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Method of Correction of Weak Spatial Inhomogeneities of a Flat-Panel X-Ray CCD Detector

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In the matrix CCD detectors which are used in X-ray single-crystal diffractometers, distortions associated with the heterogeneity of their surface may appear. The presence of such heterogeneity leads to distortion of diffraction data. In this paper, a simple and effective method for compensating of weak distortions of diffraction images caused by spatial heterogeneity of the detector surface is considered. The method is based on the use of reference images taken on calibration powders and consists of creating a special correction mask. It is shown that the described approach allows for a significant improvement in the accuracy of data collection.

Keywords: CCD detector, X-ray diffractometer, CCD detector distortion correction.

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Diffraction studies of functional materials under external influences (electric and magnetic fields, mechanical loads, etc.) applied in regimes close to those found in practical application of devices based on these materials („operando“ experiments) are becoming increasingly important at present. Notably, experiments in the „cinema“ mode, wherein sequential frames from a position-sensitive detector (CCD) are recorded without moving the diffractometer elements, are extremely informative. Detailed data on the efficiency and resolution of each detector element are crucial in this case. A method for obtaining a map of distortions of a two-dimensional CCD (charge-coupled device) detector and introducing corrections into the experimentally measured diffraction pattern is discussed below.

A detector is the key element of modern diffractometers. In the past decades, the design of X-ray detectors has evolved from simple photographic films to high-tech flat-panel multi-element devices. Two-dimensional area detectors hold a special place among modern X-ray detectors. They feature an array of detecting elements (pixels) and may be regarded as the most advanced, since they allow one to obtain directly a distribution of scattering intensity with a wide range of angles. The CCD technology is the one used most often in the design of commercial two-dimensional area detectors. Such detectors have a number of important advantages, including high data acquisition speed, low noise, and high sensitivity. They also have a wide range of applications that includes protein crystallography, single-crystal diffraction, diffuse scattering, and the study of grain orientation and deformation in polycrystals [1–3]. However, the use of CCD detectors is also fraught with certain technical difficulties related to their weak spatial homogeneity and the need for calibration and subsequent data processing [4]. Some CCD detectors also have a major drawback in convexity of their surface, which translates into spatial distortions and an uneven response [5]. One needs to specify and introduce a number of corrections in order to achieve high geometric and photometric accuracy of such a detection system [6]. It is believed that after the introduction of appropriate corrections, the detector surface may be treated as an ideal plane. However, even with all the recommended corrections applied, the device surface may still deviate from an ideal plane. In the present study, we report on a simple technique for factoring in the weak parasitic deformations of flat CCD detectors that may be used to compensate for their surface imperfections and obtain high-quality X-ray images.

Let us list the main technical characteristics of the diffractometer used:

— „Supernova“ KM4CCD diffractometer, Agilent Technologies (Oxford Diffraction);

— anode material: microfocus radiation source (MoK_α);

— reflected X-ray detector: two-dimensional high-speed CCD detector (2048×2048);

— calibration sample: LaB_6 powder.

The proposed method for factoring in the heterogeneity of the detector surface consists in constructing a calibration mask (a matrix of small shifts of planar detector pixels from their nominal position) and includes several stages. At the first stage, a series of measurements for powders are carried out using a calibration sample; the resulting images are used to find the approximate center of diffraction rings. At the second stage, a two-dimensional intensity distribution map is converted into a new polar coordinate system with equal intervals in azimuth; the center found earlier is set as its origin. The third stage involves the determination of exact positions of centers of the peaks lying along the rings via approximation of their profiles with a Gaussian

