

## 2.10. SPECIMEN PREPARATION

tering length consists of a coherent component, which is the average over all spins, and an incoherent part, which gives the deviation from this average value. In other words, coherent scattering describes interference between waves produced by the scattering of a single neutron from all the neutrons in the nuclei of the sample. On the other hand, incoherent scattering involves correlations between the position of an atom  $j$  at time zero and the position of the same atom at time  $t$ , and so the scattered waves from different nuclei no longer interfere. Thus incoherent scattering provides an excellent tool for studying processes involving atomic diffusion, but produces large backgrounds for diffraction experiments. Based on the available flux at the instrument of choice and the atom% hydrogen present in the sample, complete or partial deuteration of the sample may be necessary.

When illuminated by a neutron beam, some nuclei are converted into other radioactive nuclei (activated). Thus it may not be possible to return the specimen to the home laboratory, but it may have to be treated as radioactive waste. A sample-activation calculator is also available at <http://www.ncnr.nist.gov/resources/activation/>.

## 2.10.3. Conclusions

Powder diffraction as a technique encompasses a wide range of possible experimental setups. Diverse though they are, specimen-preparation methods are a key component in obtaining the best possible data for the best possible analysis. Many issues are common whether X-rays or neutrons are the probe of choice (e.g. particle statistics, preferred orientation), but neutrons do pose some unique issues such as sample activation and isotope-dependent scattering behaviour. Given that X-ray and neutron diffraction are frequently used in a joint analysis, some forethought may be required if the desired situation of the same sample being used for both is to be achieved.

For the common laboratory setups a recurring theme should be apparent through this overview – specimens should ideally have crystallite sizes of the order of a few  $\mu\text{m}$ . If this is the case, then many of the issues mentioned (particle statistics, preferred orientation, extinction) will either disappear or be significantly reduced. The same is true for microabsorption, except in this case it is particle as opposed to crystallite sizes that are the issue. Grinding or milling can easily reduce crystallite size to this range, but the milling action should be chosen so as to avoid damaging the crystal structure of the sample or possibly amorphizing it completely.

Ideally, the in-house laboratory should have the flexibility to tailor the experiment to the sample, using transmission or reflection geometry depending on the nature of the sample. Unfortunately, in many instances this is not possible. ‘Coping strategies’ for non-ideal samples such as diluting samples in capillaries or using very thin organic specimens on flat plates are available, but their limitations and compromises should be understood by the person receiving the data.

In many instances, the experimental configuration at a central facility, such as a synchrotron or neutron source, can be customized for a particular experiment. However, the need for high throughput for rapid-access mail-in services may dictate a more standardized setup.

In summary, specimen preparation is the foundation upon which powder diffraction measurements are built. Good specimen preparation will not guarantee excellent data, but poor preparation can pretty much guarantee poor data.

We would like to acknowledge the assistance of Mati Raudsepp of the Department of Earth and Ocean Sciences, University of British Columbia, for the production and SEM micrograph of the spray-dried mica sample. Ashfia Huq acknowledges support by the US Department of Energy (DE-AC05-00OR22725) with UT-Battelle, LLC. R.

