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Neporuġitveno preskuġanje ï Rentgenska difraktometrija na polikristaliniļ nih in amorfnih materialih ï 1. del: Sploġna naļ ela

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

Zerstörungsfreie Prüfung - Röntgendiffraktometrie von polykristallinen und amorphen Materialien - Teil 1: Allgemeine Grundlagen

Essais non destructifs - Diffraction des rayons X appliquée aux matériaux polycristallins et amorphes - Partie 1: Principes généraux

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This European Standard was approved by CEN on 14 November 2002.

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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.

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Foreword

This document (EN 13925-1:2003) 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of an identical text or by endorsement, at the latest by September 2003, and conflicting national standards shall be withdrawn at the latest by September 2003.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 Part 1: General Principles;
- EN 13925-2 Part 2: Procedures;
- prEN 13925-3 Part 3: Instruments;
- prEN 13925-4 Part 4: 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.

Annex A is 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 micro-strain, 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 and 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 most detail.

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 general principles of X-ray diffraction from polycrystalline and amorphous materials. This materials testing method has traditionally been referred to as “X-ray Powder Diffraction (XRPD)”, and is now applied to powders, bulk materials, thin film, and others. As the method can be used for various types of materials and to obtain a large variety of information, this standard reviews a large number of types of analysis but remains non-exhaustive.

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

3 Terms and definitions

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

3.1
powder
large number of crystallites and/or particles (i.e. grains, agglomerates or aggregates; crystalline or non-crystalline) irrespective of any adhesion between them

3.2
block
self-adhering single piece of interconnected particles or crystallites

3.3
specimen
portion of the sample in the specific form used in the diffraction instrument for a given data collection process

3.4
phase
portion of a physical system sharing a common molecular and inter-molecular structure irrespective of any subdivision by size distribution or shape

NOTE The term 'phase' is used as a short form for a 'crystallographic phase' or a 'thermodynamic phase' throughout this standard, unless explicitly stated otherwise.

3.5
diffraction line
region of the diffraction pattern containing an intensity maximum and corresponding to diffraction from a set of lattice planes

NOTE Often interchangeably referred to as a ‘peak’, ‘reflection’ or ‘Bragg reflection'.

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

4 General principles of X-ray powder diffraction (XRPD)

X-ray diffraction results from the interaction between X-rays and electrons of atoms. Usually, i.e. in the so called kinematic description of diffraction, atoms are conceived as point scatterers. Depending on the atomic arrangement, interferences result between the scattered rays. Interferences are constructive when the path difference between two diffracted rays differ by an integral number of wavelengths. This selective condition is described by the Bragg equation, also called Bragg's law:

$$2d_{\mathrm{hkl}}\cdot\sin\theta_{\mathrm{hkl}} = n\cdot\lambda \qquad (1)$$

where

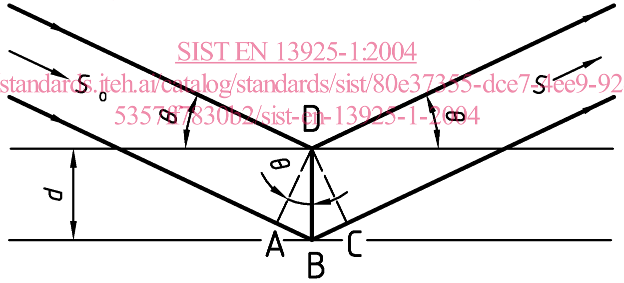
hkl is a set of lattice planes identified by the Miller indices h,k,l,

d_{hkl} is the distance between successive crystal lattice planes (also called the "d-spacing")

λ is the wavelength of the X-rays used and is of the order of magnitude of the d-spacing. θ_{hkl} is the angle between the incident beam and the hkl lattice planes and half of the so called "diffraction angle", *i.e.* the angle between the directions of the incident beam and the diffracted beam (see Figure 1).

n is the diffraction order (see hereafter):

1) the equation (1) (Bragg equation), is satisfied if the path difference (AB+BC) between the rays scattered from successive lattice planes of the same set is an exact multiple of λ (see Figure 1).