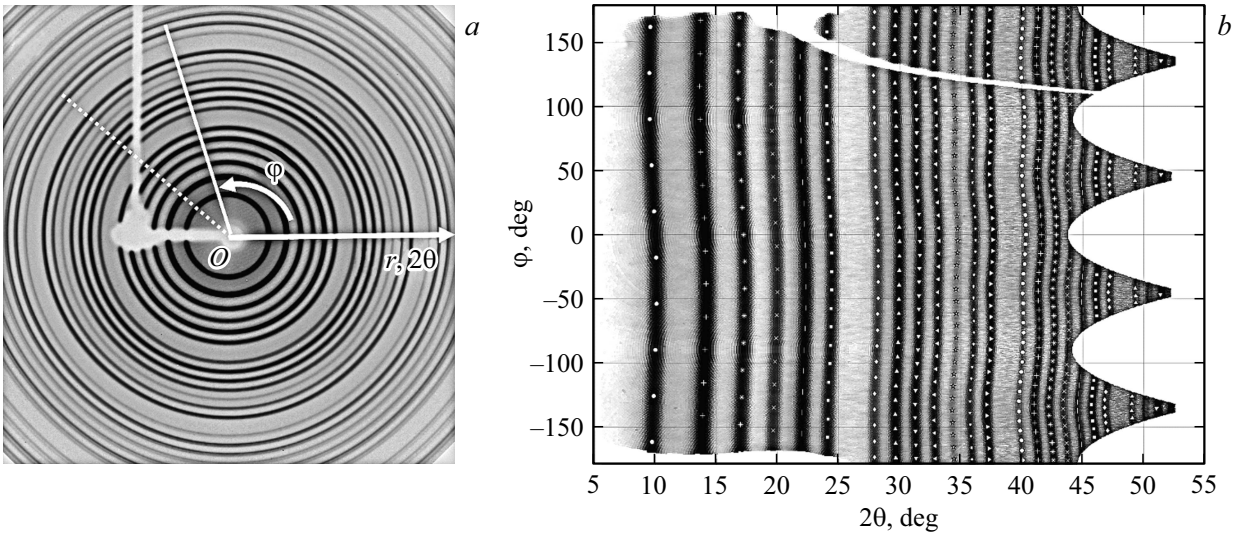

Figure 1. *a* — Illustration of the conversion of diffraction images from the detector coordinate system (x, y) to the polar one $(2\theta, \varphi)$. *b* — Scattering map in $2\theta-\varphi$ coordinates. The non-linearity of intensity isocurves caused by the detector surface heterogeneity is seen clearly. Dots mark the positions of centers of narrow angular segments of diffraction rings found during the approximation.

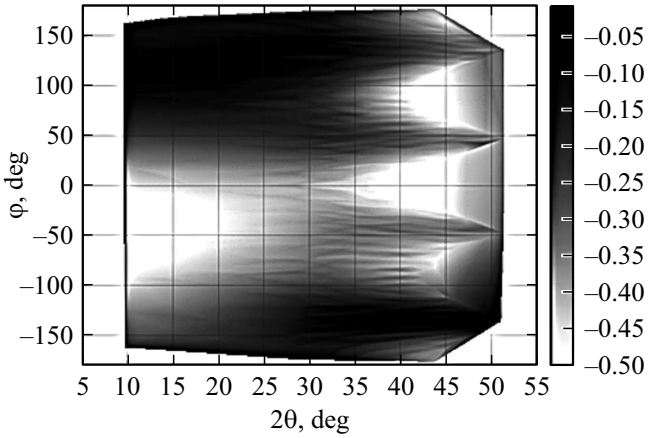

Figure 2. Two-dimensional correction mask of pixel shifts in $2\theta-\varphi$ coordinates. The shift values marked on the gradient scale indicate how much the pixel position deviates from its nominal position along the 2θ beam. The map for a perfectly flat detector should be uniform in color.

in polar coordinates. The difference between the detected and expected positions of each peak is then calculated. After this, the calculated peak shifts are converted into a Cartesian coordinate system and interpolated to the entire detector surface. The constructed matrix may be used to compensate for the unevenness of the detector surface in subsequent measurements.