## References

- Alexander, L. E. & Klug, H. P. (1948). *Basic aspects of X-ray absorption in quantitative diffraction analysis of powder mixture*. *Anal. Chem.* **20**, 886–894.
- Alexander, L., Klug, H. P. & Kummer, E. (1948). *Statistical factors affecting the intensity of X-rays diffracted by crystalline powders*. *J. Appl. Phys.* **19**, 742–753.
- Bailey, I. F., Done, R., Dreyer, J. W. & Gray, E. M. (2004). *A high-temperature high-pressure gas-handling cell for neutron scattering measurements*. *High Press. Res.* **24**, 309–315.
- Bowden, M. & Ryan, M. (2010). *Absorption correction for cylindrical and annular specimens and their containers or supports*. *J. Appl. Cryst.* **43**, 693–698.
- Bravais, A. (1866). *Etudes Cristallographiques*. Paris: Gauthier Villars.
- Brindley, G. W. (1945). *The effect of grain or particle size on X-ray reflections from mixed powders and alloys, considered in relation to the quantitative determination of crystalline substances by X-ray methods*. *London Edinb. Dubl. Philos. Mag. J. Sci.* **36**, 347–369.
- Buhrke, V. E., Jenkins, R. & Smith, D. K. (1998). *A Practical Guide for the Preparation of Specimens for X-ray Fluorescence and X-ray Diffraction Analysis*. New York: Wiley-VCH.
- Cline, J. P. & Snyder, R. L. (1987). *The effects of extinction on X-ray powder diffraction intensities*. *Adv. X-ray Anal.* **30**, 447–456.
- Dollase, W. A. (1986). *Correction of intensities for preferred orientation in powder diffractometry: application of the March model*. *J. Appl. Cryst.* **19**, 267–272.
- Donnay, J. D. H. & Harker, D. (1937). *A new law of crystal morphology extending the law of Bravais*. *Am. Mineral.* **22**, 446–467.
- Friedel, G. (1907). *Etudes sur la loi de Bravais*. *Bull. Soc. Fr. Mineral.* **30**, 326–455.
- Hillier, S. (1999). *Use of an air brush to spray dry samples for X-ray powder diffraction*. *Clay Miner.* **34**, 127–135.
- Hillier, S. (2002). *Spray drying for X-ray powder diffraction specimen preparation*. IUCr Commission on Powder Diffraction Newsletter, **27**, 7–9. [http://www.iucr.org/\\_data/assets/pdf\\_file/0017/21626/cpd27.pdf](http://www.iucr.org/_data/assets/pdf_file/0017/21626/cpd27.pdf).
- Huq, A., Richardson, J. W., Maxey, E. R., Chandra, D. & Chien, W. (2007). *Structural studies of Li<sub>3</sub>N using neutron powder diffraction*. *J. Alloys Compd.* **436**, 256–260.
- International Tables for Crystallography* (2006). Volume C, *Mathematical, Physical and Chemical Tables*, 1st online edition, edited by E. Prince. Chester: International Union of Crystallography. doi:10.1107/97809553602060000103.
- Järvinen, M. (1993). *Application of symmetrized harmonics expansion to correction of the preferred orientation effect*. *J. Appl. Cryst.* **26**, 525–531.
- Jenkins, R. & Snyder, R. (1996). *Introduction to X-ray Powder Diffraction*. New York: Wiley-Interscience.
- Kaduk, J. A. (2009). *A Rietveld tutorial – mullite*. *Powder Diffr.* **24**, 351–361.
- Kaduk, J. A. (2013). Personal communication.
- Klug, H. P. & Alexander, L. E. (1954). *X-ray Diffraction Procedures for Polycrystalline and Amorphous Materials*. New York: Wiley-Interscience.
- Lewis, J., Schwarzenbach, D. & Flack, H. D. (1982). *Electric field gradients and charge density in corundum,  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>*. *Acta Cryst.* **A38**, 733–739.
- Lobanov, N. N. & Alte de Viegas, L. (1998). *Analytic absorption correction factors for cylinders to an accuracy of .5%*. Abstract P 2-16, 6th European Powder Diffraction Conference, Budapest, Hungary, 22–25 August. Zurich: Trans Tech Publications.
- Ohashi, Y. (1984). *Polysynthetically-twinned structures of enstatite and wollastonite*. *Phys. Chem. Miner.* **10**, 217–229.
- Pitschke, W., Hermann, H. & Mattern, N. (1993). *The influence of surface roughness on diffracted X-ray intensities in Bragg–Brentano geometry and its effect on the structure determination by means of Rietveld analysis*. *Powder Diffr.* **8**, 74–83.