

Non destructive testing — X ray diffraction from polycrystalline and amorphous materials —

Part 3: Instruments

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee WEE/46, Nondestructive testing, which has the responsibility to:

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Foreword

This document (EN 13925-3:2005) 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of an identical text or by endorsement, at the latest by November 2005, and conflicting national standards shall be withdrawn at the latest by November 2005.

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

- prEN 1330-11, Terminology - Part 11: X-Ray Diffraction from Polycrystalline and Amorphous Materials
- EN 13925-1, Part 1: General principles
- EN 13925-2, Part 2: Procedures
- EN 13925-3 Part 3: Instruments
- WI 00138070, Reference Materials

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

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

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 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 document sets out the characteristics of instruments used for X-ray powder diffraction ("powder" as defined in EN 13925-1:2003, Clause 5) as a basis for their control and hence quality assurance of the measurements made by this technique. Performance testing indicators are given for diffractometer performance testing. Different types and makes of X-ray powder diffractometer vary considerably in their design and intended fields of application. This document attempts to cover as much of this range as possible by keeping to common principles. To make the standard more readily applicable, the Bragg-Brentano configuration is addressed in most detail because of its wide use. Additional considerations and adaptations may be necessary to cover some types of instruments or configuration and some fields of application. Some of these types of instrument are described in Annex B.

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

prEN 1330-11:2004, *Non-destructive testing – Terminology – Part 11: X-ray Diffraction from Polycrystalline and Amorphous Materials*

3 Terms and definitions

For the purposes of this document, the terms and definitions of prEN 1330-11:2004 apply.

4 Description of equipment

4.1 General

This description is particularly intended for instruments dedicated to the fields of application described in EN 13925-1. For other applications, additional considerations may be required.

A diffractometer generally comprises:

- goniometer;
- X-ray source;
- incident beam optics which may include monochromatisation or filtering, collimation and/or focusing or parallelism of the beam;
- diffracted beam optics which may include monochromatisation or filtering, collimation and/or focusing or parallelism of the beam;
- specimen stage;
- detector;
- data collection system.

These parts of the instrument are considered in more detail below. A data processing system is also required to produce measurements from the instrument. Data processing systems, whether manual or computerised, shall be included with the data processing procedures of EN 13925-2.

A well-controlled environment (temperature and pressure) is strongly recommended for analysis where reproducible measurement of line profile position, width and shape is required.

Humidity may be important because compounds in the specimen may react with water or absorb it with a consequent change in their lattice constants, e.g. clay minerals.

The X-ray beam is partially scattered and attenuated by the air in the beam path with consequential effects on the detected diffraction pattern background and intensity. This effect has sometimes been minimised by use of an evacuated or helium filled beam path.

For all the items described in this clause the corresponding main characteristics to be controlled are given in Clause 5.

4.2 X-ray sources

4.2.1 General

There are several types of x-ray source that can be used for XRPD measurements ranging from conventional laboratory sources to intense and well-collimated synchrotron sources. Each source exhibits characteristics that make it more suitable for particular types of analysis. The main source types are described below.

4.2.2 Conventional X-ray sources (sealed tubes and rotating anode sources)

X-rays are obtained by bombarding a metal anode with electrons emitted by the thermoionic effect and accelerated in a strong electric field produced by a high-voltage generator. Most of the kinetic energy of the electrons is converted to heat, which limits the power of the tubes and requires efficient anode cooling. An increase of about two orders of magnitude in brilliance can be obtained using rotating anodes instead of sealed tubes. Microfocus sources operate at relatively low power settings but maintain brightness by electrostatically or magnetically steering the beam inside the X-ray tube onto the target. The spectrum emitted by a conventional X-ray source operating at sufficiently high voltage consists firstly of a continuous background of polychromatic radiation with a sharp cut off at short wavelengths determined by the maximum voltage applied. Upon this is superimposed a limited number of narrow characteristic lines whose wavelengths are characteristic of the anode material. The emitted radiation is not polarised.

The type of X-ray source and the electron emission current, and accelerating voltage applied to it by the high voltage generator have a marked effect on X-ray intensity and its energy distribution. The emission current and accelerating voltage of a conventional X-ray tube normally give reproducible adjustment of the X-ray beam intensity and energy spectrum on a time scale of days. However, experience has shown that the absolute X-ray intensity differs significantly for nominally equal sources and that it decreases with source age.

