International Tables for Crystallography (2018). Vol. H, References, pp. 147–149.

$$d = k\left\{\frac{p_{hkl}b^2 \arcsin[\cos\theta\sin(\Delta\gamma/2)]}{\mu N_s}\right\}^{1/3}, \quad (2.5.92)$$

where d is the diameter of the crystallite particles, p_{hkl} is the multiplicity of the diffracting planes, b is the size of the incident beam (*i.e.* its diameter), $\Delta\gamma$ is the γ range of the diffraction ring, μ is the linear absorption coefficient and N_s is the number of spots within $\Delta\gamma$. For transmission mode, we have

$$d = k\left\{\frac{p_{hkl}b^2 t \arcsin[\cos\theta\sin(\Delta\gamma/2)]}{N_s}\right\}^{1/3}, \quad (2.5.93)$$

where t is the sample thickness. In transmission mode with the incident beam perpendicular to the sample surface, the linear absorption coefficient affects the relative scattering intensity, but not the actual sampling volume. In other words, all the sample volume irradiated by the incident beam contributes to the diffraction. Therefore, it is reasonable to ignore the absorption effect $\exp(\mu t)$ for crystallite-size analysis as long as the sample is thin enough for transmission-mode diffraction. The effective sampling volume reaches a maximum for transmission-mode diffraction when $t = 1/\mu$.

For both reflection and transmission,

$$k = \left(\frac{3\beta}{8\pi}\right)^{1/3}, \quad (2.5.94)$$

where β is the divergence of the incident beam. Without knowing the precise instrumental broadening, k can be treated as a calibration factor determined from the 2D diffraction pattern of a known standard. Since only a limited number of spots along the diffraction ring can be resolved, it can be seen from equation (2.5.94) that a smaller X-ray beam size and low-multiplicity peak should be used if a smaller crystallite size is to be determined.

Fig. 2.5.29(*c*) shows the measurement ranges of 2θ-profile and γ-profile analysis. The 2θ-profile analysis is suitable for crystallite sizes below 100 nm (1000 Å), while γ-profile analysis is suitable for crystallite sizes from as large as tens of µm down to 100 nm with a small X-ray beam size. By increasing the effective diffraction volume by translating the sample during data collection or multiple sample integration (or integrating data from multiple samples), the measurement range can be increased up to millimetres. Multiple sample integration can deal with large crystallite sizes without recalibration. The new calibration factor is given as

$$k_n = n^{1/3}k, \quad (2.5.95)$$

where n is the number of targets that are integrated. The number of crystallites can be counted by the number of intersections of the γ profile with a threshold line. Every two intersections of the γ profile with this horizontal line represents a crystallite. In order to cancel out the effects of preferred orientation and other material and instrumental factors on the overall intensity fluctuation along the γ profile, one can use a trend line fitted to the γ profile by a second-order polynomial. It is always necessary to calibrate the system with a known standard, preferably with a comparable sample geometry and crystallite size. For reflection mode, it is critical to have a standard with a comparable linear absorption coefficient so as to have similar penetration.

References

Alexander, L. E. (1985). *X-ray Diffraction Methods in Polymer Science*. Malabar: Krieger Publishing Company.

Allahkarami, M. & Hanan, J. C. (2011). *X-ray diffraction mapping on a curved surface*. *J. Appl. Cryst.* **44**, 1211–1216.

Arndt, U. W. (1986). *X-ray position-sensitive detectors*. *J. Appl. Cryst.* **19**, 145–163.

Arndt, U. W. (1990). *Focusing optics for laboratory sources in X-ray crystallography*. *J. Appl. Cryst.* **23**, 161–168.

Bergese, P., Bontempi, E., Colombo, I. & Depero, L. E. (2001). *Micro X-ray diffraction on capillary powder samples: a novel and effective technique for overcoming preferred orientation*. *J. Appl. Cryst.* **34**, 663–665.