In powder diffraction, reflected beams form a straight circular diffraction cone with its apex at the sample. The image produced by beams incident on a perfectly flat two-dimensional detector is a conical section. In the general case, a flat 2D detector positioned orthogonally to an incident X-ray beam has a slight tilt (within fractions of a degree). This is attributable to the fact that perfect orthogonality is hard to achieve in practice. Thus, a diffraction ring obtained on a perfectly flat planar 2D detector is actually an ellipse. It should be noted that the position of the incident beam projected onto the detector (the magnitude of unreduced wave vector $Q = 0$) coincides neither with the center of the ellipse nor with one of its foci, but is located between them [7,8]. An approximation of diffraction rings with the smallest radius was used to determine the position of the incident beam, since the curvature of the detector surface has the least effect on them.

With the approximate center determined, the intensity distribution is converted from the detector's own Cartesian coordinate system (x, y), where the coordinates of each pixel correspond to the indices containing its row and column, into polar coordinates $(2\theta, \varphi)$. Scattering angle 2θ is taken as the radial coordinate. Polar angle φ is the angle of rotation of the pixel in the image about the primary beam. The origin of coordinates (pole) coincides with the previously found position $Q = 0$. The polar axis is plotted parallel to one of the geometric axes of the detector (Fig. 1, *a*).

A set of 2θ beams originating from pole O and spaced equally in azimuth φ is isolated in polar coordinates. The radial coordinate interval is set equal to the width of an individual detector pixel (in the case where diffraction rings are so wide as to cover several pixels at once). In order to minimize the loss of coordinate accuracy at this stage, the resolution of the original image is increased several-fold by linear interpolation.

Following the conversion of the scattering map into polar coordinates, diffraction rings are divided into extremely small angular sections (arcs), and the positions of their

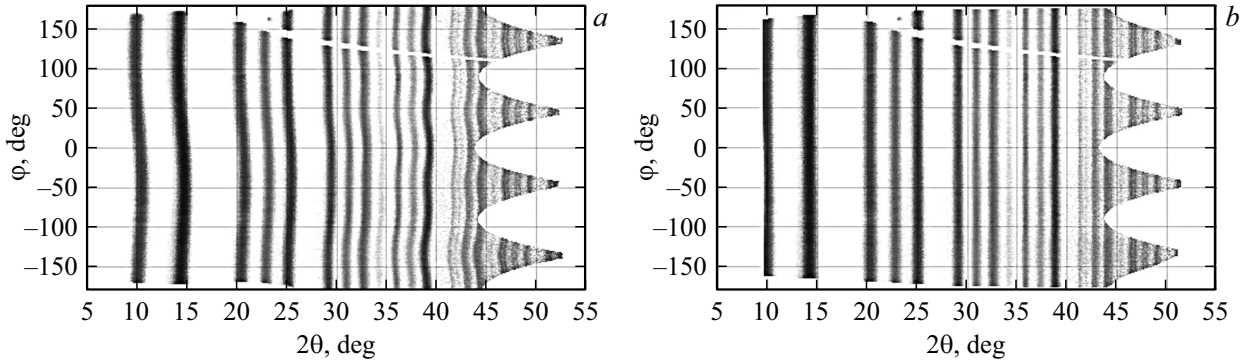

Figure 3. Two-dimensional maps for scattering off a KNN polycrystal in $2\theta-\varphi$ coordinates. *a* — Before the application of the shift mask; *b* — after its application. The detector is in a nominally perpendicular position with respect to the primary beam.

centers are determined. In order to find the coordinates of each angular element of the ring in the $2\theta-\varphi$ space, one needs to separate its region of visibility from the neighboring diffraction rings. This is done by summing the $2\theta-\varphi$ map along φ within a wide angular range of several degrees. A complete one-dimensional powder diffraction pattern corresponding to each angular sector and providing a plot of the dependence of average intensity on 2θ is then constructed. It is used to obtain the positions of local maxima (these will be averaged within a φ sector) and local minima corresponding to the boundaries of the field of observation of individual peaks. This method works well for both continuous and patchy rings and remains efficient in experiments with noisy data. It is also the preferred choice in cases where adjacent rings are hard to separate.