4.2.3 Synchrotron radiation sources

A beam of charged particles strongly accelerated in an electric field and deflected in a magnetic field emits a continuous spectrum of X-rays that is as much as 10^{13} times as brilliant as sealed X-ray tubes. It is called "synchrotron radiation". This increased brilliance relates to the total energy spectrum. Monochromatisation of the beam typically results in diffraction intensities one or two orders of magnitude greater than from conventional sources.

The main advantages of using synchrotron radiation for XRPD measurements are:

- nearly parallel-beam diffraction geometry;
- highly monochromatised and tunable radiation;
- very small and almost symmetric contribution of the instrument to the observed line shape that leads to simpler characterisation of line profiles and very good angular resolution.

This type of X-ray radiation source requires beam flux monitoring with time (the beam flux can decrease significantly during the experiment) and wavelength calibration. The emitted radiation is strongly polarised in the plane of deflection of the charged particles.

4.3 Incident and diffracted X-ray beam optics

4.3.1 General

The main characteristics of the incident and diffracted beams are their wavelength spectrum, their direction of propagation, their cross-sectional area and shape at the specimen as well as their degree of collimation (axial and equatorial divergence) and focusing. The items of equipment that determine these characteristics are called "beam optics". The effects of these items are referred to by the general term "beam conditioning".

The items of equipment described below are used to obtain different degrees of radiation purity and different geometries, i.e. a so-called "focused" beam, "monochromatised" beam, "collimated" beam, or "parallel" beam.

4.3.2 Monochromators

4.3.2.1 General

The wavelengths conventionally used with laboratory sources correspond to the characteristic spectral lines from specific anode materials.

For many XRPD applications it is advantageous to eliminate all other spectral components emitted from the X-ray source or the specimen. When using the $K_{\alpha 1,2}$ doublet is sometimes helpful to additionally suppress the $K_{\alpha 2}$ -line, i.e. to work with highly monochromatic $K_{\alpha 1}$ -radiation.

Monochromatisation devices can be used singly or in combination.

4.3.2.2 Filters and β filters

Partial monochromatisation can be obtained using K_{β} filters, i.e. foils made of a metal selected as having an absorption edge between the wavelengths of the K_{α} and K_{β} radiation emitted by the source.

Standard K_{β} filters are designed to reduce the K_{β} intensity to about two orders of magnitude less than the K_{α} intensity. Such a filter also attenuates the K_{α} doublet intensity, typically by a factor of 2, and reduces the polychromatic radiation from the source. Two filters are sometimes combined to form so-called "balanced filters" [1].

Spectral filters shall be used with caution because the absorption edges of the filter material cause significant steps in the background close to each of the observed diffraction lines. Such steps impede accurate analysis when the complete line profile(s) are used, for example, in line-broadening analysis.

In some cases it is useful to attenuate the incident or the diffracted beam. Then a foil is used that is made of a metal (often aluminium) selected as having no absorption edge near the radiation wavelength used.

4.3.2.3 Crystal Monochromators

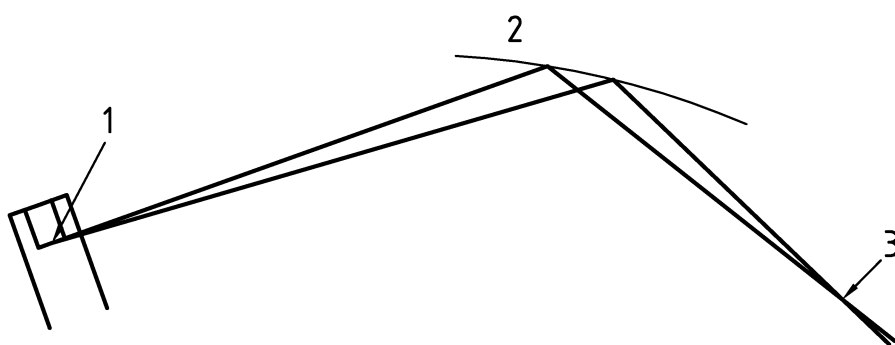
Monochromatisation of radiation is most often accomplished by diffraction from a crystal. We may distinguish:

a) a mosaic crystal. The relative energy or wavelength resolution ($\Delta E/E$ and $\Delta \lambda/\lambda$, respectively) is typically a few parts per hundred. Most of the K_{β} radiation and of the continuous polychromatic radiation is eliminated. The orientations of the coherently diffracting parts of the mosaic crystal (generally graphite) are spread over about 0,4 degrees for many commercially available monochromators. For such crystals more than 50 % of the incident intensity of the $K_{\alpha 1,2}$ doublet can be retained. Mosaic crystal monochromators are usually installed in the diffracted beam. In this configuration, they also eliminate specimen fluorescence from all elements except the one that constitutes the anode.