Bhuvanesh, N. S. P. & Reibenspies, J. H. (2003). *A novel approach to micro-sample X-ray powder diffraction using nylon loops*. *J. Appl. Cryst.* **36**, 1480–1481.

Birkholz, M. (2006). *Thin Film Analysis by X-ray Scattering*, pp. 191–195. Weinheim: Wiley-VCH.

Blanton, T. N. (1994). *X-ray diffraction orientation studies using two-dimensional detectors*. *Adv. X-ray Anal.* **37**, 367–373.

Blanton, T. N. (2003). *X-ray film as a two-dimensional detector for X-ray diffraction analysis*. *Powder Diffr.* **18**, 91–98.

Bloomer, A. C. & Arndt, U. W. (1999). *Experiences and expectations of a novel X-ray microsource with focusing mirror. I*. *Acta Cryst.* D**55**, 1672–1680.

Boesecke, P. (2007). *Reduction of two-dimensional small- and wide-angle X-ray scattering data*. *J. Appl. Cryst.* **40**, s423–s427.

Bontempi, E., Benedetti, D., Massardi, A., Zacco, A., Borgese, L. & Depero, L. E. (2008). *Laboratory two-dimensional X-ray microdiffraction technique: a support for authentication of an unknown Ghirlandaio painting*. *Appl. Phys. A*, **92**, 155–159.

Borgonovi, G. M. (1984). *Determination of residual stress from two-dimensional diffraction pattern*. *Nondestructive Methods for Material Property Determination*, edited by C. O. Ruud & R. E. Green Jr, pp. 47–57. New York: Plenum Publishing Corporation.

Bourgeois, D., Moy, J. P., Svensson, S. O. & Kvick, Å. (1994). *The point-spread function of X-ray image-intensifiers/CCD-camera and imaging-plate systems in crystallography: assessment and consequences for the dynamic range*. *J. Appl. Cryst.* **27**, 868–877.

Bruker (2000). *Percent crystallinity in polymer*. Bruker AXS Lab Report No. L86-E00005.

Bunge, H. J. (1983). *Texture Analysis in Materials Science*. London: Butterworth.

Bunge, H. J. & Klein, H. (1996). *Determination of quantitative, high-resolution pole-figures with the area detector*. *Z. Metallkd.* **87**(6), 465–475.

Campbell, J. W., Harding, M. M. & Kariuki, B. (1995). *Spatial-distortion corrections, for Laue diffraction patterns recorded on image plates, modelled using polynomial functions*. *J. Appl. Cryst.* **28**, 43–48.

Cervellino, A., Giannini, C., Guagliardi, A. & Ladisa, M. (2006). *Folding a two-dimensional powder diffraction image into a one-dimensional scan: a new procedure*. *J. Appl. Cryst.* **39**, 745–748.

Chung, F. H. & Scott, R. W. (1973). *A new approach to the determination of crystallinity of polymers by X-ray diffraction*. *J. Appl. Cryst.* **6**, 225–230.

Cullity, B. D. (1978). *Elements of X-ray Diffraction*, 2nd ed. Reading, MA: Addison-Wesley.

Derewenda, Z. & Helliwell, J. R. (1989). *Calibration tests and use of a Nicolet/Xentronics imaging proportional chamber mounted on a conventional source for protein crystallography*. *J. Appl. Cryst.* **22**, 123–137.

Dickerson, M. B., Pathak, K., Sandhage, K. H., Snyder, R. L., Balachandran, U., Ma, B., Blaugher, R. D. & Bhattacharya, R. N. (2002). *Applications of 2D detectors in x-ray analysis*. *Adv. X-ray Anal.* **45**, 338–344.

Durst, R. D., Carney, S. N., Diawara, Y. & Shuvalov, R. (2002). *Readout structure and technique for electron cloud avalanche detectors*. US Patent No. 6, 340, 819.

