

2. DIFFRACTION GEOMETRY AND ITS PRACTICAL REALIZATION

has been annealed. Then a model for the diffusion is assumed, and the coefficients are calculated. Using standard techniques, researchers are unable to detect the movement of an atom through a sample of like atoms. However, using single bilayers of amorphous ^{10}B and ^{11}B , it was shown (Smith, Hamilton, Fitzsimmons, Baker, Hubbard, Nastasi, Hirvonen & Zocco, 1992) through neutron-reflectivity measurements that the diffusion of boron in boron could be measured by studying the density profile (see Figs. 2.9.7.1 and 2.9.7.2) of one isotope in the other as a function of annealing time. Also, because of the sensitivity of the technique to the interfacial density profile, it was found that standard Fickian diffusion models could not explain the measured density profiles.

2.9.7.2. Magnetic multilayers

In order to understand interlayer coupling mechanisms, it is necessary to know what the magnetic superstructure is for a given nonmagnetic spacer layer thickness and/or applied field strength. Fig. 2.9.7.3 shows the spin-dependent reflectivities for a Co/Cu (111) multilayer along with the nuclear (Nb) and magnetic (Np) scattering-density profiles deduced from the data of Schreyer, Zeidler, Morawe, Metoki, Zabel, Ankner & Majkrzak (1993). In this specific case, the in-plane ferromag-

netic Co layers are themselves coupled ferromagnetically across the nonmagnetic Cu, all at a constant angle.

2.9.7.3. Hydrogenous materials

There are a substantial number of applications of neutron reflectometry in the study of hydrogenous films and multilayers, including diblock copolymer, surfactant, Langmuir–Blodgett, self-assembled monolayer, and lipid bilayer films. Reviews of the extensive research that has already been done have been written by Russell (1990) and Penfold & Thomas (1990). Only one specific example will be given here.

Fig. 2.9.7.4 shows neutron reflectivity data and the corresponding density profile for a Langmuir–Blodgett film composed of alternating bilayers of deuterated and hydrogenated stearic acid [after Wiesler, Feigin, Majkrzak, Ankner, Berzina & Troitsky (1995)]. Also shown in Fig. 2.9.7.4 is the scattering-density profile for the same sample as seen by X-rays. It is obvious that the X-rays are more sensitive to the high-Z barium in the head group, whereas the neutrons are especially good at distinguishing the degree of mixing between adjacent hydrogenated and deuterated hydrocarbon tails. This is a good example of the complementary nature of neutron and X-ray reflectivities.

References

2.1–2.2

- Amorós, J. L., Buerger, M. J. & Amorós, M. C. (1975). *The Laue method*. New York: Academic Press.
- Andrews, S. J., Hails, J. E., Harding, M. M. & Cruickshank, D. W. J. (1987). *Acta Cryst.* **A43**, 70–73.
- Arndt, U. W. (1986). *X-ray position-sensitive detectors*. *J. Appl. Cryst.* **19**, 145–163.
- Arndt, U. W. & Willis, B. T. M. (1966). *Single crystal diffractometry*. Cambridge University Press.
- Arndt, U. W. & Wonacott, A. J. (1977). *The rotation method in crystallography*. Amsterdam: North-Holland.
- Artymiuk, P. & Phillips, D. C. (1985). *On the design of diffractometers to measure a number of reflections simultaneously*. *Methods Enzymol.* **114A**, 397–415.
- Bernal, J. D. (1927). *A universal X-ray photogoniometer*. *J. Sci. Instrum.* **4**, 273–284.
- Bijvoet, J. M., Burgers, W. G. & Hägg, G. (1969). *Early papers on diffraction of X-rays by crystals*, Vol. I. Dordrecht: Kluwer Academic Publishers.
- Bijvoet, J. M., Burgers, W. G. & Hägg, G. (1972). *Early papers on diffraction of X-rays by crystals*, Vol. II. Dordrecht: Kluwer Academic Publishers.
- Blundell, T. L. & Johnson, L. N. (1976). *Protein crystallography*. New York: Academic Press.
- Bonse, U., Materlik, G. & Schröder, W. (1976). *Perfect-crystal monochromators for synchrotron X-radiation*. *J. Appl. Cryst.* **9**, 223–230.
- Bragg, W. H. (1928). *An introduction to crystal structure analysis*. London: Bell.
- Bragg, W. L. (1949). *The crystalline state: a general survey*, pp. 30–33. London: Bell.
- Brooks, I. & Moffat, K. (1991). *Laue diffraction from protein crystals using a sealed-tube X-ray source*. *J. Appl. Cryst.* **24**, 146–148.
- Buerger, M. J. (1942). *X-ray crystallography*. New York: John Wiley.
- Buerger, M. J. (1964). *The precession method*. New York: John Wiley.
- Carr, P. D., Cruickshank, D. W. J. & Harding, M. M. (1992). *The determination of unit-cell parameters from Laue diffraction patterns using their gnomonic projections*. *J. Appl. Cryst.* **25**, 294–308.
- Cassetta, A., Deacon, A., Emmerich, C., Habash, J., Helliwell, J. R., McSweeney, S., Snell, E., Thompson, A. W. & Weisgerber, S. (1993). *The emergence of the synchrotron Laue method for rapid data collection from protein crystals*. *Proc. R. Soc. London Ser. A*, **442**, 177–192.
- Charpak, G., Demierre, C., Kahn, R., Santiard, J. C. & Sauli, F. (1977). *Some properties of spherical drift chambers*. *Nucl. Instrum. Methods*, **141**, 449.
- Coppens, P. (1992). *Synchrotron radiation crystallography*. New York: Academic Press.
- Cruickshank, D. W. J., Carr, P. D. & Harding, M. M. (1992). *Estimation of d_{\min} , λ_{\min} and λ_{\max} from the gnomonic projections of Laue patterns*. *J. Appl. Cryst.* **25**, 285–293.
- Cruickshank, D. W. J., Helliwell, J. R. & Moffat, K. (1987). *Multiplicity distribution of reflections in Laue diffraction*. *Acta Cryst.* **A43**, 656–674.
- Cruickshank, D. W. J., Helliwell, J. R. & Moffat, K. (1991). *Angular distribution of reflections in Laue diffraction*. *Acta Cryst.* **A47**, 352–373.
- Evans, H. T. & Lonsdale, K. (1959). *Diffraction geometry*. *International tables for X-ray crystallography*, Vol. II, p. 164. Birmingham: Kynoch Press.
- Friedrich, W., Knipping, P. & von Laue, M. (1912). *Interferenz-Erscheinungen bei Röntgenstrahlen*. *Sitzungsber. K. Bayer. Akad. Wiss. Muenchen*, pp. 303–322.
- Glusker, J. P. & Trueblood, K. N. (1971). *Crystal structure analysis*, pp. 35–47. Oxford University Press.
- Glusker, J. P. & Trueblood, K. N. (1985). *Crystal structure analysis*, 2nd ed., pp. 42–60. Oxford University Press.