b) a single crystal. The energy resolution is typically better than two parts per thousand. The two components of the $K_{\alpha 1,2}$ doublet can be separated and the $K_{\alpha 2}$ radiation eliminated. This type of monochromatisation causes a large loss in intensity as typically only about 15 % of the incident intensity of a spectral line is retained. Single crystal monochromators (usually almost perfect single crystals of quartz, silicon or germanium) are generally used on the primary beam often in a focusing arrangement. The intensity loss may be largely offset by the associated reduction in axial divergence of the beam, eliminating the need for one set of Soller slits (see 4.3.3.3.2).

Two or more crystals (e.g. [2]) may be combined to achieve much better resolution than one, but the use of multiple crystals may yield insufficient intensity for normal powder diffraction. Care shall be taken to ensure that the harmonics of the chosen wavelength are eliminated by the positioning of suitable apertures (slits) within the monochromator, or by other means.

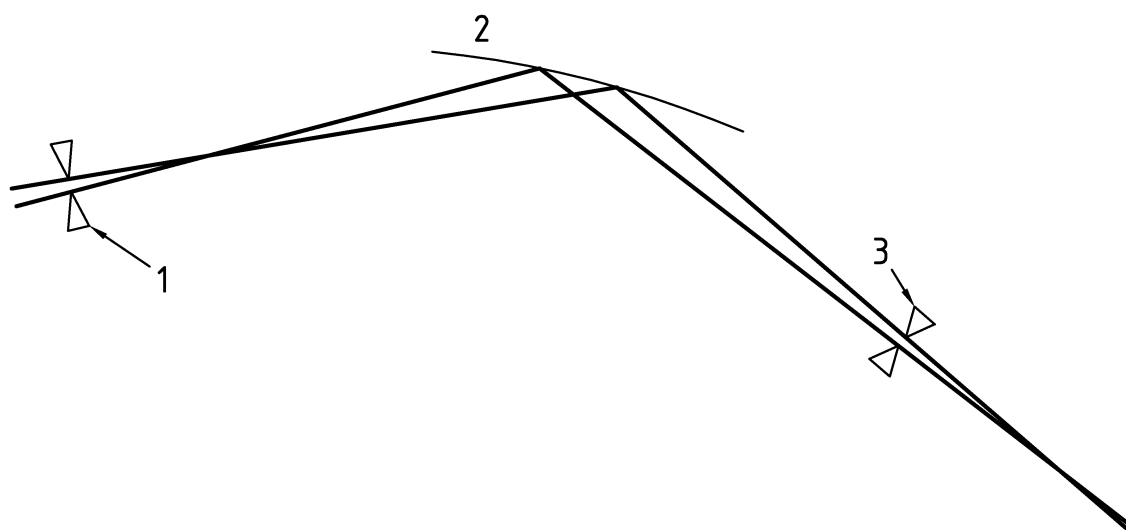
These devices can be used in the incident and/or diffracted beams (see Figures 1 and 2).



Key

- 1 Focus of X-ray tube
- 2 Monochromator
- 3 Apparent source (on the goniometer circle)

Figure 1 - Positioning the monochromator on the incident beam



Key

- 1 Receiving slit
- 2 Monochromator
- 3 Detector slit

Figure 2 - Positioning the monochromator on the diffraction beam

4.3.2.4 Electronic filters

Electronic filtering uses photon-counting detectors (see 4.4) and is often referred to as pulse height discrimination or “discrimination” or “energy discrimination”. It electronically selects the pulses arising from the photons of the radiation chosen for the experiment.

An effect similar to partial monochromatisation can be achieved by the use of a solid-state detector, e.g. Peltier-cooled. The relative energy resolution of these systems is of the same order as that of a mosaic crystal monochromator (about one part per 100), i.e. it is sufficient to eliminate the K_{β} component and most of the continuous background, but not to separate the $K_{\alpha 1,2}$ doublet. Detectors of this type typically have high counting efficiencies but are susceptible to dead time effects (prEN 1330-11). Such effects result in a non-linear response above about 10^4 counts per second, including all wavelengths registered by the detector, whether or not used for the measurement. Care shall therefore be taken to limit the maximum count rate observed to a level at which the detector function properly. If necessary an appropriate dead time correction shall be applied.