Eatough, M. O., Rodriguez, M. A., Blanton, T. N. & Tissot, R. G. (1997). *A comparison of detectors used for microdiffraction applications*. *Adv. X-ray Anal.* **41**, 319–326.

Ercan, A., Tate, M. W. & Gruner, S. M. (2006). *Analog pixel array detectors*. *J. Synchrotron Rad.* **13**, 110–119.

Copyright © 2018 International Union of Crystallography

European Standard (2008). *Non-destructive Testing – Test Method for Residual Stress Analysis by X-ray Diffraction*. EN15305–2008.

Flemming, R. L. (2007). *Micro X-ray diffraction (μXRD): a versatile technique for characterization of Earth and planetary materials*. *Can. J. Earth Sci.* **44**, 1333–1346.

Fuentes-Montero, L., Montero-Cabrera, M. E. & Fuentes-Cobas, L. (2011). *The software package ANAELU for X-ray diffraction analysis using two-dimensional patterns*. *J. Appl. Cryst.* **44**, 241–246.

Fujii, N. & Kozaki, S. (1993). *Highly sensitive x-ray stress measurement in small area*. *Adv. X-ray Anal.* **36**, 505–513.

Fujita, H., Tsai, D.-Y., Itoh, T., Doi, K., Morishita, J., Ueda, K. & Ohtsuka, A. (1992). *A simple method for determining the modulation transfer function in digital radiography*. *IEEE Trans. Med. Imag.* **11**, 34–39.

Gelfi, M., La Vecchia, G. M., Lecis, N. & Troglio, S. (2005). *Relationship between through-thickness residual stress of CrN-PVD coatings and fatigue nucleation sites*. *Surf. Coat. Technol.* **192**, 263–268.

Giomatartis, Y. (1998). *Development and prospects of the new gaseous detector 'Micromegas'*. *Nucl. Instrum. Methods A*, **419**, 239.

Guggenheim, S. (2005). *Simulations of Debye–Scherrer and Gandolfi patterns using a Bruker Smart Apex diffractometer system*. Bruker AXS Application Note 373.

Hammersley, A. P. (2016). *FIT2D: a multi-purpose data reduction, analysis and visualization program*. *J. Appl. Cryst.* **49**, 646–652.

Hammersley, A. P., Svensson, S. O., Hanfland, M., Fitch, A. N. & Häusermann, D. (1996). *Two-dimensional detector software: From real detector to idealised image or two-theta scan*. *High Pressure Res*. **14**, 235–248.

Hanan, J., Űstündag, E. & Almer, J. D. (2004). *A new analysis method for two-dimensional X-ray data*. *Adv. X-ray Anal.* **47**, 174–180.

Hanna, S. & Windle, A. H. (1995). *A novel polymer fibre diffractometer, based on a scanning X-ray-sensitive charge-coupled device*. *J. Appl. Cryst.* **28**, 673–689.

He, B. (1992). *X-ray Diffraction from Point-Like Imperfection*. PhD dissertation, Virginia Tech, USA.

He, B. B. (2000). *Residual stress measurement with two-dimensional diffraction*. *The 20th ASM Heat Treating Society Conference Proceedings*, Vol. 1, pp. 408–417. St Louis, USA.

He, B. B. (2003). *Introduction to two-dimensional X-ray diffraction*. *Powder Diffr.* **18**, 71–85.

He, B. B. (2004). *Microdiffraction using two-dimensional detectors*. *Powder Diffr.* **19**, 110–118.

He, B. B. (2006). *Measurement of residual stresses in thin films by two-dimensional XRD*. *Mater. Sci. Forum*, **524–525**, 613–618.

He, B. B. (2009). *Two-dimensional X-ray Diffraction*. New York: John Wiley & Sons.

He, B. B., Anzelmo, J., LaPuma, P., Preckwinkel, U. & Smith, K. (2001). *XRD rapid screening system for combinatorial chemistry*. *Adv. X-ray Anal.* **44**, 1–5.

He, T., Durst, R. D., Becker, B. L., Kaercher, J. & Wachter, G. (2011). *A large area X-ray imager with online linearization and noise suppression*. *Proc. SPIE*, **8142**, 81421Q.

He, B. B. & Preckwinkel, U. (2002). *X-ray optics for two-dimensional x-ray diffraction*. *Adv. X-ray Anal.* **45**, 332–337.

He, B., Rao, S. & Houska, C. R. (1994). *A simplified procedure for obtaining relative x-ray intensities when a texture and atomic displacements are present*. *J. Appl. Phys.* **75**, 4456–4464.

He, B. B. & Smith, K. L. (1997). *Strain and stress measurement with two-dimensional detector*. *Adv. X-ray Anal.* **41**, 501–508.

He, B. B. & Smith, K. L. (1998). *Computer simulation of diffraction stress measurement with 2D detectors*. *Proceedings of 1998 SEM Spring Conference on Experimental and Applied Mechanics*, Houston, USA.

He, B. B., Xu, K., Wang, F. & Huang, P. (2005). *Two-dimensional X-ray diffraction for structure and stress analysis*. *Mater. Sci. Forum*, **490–491**, 1–6.

Helming, K., Lyubchenko, M., He, B. & Preckwinkel, U. (2003). *A new method for texture measurements using a general area detector diffraction system*. *Powder Diffr.* **18**, 99–102.

Hinrichsen, B. (2007). *Two-dimensional X-ray powder diffraction*. PhD dissertation, University of Stuttgart, Germany.

Jabeen, S., Raza, S. M., Ahmed, M. A., Zai, M. Y. & Erlacher, K. (2011). *Two dimensional X-ray diffraction (2D-XRD) studies on olivine of U. S. A*. *J. Chem. Soc. Pak.* **33**(5), 612–618.

James, M. R. & Cohen, J. B. (1980). *The measurement of residual stresses by x-ray diffraction techniques*. *Treatise on Materials Science and Technology*, **19A**, edited by H. Herman. New York: Academic Press.

Jenkins, R. & Snyder, R. L. (1996). *Introduction to X-ray Powder Diffractometry*. New York: John Wiley & Sons.

Kämpfe, A., Kämpfe, B., Goldenbogen, S., Eigenmann, B., Macherauch, E. & Löhe, D. (1999). *X-ray stress analysis on polycrystalline materials using two-dimensional detectors*. *Adv. X-ray Anal.* **43**, 54–65.

Kasai, N. & Kakudo, M. (2005). *X-ray Diffraction by Macromolecules*, pp. 393–417. Tokyo: Kodansha/Springer.

Kerr, K. A. & Ashmore, J. P. (1974). *Systematic errors in polarization corrections for crystal-monochromatized radiation*. *Acta Cryst.* A**30**, 176–179.

Khazins, D. M., Becker, B. L., Diawara, Y., Durst, R. D., He, B. B., Medved, S. A., Sedov, V. & Thorson, T. A. (2004). *A parallel-plate resistive-anode gaseous detector for X-ray imaging*. *IEEE Trans. Nucl. Sci.* **51**, 943–947.

Klein, J., Lehmann, C. W., Schmidt, H.-W. & Maier, W. F. (1998). *Combinatorial material libraries on the microgram scale with an example of hydrothermal synthesis*. *Angew. Chem. Int. Ed.* **37**, 3369–3372.

Klug, H. P. & Alexander, L. E. (1974). *X-ray Diffraction Procedures for Polycrystalline and Amorphous Materials*. New York: John Wiley & Sons.

Korhonen, M. A., Lindroos, V. K. & Suominen, L. S. (1989). *Application of a new solid state x-ray camera to stress measurement*. *Adv. X-ray Anal.* **32**, 407–413.

Krause, K. L. & Phillips, G. N. (1992). *Experience with commercial area detectors: a 'buyer's' perspective*. *J. Appl. Cryst.* **25**, 146–154.

Kugler, W. (2003). *X-ray diffraction analysis in the forensic science: the last resort in many criminal cases*. *Adv. X-ray Anal.* **46**, 1–16.

Lewis, R. (1994). *Multiwire gas proportional counters: decrepit antiques or classic performers?* *J. Synchrotron Rad.* **1**, 43–53.

Lu, J. (1996). *Handbook of Measurement of Residual Stress*. Society for Experimental Mechanics. Lilburn: The Fairmont Press.

Lutz, G. (2006). *Silicon drift and pixel devices for X-ray imaging and spectroscopy*. *J. Synchrotron Rad.* **13**, 99–109.

Maslen, E. N. (1992). *X-ray absorption*. *International Tables for Crystallography*, Vol. C, edited by A. J. C. Wilson, pp. 520–529. Dordrecht: Kluwer Academic Publishers.

Moy, J. P., Hammersley, A. P., Svensson, S. O., Thompson, A., Brown, K., Claustre, L., Gonzalez, A. & McSweeney, S. (1996). *A novel technique for accurate intensity calibration of area X-ray detectors at almost arbitrary energy*. *J. Synchrotron Rad.* **3**, 1–5.

Mudie, S. T., Pavlov, K. M., Morgan, M. J., Hester, J. R., Tabuchi, M. & Takeda, Y. (2004). *Collection of reciprocal space maps using imaging plates at the Australian National Beamline Facility at the Photon Factory*. *J. Synchrotron Rad.* **11**, 406–413.

Murthy, N. S. & Barton, R. Jr (2000). *Polymer industry*. *Industrial Applications of X-ray Diffraction*, edited by F. H. Chung & D. K. Smith, pp. 495–509. New York: Marcel Dekker.

Ning, G. & Flemming, R. L. (2005). *Rietveld refinement of LaB_6: data from μXRD*. *J. Appl. Cryst.* **38**, 757–759.

Noyan, I. C. & Cohen, J. B. (1987). *Residual Stress*. New York: Springer-Verlag.

Paciorek, W. A., Meyer, M. & Chapuis, G. (1999). *On the geometry of a modern imaging diffractometer*. *Acta Cryst.* A**55**, 543–557.

Pecharsky, V. K. & Zavalij, P. Y. (2003). *Fundamentals of Powder Diffraction and Structure Characterization of Materials*. Boston: Kluwer Academic Publishers.

Pitschke, W., Collazo, J. A. L., Hermann, H. & Hildenbrand, V. D. (1996). *Absorption corrections of powder diffraction intensities recorded in transmission geometry*. *J. Appl. Cryst.* **29**, 561–567.

Ponchut, C. (2006). *Characterization of X-ray area detectors for synchrotron beamlines*. *J. Synchrotron Rad.* **13**, 195–203.

Pople, J. A., Keates, P. A. & Mitchell, G. R. (1997). *A two-dimensional X-ray scattering system for in-situ time-resolving studies of polymer structures subjected to controlled deformations*. *J. Synchrotron Rad.* **4**, 267–278.

Rodriguez-Navarro, A. B. (2006). *XRD2DScan: new software for polycrystalline materials characterization using two-dimensional X-ray diffraction*. *J. Appl. Cryst.* **39**, 905–909.

Ross, C. R. (1992). *Measurement of powder diffraction sample absorption coefficients using monochromated radiation and transmission geometry*. *J. Appl. Cryst.* **25**, 628–631.

Rowe, R. (2009). *New statistical calibration approach for Bruker AXS D8 Discover microdiffractometer with Hi-Star detector using GADDS*

software. *Powder Diffr.* **24**, 263–271.

Rudolf, P. R. & Landes, B. G. (1994). *Two-dimensional X-ray diffraction and scattering of microcrystalline and polymeric materials*. *Spectroscopy*, **9**(6), 22–33.

