Universität Siegen

Naturwissenschaftlich-Technische Fakultät

Manual

X-ray structural analysis of cubic crystal systems

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Basics of XRD
for cubic crystal systems

The X-ray Diffraction (XRD) technique is the most widely used non-destructive technique, specialising in the measurement of surfaces, interfaces as well bulk materials. The XRD measures structures with different orientations and the material's inter-atomic spacing.

Typically X-rays have wavelengths of the order of a few angstroms (Å), which is the same order of magnitude as the typical inter-atomic/inter-planar distances in crystalline solids. X-rays scattered from a crystalline solid can constructively interfere producing a diffracted beam. The angles at which the maximum diffracted intensities occur are measured. From these angles it is possible to obtain the interplanar spacing, *d*, of the diffraction planes using Bragg's law (see Figure 1).

If X-rays hit a parallel lattice plane family with the interplanar distance under the glancing angle, the radiation will be reflected in a constructive manner provided that the so-called Bragg condition (1) is fulfilled:

$$2d\sin(\theta) = n\lambda \qquad (1)$$

where
d – interplanar distance,
θ – Bragg angle,
n – diffraction order,
λ – wavelengths of X-rays.

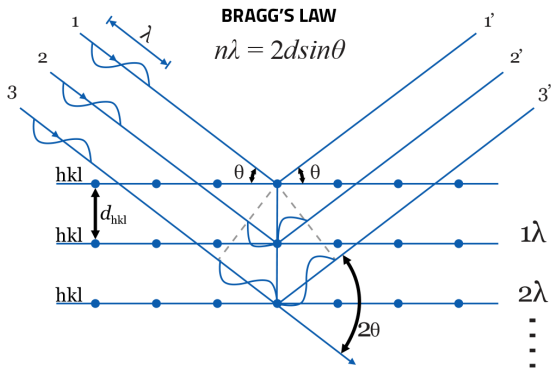

Figure 1. Conditions for diffraction and derivation of Bragg's law.
[source: VEQTER Ltd., UK]

The Miller indices ($hk\ell$) are used to specify the various planes and directions in a crystal. They basically indicate the points of intersection of an imaginary section through the three-dimensional unit cell of the crystal. A unit-cell is the smallest building block of a crystal and is representative unit of the repetitive motifs in the crystal structure.

In a cubic crystal lattice all of the sides of this cell are of the same length and all basis vectors are orthogonal to each other. The side length of such a cell is referred to as the lattice constant a. There are three main varieties of cubic crystals, namely: the primitive or simple cubic (sc), the body-centered cubic (bcc) and the face-centered cubic (fcc).

The fcc lattice is shown in Figures 2 for the case of NaCl monocrystals. In depicted case, an Na+ ion has the coordinates (0,0,0) and a Cl- ion has the coordinates (1/2, 1/2, 1/2).

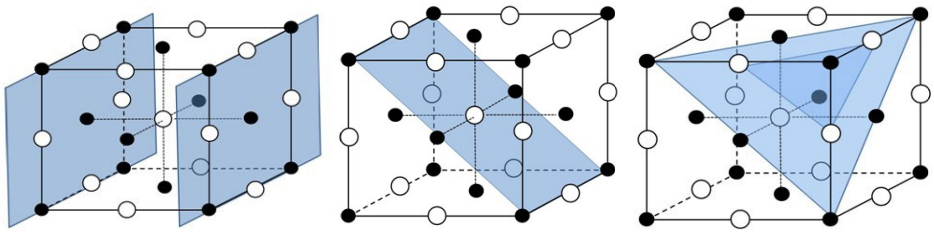

Figure 2. NaCl crystal with a drawn-in (100) lattice planes – left, (110) lattice planes – middle, (111) and (222) lattice planes – right. [source: PHYWE]

Generally, for a cubic crystal with the lattice constant a, the lattice planes that are characterised by the Miller indices ($hk\ell$) have the following interplanar spacing d:

$$d = \frac{a}{\sqrt{h^2+k^2+l^2}} \quad (2)$$

The relative intensity of the reflected radiation is determined by the scattering power and position of the individual atoms in the unit cell of the crystal. It is described by the so-called structure factor *F(hkl)*:

$$F(hkl) = \sum_n f_n e^{-2\pi i(hu_n+kv_n+lw_n)} \quad (3)$$

where f_n is the atomic form factor (alternatively atomic scattering factor), and u_n, v_n, w_n are the coordinates of the n^{th} atom in the unit cell.

The total scattered intensity I is:

$$I = |F(hkl)|^2 \qquad (4)$$

From equation (3) it follows that the structure factor F is strongly dependent on the *h, k, l* triplet. The diffraction pattern of observed for primitive cubic systems is different from *bcc* and *fcc* ones. This difference arises because the centring leads to destructive interference for some reflections and the missing reflections are known as forbidden reflections.

Tasks

Task 1

Explain how to define the Miller indices ($hk\ell$). Draw the *(222)* and *(020)* lattice planes in a simple cubic system.

Task 2

Explain a basic mechanism of characteristic X-rays radiation. Calculate the wavelength of Cu K_α radiation.

Task 3

Describe main differences between *sc*, *bcc* and *fcc* systems, which reflections are forbidden for these systems?

Task 4

For the given crystals

a) Potassium bromide (KBr)

b) Lithium fluoride (LiF)

using PHYWE XR 4.0 expert unit measure diffraction spectra as a function of the Bragg angle. Explain how to define the Miller indices ($hk\ell$) and assign the Miller indices for the measured profiles.

Task 5

For the measured profiles in task 1 for the KBr and LiF crystals determine the lattice constants and calculate the interplanar distances.

Literature:

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