4.3.2.5 Multilayer mirrors

Multilayer mirrors usually consist of alternating thin layers of two elements (e.g. W and C) deposited onto an appropriately curved substrate. The layers have a thickness of the order of a few times the wavelength of the X-radiation used [3].

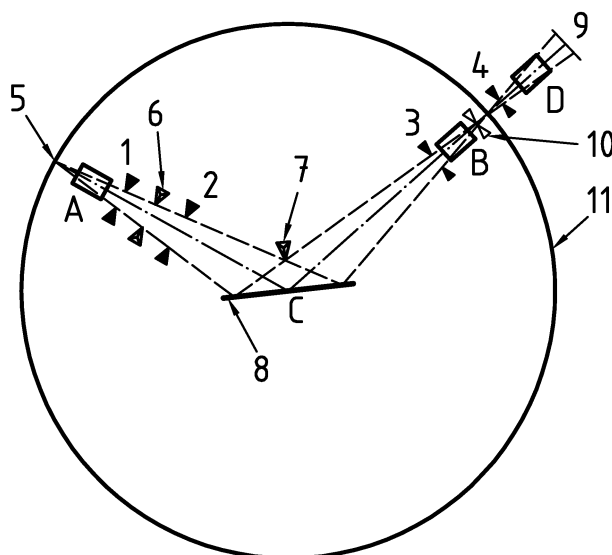
Multilayer mirrors for X-radiation are primarily used to reconfigure a divergent beam into an intense parallel or convergent beam (see 4.3.3.3.4). The mirror unit also acts as a monochromator by suppressing the K_{β} and polychromatic radiation from the source. The thickness of the layers deposited on the surface varies along the length of the mirror in such a way that Bragg's law is satisfied over a range of beam divergence angles and the corresponding incidence angles at the mirror. These systems reach a reflectivity close to 100 % and thereby provide high intensity beams. A common configuration uses a parabolic graded mirror to transform the divergent incident radiation from the source into an intense nearly parallel beam. Elliptic graded mirrors may be used to transform the divergent radiation from the source into a convergent (focused) beam. For special applications several mirrors can be combined in the incident and/or diffracted beams.

4.3.3 Beam dimensions and geometry

4.3.3.1 General

The geometric dimensions of the incident and diffracted X-ray beams can be controlled using collimators and slits. The geometric dimensions may also be altered significantly by crystal monochromators and graded multilayer mirrors.

The items described below define the beam dimensions and geometry and are illustrated schematically in Figure 3.



Key

- 1, 2 Possible position of divergence anti-scatter slit
- 3, 4 Possible position of receiving anti-scatter slit
- 5 Source
- 6 Divergence slit
- 7 Knife edge
- 8 Specimen surface
- 9 Detector window
- 10 Receiving slit
- 11 Goniometer circle
- A, B, D Possible position of Soller slit
- C Centre of specimen surface and of the goniometer circle

Figure 3 — Schematic arrangement of the devices for control of beam dimension and geometry with respect to the centre of the goniometer circle (C)

4.3.3.2 Slits

4.3.3.2.1 General

Different types of slit can be placed in the incident and diffracted beam paths, usually to limit their equatorial and axial (see 4.5.1) divergence. Slits can also serve to reduce parasitic scattering, to define the radius of the focusing circle and to control the angular resolution. They are often made of a metal with high X-ray attenuation. Orthogonal slit pairs are used for some measurements (e.g. for crystallographic texture measurements) to control the equatorial and axial beam divergence. In 4.3.3.2.2 to 4.3.3.2.7 only slits will be dealt with, because in XRPD slits are far more widely used than other apertures.

A useful quantitative geometric treatment of slits for Bragg-Brentano diffractometers is described in [4].

4.3.3.2.2 Variable aperture slits

Mechanically or computer controlled slits can be applied as variable divergence, antiscatter and receiving slits.

A variable aperture slit is most often used in diffractometers with Bragg-Brentano geometry to maintain a constant irradiated surface area of the specimen while the angle between the incident beam and the specimen surface is varied. As a consequence, the irradiated specimen volume varies during the 2θ scan.

A matched variable anti-scatter slit may be used in conjunction with a variable divergence slit. This can be particularly effective in minimising parasitic scattering at low 2θ angles.

4.3.3.2.3 Divergence slits

Divergence slits between the X-ray tube and the specimen. They limit the equatorial divergence of the incident beam.