Scheidegger, S., Estermann, M. A. & Steurer, W. (2000). *Correction of specimen absorption in X-ray diffuse scattering experiments with area-detector systems*. *J. Appl. Cryst.* **33**, 35–48.

Schmidbauer, M., Schäfer, P., Besedin, S., Grigoriev, D., Köhler, R. & Hanke, M. (2008). *A novel multi-detection technique for three-dimensional reciprocal-space mapping in grazing-incidence X-ray diffraction*. *J. Synchrotron Rad.* **15**, 549–557.

Smilgies, D.-M. & Blasini, D. R. (2007). *Indexation scheme for oriented molecular thin films studied with grazing-incidence reciprocal-space mapping*. *J. Appl. Cryst.* **40**, 716–718.

Smith, K. L. & Ortega, R. B. (1993). *Use of a two-dimensional, position sensitive detector for collecting pole figures*. *Adv. X-ray Anal.* **36**, 641–647.

Sprauel, J. M. & Michaud, H. (2002). *Global X-ray method for the determination of stress profiles*. *Mater. Sci. Forum*, **404–407**, 19–24.

Stanton, M., Phillips, W. C., Li, Y. & Kalata, K. (1992). *Correcting spatial distortions and nonuniform response in area detectors*. *J. Appl. Cryst.* **25**, 549–558.

Sulyanov, S. N., Popov, A. N. & Kheiker, D. M. (1994). *Using a two-dimensional detector for X-ray powder diffractometry*. *J. Appl. Cryst.* **27**, 934–942.

Tate, M. W., Eikenberry, E. F., Barna, S. L., Wall, M. E., Lowrance, J. L. & Gruner, S. M. (1995). *A large-format high-resolution area X-ray detector based on a fiber-optically bonded charge-coupled device (CCD)*. *J. Appl. Cryst.* **28**, 196–205.

Tissot, R. G. (2003). *Microdiffraction applications utilizing a two-dimensional proportional detector*. *Powder Diffr.* **18**, 86–90.

Ungár, T. (2000). *Warren–Averbach applications*. *Industrial Applications of X-ray Diffraction*, edited by F. H. Chung & D. K. Smith, pp. 847–867. New York: Marcel Dekker.

Walter, N. M. (1971). *Residual Stress Measurement by X-ray Diffraction* – SAE J784a. Society of Automotive Engineering.

Warren, B. E. (1990). *X-ray Diffraction*. New York: Dover Publications.

Wenk, H.-R. & Grigull, S. (2003). *Synchrotron texture analysis with area detectors*. *J. Appl. Cryst.* **36**, 1040–1049.

Westbrook, E. M. (1999). *Performance characteristics needed for protein crystal diffraction X-ray detectors*. *Proc. SPIE*, **3774**, 2–16.

Wiesmann, J., Graf, J., Hoffmann, C. & Michaelsen, C. (2007). *New possibilities for x-ray diffractometry*. *Physics meets Industry*, edited by J. Gegner & F. Haider. Renningen: ExpertVerlag. ISBN 978-3-8169-2740-2.

Wilson, A. J. C. (1971). *Some further considerations in particle-size broadening*. *J. Appl. Cryst.* **4**, 440–443.

Winter, D. J. & Squires, B. A. (1995). *A new approach in performing microdiffraction analysis*. *Adv. X-ray Anal.* **38**, 551–556.

Yagi, N. & Inoue, K. (2007). *CMOS flatpanel detectors for SAXS/WAXS experiments*. *J. Appl. Cryst.* **40**, s439–s441.

Yoshioka, Y. & Ohya, S. (1992). *X-ray analysis of stress in a localized area by use of image plate*. *Adv. X-ray Anal.* **35**, 537–543.

Zuev, A. D. (2006). *Calculation of the instrumental function in X-ray powder diffraction*. *J. Appl. Cryst.* **39**, 304–314.