Key

d Separation between two successive lattice planes.

θ Angle between the incident beam and the lattice planes separated by a distance of d.

A and C Points on the incident beam (So) and the diffracted beam (S).

B and D represent two of the point scatterers, one in each lattice plane.

Figure 1 — Diffraction of X-rays from two successive lattice planes

2) the diffraction of n^{th} order from lattice plane hkl is equivalent to a diffraction of first order from the set of lattice planes nh, nk, nl. For most purposes in XRPD, n is taken as unity and equation (1) is more commonly used in the form:

$$2d_{\mathrm{hkl}}\cdot\sin\theta_{\mathrm{hkl}} = \lambda \qquad (2)$$

where the symbols are as in equation (1).

In XRPD the specimen is ideally polycrystalline with crystallites in all orientations, enabling diffraction to be observed according to equation (1). The positions of the diffraction lines are characteristic of the crystal lattice, their intensities depend on the crystallographic unit cell content, and the line profiles on the perfection and extent of the crystal lattice.

Under these conditions the diffraction line has a finite distribution of intensity arising from atomic arrangement, thermal motion and structural imperfections as well as from instrument characteristics. This intensity distribution is described as the convolution of a function combining the contributions of the various instrument characteristics with a function describing the intrinsic specimen contributions to broadening.

To determine the diffraction pattern of a specimen, two experimental methods can be used alternatively:

- the “angular dispersive technique” where the X-rays are monochromatic and the measurement is made by scanning the diffraction angle,
- the “energy dispersive technique“ where the X-rays are polychromatic and the energy or wavelength of the X-rays photons is measured at a fixed diffraction angle.

A series of references of general values to X-ray diffraction and Crystallography are used in this standard (see Bibliography [1, 2, 3, 4, 5, 6, 7]).

5 Meaning of the word “powder” in terms of X-ray diffraction

The term “powder”, as used in XRPD, does not strictly correspond to the usual sense of the word in common language. In X-ray powder diffraction the specimen can be a ”solid substance divided into very small particles”, but it can also be a solid block for example of metal, ceramic, polymer, glass or a thin film or even a liquid. The reason for this is that the important parameters for defining the concept of a powder for a diffraction experiment are the number and size of the individual crystallites that form the specimen and not their degree of accretion.

An ‘ideal’ powder for a diffraction experiment consists of a large number of small, randomly oriented crystallites (coherently diffracting crystalline domains). If this number is sufficiently large, there are always enough crystallites in any diffracting orientation to give reproducible diffraction patterns. To obtain a reproducible measurement of the intensity of diffracted X-rays, the crystallite size has to be sufficiently small. This size depends on the volume of the specimen from which diffracted X-rays are detected and hence on the X-ray absorption, specimen shape and diffraction geometry (see EN 13925-2:2003, 4.3.2) [4 (p.365ff)].

6 Characteristics of powder diffraction line profiles

The main characteristics of diffraction line profiles are position, maximum intensity, area and shape (characterised *e.g.* by width, asymmetry or by analytical function, empirical representation). Diffraction patterns are often interpreted quantitatively to various levels of approximation using:

a) line position: often taken as the 2θ location of the maximum of the diffraction line, but can be defined by various statistical and convenience criteria;

b) range of line profile: 2θ region where the intensity is assumed to pertain to the diffraction effect;

c) peak height: intensity above the background at the 2θ position of the maximum line intensity (I_{max});

d) integrated intensity: area of the diffraction line profile above the background (I_{int});

e) line width: typically Full Width at Half Maximum (FWHM, the line breadth at half of the peak height) or integral breadth (the width of the rectangle having the same area and height as the observed line profile, $\beta = I_{\text{int}}/I_{\text{max}}$), but can be defined by various statistical and convenience criteria;