4.3.3.2.4 Antiscatter slits

Antiscatter slits reduce parasitic scattering caused by the interaction of the incident and diffracted beams with the air, the specimen stage and the components of the optical system. They are placed in the path of the X-ray beams directly before or after the specimen or after the receiving slit. They may be used singly or in combination. If properly aligned they do not restrict the incident or diffracted beams and they do not change the diffraction geometry.

4.3.3.2.5 Knife edge

The so-called "knife edge" can be used for flat specimens. It is a flat blade or wedge with a straight edge positioned close to the specimen surface, parallel to the specimen surface and the goniometer axis. A knife edge reduces the parasitic scattering by limiting the irradiated area of the specimen. It has the same function as the divergence and the antiscatter slits. The position of the knife edge can be stationary or variable, and it can be mechanically or computer controlled.

4.3.3.2.6 Receiving slits

A receiving slit serves to select the radiation seen by the detector at the angular position sampled. It is placed in the diffracted beam, in front of the detector or in front of the diffracted beam monochromator, if present.

For Bragg-Brentano geometry, the receiving slit shall be placed on the focusing circle (see 4.5.1). Its size is an important factor in determining the angular resolution. It is sometimes called a "focusing slit", or "resolution slit".

4.3.3.2.7 Detector slits

A detector slit selects the radiation diffracted by the specimen that is allowed to enter the detector. It prevents parasitic radiation from entering the detector, e.g. radiation scattered by components of the instrument, or K_{β} radiation when the monochromator is tuned to K_{α} radiation. The receiving slit often serves as detector slit.

If a monochromator is used in the diffracted beam, a detector slit can be placed in the diffracted beam between the monochromator and the detector and is often incorporated in the monochromator housing. For (para)focusing monochromators it is usually positioned at the focusing point of the radiation diffracted by the monochromator.

4.3.3.3 Collimators and related components

4.3.3.3.1 General

A collimator is a device intended to render the X-ray beam more nearly parallel, accomplishing this by restricting the beam dimensions and / or the divergence of the beam. It should be constructed in such a way that the stray radiation produced from the slits or diaphragms used for this task is excluded as completely as possible from the diffraction pattern of the specimen registered by the detector. The design of collimators may differ considerably and depends on the diffractometer configuration and the diffraction geometry used. They are often made of a metal with high X-ray attenuation. Simple slits, crystal monochromators and graded multi-layer mirrors are normally not regarded to be collimators, although some of their functions may be similar.

4.3.3.3.2 Parallel plate collimators (Soller slits)

A parallel plate collimator (or set of Soller slits) comprises an array of parallel thin plates (foils) that limit the beam divergence, in the direction perpendicular to the foils. They can be used in the incident and/or diffracted beams and they can be used to limit the divergence in the axial and/or equatorial direction.

4.3.3.3.3 Tube collimators

A tube collimator is a tube with diaphragms or sets of slits at each end to control the dimensions and the divergence of the beam.

4.3.3.3.4 Capillary optics

Capillary optics are formed from hollow optical fibres that guide the radiation by total reflection on their inner walls. Placed in the incident beam, they typically limit its divergence to about 0,3 degrees and suppress short wavelength radiation while increasing the intensity at the specimen. A single capillary produces a beam with a very small cross-section. An assembly made from a large number of parallel fibres bundled together produces a large cross-section beam with low divergence and reduced short wavelength radiation [5].

Micromirrors are monocapillaries with a shaped inner surface, normally parabolic and gold coated. The curvature changes with position along the mirror so that a convergent (focused) X-ray beam is formed by single bounce total reflection (see 4.3.2.4).

4.4 Detectors

4.4.1 Types of detector

Detectors used in XRPD may be very different in type, size, shape (curvature) and in the physical principles by which they function. They include photographic films, gas ionisation counters (Geiger-Müller and proportional), solid-state detectors (scintillators or semiconductors), fluorescent screens and image plates. Detectors can be categorised by considering:

a) physical principle of radiation detection:

- integrating detectors: photographic films, image plates and charge coupled devices (CCD). These detectors accumulate X-rays over a time interval and with intrinsic spatial resolution. They have a limited dynamic range (total number of X-rays per angular interval) and low energy resolution. Electronic readout of data from these detectors is required for reproducible measurements;
- photon counting detectors: Geiger-Müller counters, proportional counters, scintillation counters and semiconductor detectors. They have, in principle, an unlimited dynamic range, when counts are summed into a computer memory. However, they have a limited linear response range for count rates;

b) position sensitivity of the detector: