High-resolution synchrotron powder diffraction with the use of scanning 2D detector

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Simultaneous recording of diffraction patterns in a large solid angle with the subsequent conversion of a two-dimensional histogram into a one-dimensional intensity – diffraction angle dependence [I (2θ)] is obviously a highly efficient data collection method for polycrystalline samples, the diffraction pattern of which is axially symmetric. This approach provides a high measurement rate with the required statistical accuracy. Shooting time is smaller by orders of magnitude compared to a point or linear detector. The negative effect of graininess and preferential orientation (texture) on data quality is reduced. However, due to the limited size of two-dimensional detectors, the resulting angular range is very limited and insufficient to obtain accurate structural information about the studied objects. In this regard, the principle of a scanning two-dimensional detector was used at the X-ray structural analysis beamline (XSA) mounted on a beam from a bending magnet of Kurchatov Synchrotron Radiation Source. The optical scheme is standard and includes a monochromator with a sagittal bend of the second crystal to focus the beam in the horizontal plane to obtain maximum intensity values [1].

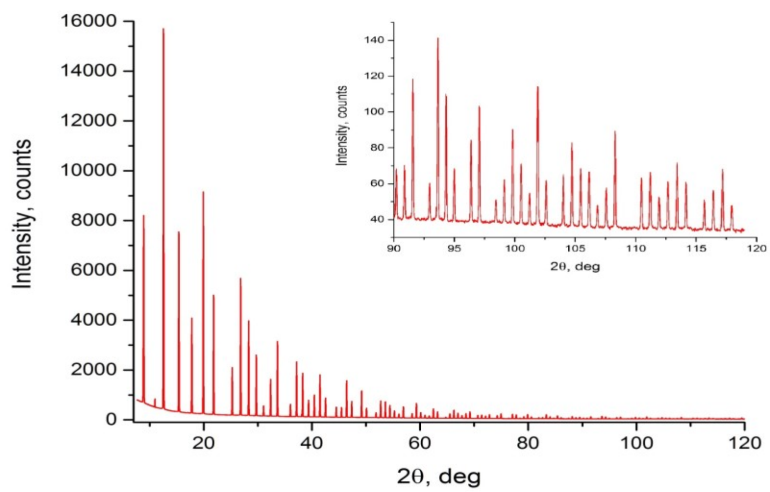

Figure 1. Diffraction pattern of LaB_6 at XSA beamline

The goniometer provides rotation of the test sample (placed in a special cryoloop or thin-walled capillary) around the horizontal axis φ, to ensure averaging of diffraction patterns over the orientations of the sample, as well as rotation of the detector around the 2θ axis, which allows high quality data to be obtained up to large values of sinθ / λ. The use of such a scheme made it possible to obtain the following parameters of the diffraction experiment:

- an angular range of up to 140º in 2θ ($q = 14.8$ Å^{-1})
- instrumental contribution to the peak broadening from 0.039º
- angular accuracy $\Delta 2\theta = 0.001$ °,
- the accuracy of determining the intensities of the Bragg peaks of the standard – 0.5%,
- the range of recorded intensities of the Bragg peaks $I_{max} / I_{min} = 10^5$.

[1] Svetogorov R.D., Dorovatovskii P.V., Lazarenko V.A. (2020) Cryst. Res. Tech. *in press*

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