At the previous stage, approximate centers and boundaries of individual regions containing a single peak were determined in the $2\theta-\varphi$ coordinate system. It is now needed to determine the exact locations of peaks along each of the 2θ beams. Precise 2θ and φ coordinates may be assigned to all peaks along all beams by fitting their profiles with a Gaussian function in each found region. The center of the obtained Gaussian (Fig. 1, *b*) is the exact 2θ position of a peak.

The expected 2θ position of each peak is then calculated based on the wavelength, lattice parameters, and the distance to the detector. The difference between the experimentally observed and expected peak positions is the sought-for shift. A two-dimensional shift matrix (Fig. 2) is constructed based on the set of shift values for all peaks. The calculated peak shifts are converted into a Cartesian coordinate system and interpolated to the entire detector surface.

In further experiments with the diffractometer, one needs just to apply the pre-compiled shift mask to diffraction images to compensate for the detector surface heterogeneity. The obtained mask may be used to process diffraction maps measured with the same instrumental parameters as those set for the calibration sample. A unique mask corresponds to each detector position.. Figure 3 shows diffraction images of the $K_{0.5}Na_{0.5}NbO_3$ (KNN) test sample in the polar coordinate system without the application of the shift mask (*a*) and with the mask prepared in advance using the LaB_6 calibration sample (*b*).

Thus, a method for correcting diffraction images obtained with a two-dimensional position-sensitive detector was developed. The results provided by existing approaches were found to be unsatisfactory. The proposed method is easy to implement and is based on the use of a diffraction pattern of a reference sample with subsequent conversion of the obtained pattern into a polar coordinate system. Preliminary identification and determination of the exact position of peaks are performed next. A two-dimensional correction matrix is formed based on a complete map of calculated positions. The constructed correction mask is used in subsequent measurements to take into account slight distortions of diffraction images induced by spatial heterogeneity of the detector surface. The discussed method allows for quick and efficient compensation of weak spatial heterogeneities of the detector surface. A demonstration experiment revealed that calibration and correction provided a significant improvement in accuracy of diffraction images.

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Conflict of interest

The authors declare that they have no conflict of interest.

References

[1] J.R. Helliwell, Nucl. Instrum. Meth. Phys. Res., **201** (1), 153 (1982). DOI: 10.1016/0167-5087(82)90023-0
[2] A. Evans, N. Jones, D. Rugg, T. Lindley, D. Dye, J. Nucl. Mater., **424** (1-3), 123 (2012). DOI: 10.1016/j.jnucmat.2012.02.013
[3] T. Sui, G. Landini, A. Korsunsky, AIP Conf. Proc., **1394**, 113 (2011). DOI: 10.1063/1.3649941

[4] C. Ferreira, M. Françis, R. Guillén, Mater. Sci. Forum, **524-525**, 761 (2006). DOI: 10.4028/www.scientific.net/MSF.524-525.761
[5] A.P. Hammersley, K. Brown, W. Burmeister, L. Claustre, A. Gonzalez, S. McSweeney, E. Mitchell, J.-P. Moy, S.O. Svensson, A.W. Thompson, J. Synchrotron Rad., **4** (2), 67 (1997). DOI: 10.1107/S0909049596015087
[6] W.A. Paciorek, M. Meyer, G. Chapuis, J. Appl. Cryst., **32**, 11 (1999). DOI: 10.1107/S0021889898005172
[7] M.L. Hart, M. Drakopoulos, C. Reinhard, T. Connolley, J. Appl. Cryst., **46**, 1249 (2013). DOI: 10.1107/S0021889813022437
[8] A. Cervellino, C. Giannini, A. Guagliardi, M. Ladisa, J. Appl. Cryst., **41**, 701 (2008). DOI: 10.1107/S0021889808019092

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