensions below about 0,5 μm , may cause line broadening and significant changes to the intrinsic characteristics of the specimen, such as:

- sample contamination by particles abraded from the milling instruments (*e.g.* mortar, pestle, balls etc.);
- partial amorphisation of the near-surface region of the sample particles;
- transition to different polymorphic crystallographic forms;