REFERENCES

2.1–2.2 (cont.)

- Greenhough, T. J. & Helliwell, J. R. (1982). *Oscillation camera data processing: reflecting range and prediction of partiality. 2. Monochromatic synchrotron X-radiation from a singly bent triangular monochromator. J. Appl. Cryst.* **15**, 493–508.
- Hamilton, W. C. (1974). *Angle settings for four-circle diffractometers. International tables for X-ray crystallography*, Vol. IV, pp. 273–284. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- Hamlin, R. (1985). *Multi-wire area X-ray diffractometers. Methods Enzymol.* **114A**, 416–451.
- Hamlin, R., Cork, C., Howard, A., Nielsen, C., Vernon, W., Matthews, D., Xuong, Ng. H. & Perez-Mendez, V. (1981). *Characteristics of a flat multiwire area detector for protein crystallography. J. Appl. Cryst.* **14**, 85–93.
- Harrison, S. C., Winkler, F. K., Schutt, C. E. & Durbin, R. (1985). *Oscillation method with large unit cells. Methods Enzymol.* **114A**, 211–236.
- Hart, M. (1971). *Bragg reflection X-ray optics. Rep. Prog. Phys.* **34**, 435–490.
- Hastings, J. B. (1977). *X-ray optics and monochromators for synchrotron radiation. J. Appl. Phys.* **48**, 1576–1584.
- Hastings, J. B., Kincaid, B. M. & Eisenberger, P. (1978). *A separated function focussing monochromator system for synchrotron radiation. Nucl. Instrum. Methods*, **152**, 167–171.
- Helliwell, J. R. (1984). *Synchrotron X-radiation protein crystallography: instrumentation, methods and applications. Rep. Prog. Phys.* **47**, 1403–1497.
- Helliwell, J. R. (1985). *Protein crystallography with synchrotron radiation. J. Mol. Struct.* **130**, 63–91.
- Helliwell, J. R. (1992). *Macromolecular crystallography with synchrotron radiation*. Cambridge University Press.
- Helliwell, J. R. & Wilkinson, C. (1994). *X-ray and neutron Laue diffraction*. In *Neutron and synchrotron radiation for condensed matter studies: applications to soft condensed matter and biology*, Vol. III, edited by J. Baruchel, J. L. Hodeau, M. S. Lehmann, J. R. Regnard & C. Schlenker. Berlin: Springer Verlag.
- Henry, N. F. M., Lipson, H. & Wooster, W. A. (1951). *The interpretation of X-ray diffraction photographs*. London: Macmillan.
- Higashi, T. (1989). *The processing of diffraction data taken on a screenless Weissenberg camera for macromolecular crystallography. J. Appl. Cryst.* **22**, 9–18.
- Howard, A., Nielsen, C. & Xuong, Ng. H. (1985). *Software for a diffractometer with multi-wire area detector. Methods Enzymol.* **114A**, 452–472.
- International Tables for X-ray Crystallography* (1959). Vol. II. Birmingham: Kynoch Press.
- Jeffery, J. W. (1958). *An investigation of the blank areas on Laue photographs round: 1. The direct beam, and 2. Reflections with simple indices. Z. Kristallogr.* **110**, 321–328.
- Kohra, K., Ando, M., Matsushita, T. & Hashizume, H. (1978). *Design of high-resolution X-ray optical system using dynamical diffraction for synchrotron radiation. Nucl. Instrum. Methods*, **152**, 161–166.
- Lairson, B. M. & Bilderback, D. H. (1982). *Transmission X-ray mirror – a new optical element. Nucl. Instrum. Methods*, **195**, 79–83.
- Lemonnier, M., Fourme, R., Rousseaux, F. & Kahn, R. (1978). *X-ray curved-crystal monochromator system at the storage ring DCI. Nucl. Instrum. Methods*, **152**, 173–177.
- McKie, D. & McKie, C. (1986). *Essentials of crystallography*. Oxford: Blackwell Scientific Publications.
- Moffat, K., Schildkamp, W., Bilderback, D. H. & Volz, K. (1986). *Laue diffraction from biological samples. Nucl. Instrum. Methods*, **A246**, 617–623.
- Rabinovich, D. & Lourie, B. (1987). *Use of the polychromatic Laue method for short-exposure X-ray diffraction data acquisition. Acta Cryst.* **A43**, 774–780.
- Rossmann, M. G. (1985). *Determining the intensity of Bragg reflections from oscillation photographs. Methods Enzymol.* **114A**, 237–280.
- Sakabe, N. (1983). *A focusing Weissenberg camera with multilayer-line screens for macromolecular crystallography. J. Appl. Cryst.* **16**, 542–547.
- Sakabe, N. (1991). *X-ray diffraction data collection systems for modern protein crystallography with a Weissenberg camera and an imaging plate using synchrotron radiation. Nucl. Instrum. Methods*, **A303**, 448–463.
- Stout, G. H. & Jensen, L. H. (1968). *X-ray structure determination: a practical guide*, pp. 83–194. New York: Macmillan.
- Vainshtein, B. K. (1981). *Modern crystallography*. I, pp. 297–300. Berlin: Springer.
- Weisgerber, S. & Helliwell, J. R. (1993). *High-resolution crystallographic studies of native concanavalin A using rapid Laue data collection methods and the introduction of a monochromatic large-angle oscillation technique (LOT). J. Chem. Soc. Faraday Trans.* **89**, 2667–2675.
- Weissenberg, K. (1924). *Ein neues Röntgengoniometer. Z. Phys.* **23**, 229–238.
- Witz, J. (1969). *Focusing monochromators. Acta Cryst.* **A25**, 30–42.
- Wlodawer, A. (1985). *Methods Enzymol.* **114A**, 551–564.
- Woolfson, M. M. (1970). *Introduction to X-ray crystallography*. Cambridge University Press.
- Woolfson, M. M. (1997). *Introduction to X-ray crystallography*, 2nd ed. Cambridge University Press.
- Wyckoff, H. W. (1985). *Diffractometry. Methods Enzymol.* **114A**, 330–385.
- Wyckoff, H. W., Hirs, C. H. W. & Timasheff, S. N. (1985). *Diffraction methods for biological macromolecules. Part A. Methods in Enzymol.* **114A**, 199–588.
- Xuong, Ng. H., Nielsen, C., Hamlin, R. & Anderson, D. (1985). *Strategy for data collection from protein crystals using a multiwire counter area detector diffractometer. J. Appl. Cryst.* **18**, 342–350.

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- Ahtee, M., Nurmela, M., Suortti, P. & Järvinen, M. (1989). *Correction for preferred orientation in Rietveld refinement. J. Appl. Cryst.* **22**, 261–268.
- Alexander, L. E. (1969). *X-ray diffraction methods in polymer science*. New York: John Wiley. [Reprint 1979; Huntington, New York: Krieger.]
- Anderson, C. A. F., Zolensky, M. E., Smith, D. K., Freeborn, W. P. & Scheetz, B. E. (1981). *Applications of Gandolfi X-ray diffraction to the characterization of reaction products from the alteration of simulated nuclear wastes. Adv. X-ray Anal.* **24**, 265–269.
- Andrews, S. J., Papiz, M. Z., McMeeking, R., Blake, A. J., Lowe, B. M., Franklin, K. R., Helliwell, J. R. & Harding, M. M. (1988). *Piperazine silicate (EU 19): the structure of a very small crystal determined with synchrotron radiation. Acta Cryst.* **B44**, 73–77.