The HKU Scholars Hub   The University of Hong Kong   香港大學學術庫

| Title	Powder to become crystal clear
Author(s)	Hao, Q
Citation	IUCrJ, 2015, v. 2 n. 3, p. 307-308
Issued Date	2015
URL	http://hdl.handle.net/10722/214503
Rights	Creative Commons: Attribution 3.0 Hong Kong License

Powder to become crystal clear

Quan Hao*

Physiology, L04-48 Laboratory Block, University of Hong Kong, 21 Sassoon Road, Hong Kong. *Correspondence e-mail: qhao@hku.hk

The development of X-ray free electron lasers (XFELs) has opened up new opportunities for experiments that seem impossible now to become a reality in the near future. One of the new capabilities of XFELs is to collect single-crystal diffraction data from randomly oriented sub-micron-sized crystals using serial femtosecond crystallography (SFX) (Chapman *et al.*, 2011).

Many important materials, such as zeolites, are polycrystalline (powders) and cannot be grown as single crystals. Furthermore, different types of samples (multiphase) may be mixed during production of a material; for example, zeolite NU-87 may occur as an impurity in zeolite TNU-9 (Hong *et al.*, 2007). X-ray diffraction from such samples will usually result in a one-dimensional powder pattern (Fig. 1, left). Because of the relatively large molecular size (76 non-hydrogen atoms in the case of TNU-9), the powder diffraction pattern from a zeolite can be difficult to interpret (Gramm *et al.*, 2006). The powder diffraction pattern from a mixture of TNU-9 and NU-87 would be impossible to process.
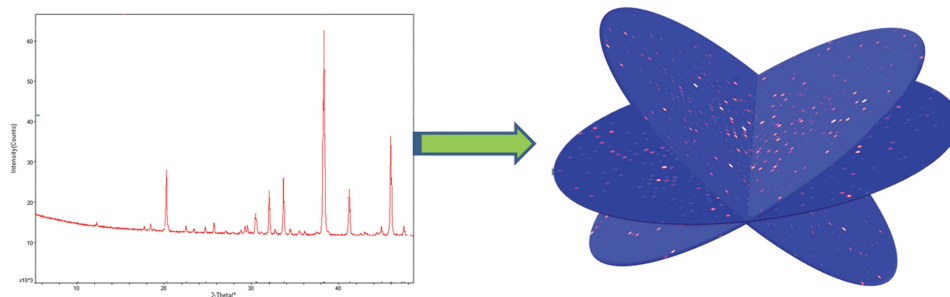

Figure 1
A one-dimensional powder diffraction pattern seen using conventional methods (left) may potentially be analysed as three-dimensional single-crystal patterns using serial crystallography (right).

Powder samples are essentially a mixture of sub-micron-sized (typically 100 nm) single crystals. The latest sample handling techniques, such as liquid jet injectors, can deliver the crystals to the beam one at a time and the extremely intense XFEL beam can capture a diffraction image of each crystal in a sub-nanosecond time scale (Spence *et al.*, 2012). In this issue, Zhang *et al.* (2015) have proposed the use of serial crystallography to turn powder diffraction into single-crystal diffraction (Fig. 1). A test has been performed using simulated diffraction patterns. The test sample is a mixture of zeolites TNU-9 and NU-87 with crystal grain sizes as small as 100 nm. X-ray diffraction snapshots by SFX were simulated and processed using the program suite *CrystFEL* (White *et al.*, 2012). Identification according to the primitive unit-cell volume determined from individual snapshots was able to separate the whole set of snapshots into two subsets, which matched the two zeolites in the sample. Monte Carlo integration in *CrystFEL* was then applied to them separately. Two sets of three-dimensional single-crystal diffraction intensities could then be derived. The crystal structures of the two zeolites were solved using the direct methods program *SHELXD* (Sheldrick, 2008) with default parameters.

Turning one-dimensional diffraction from polycrystalline (powder) samples, particularly from multiphase samples, into three-dimensional single-crystal diffraction patterns

scientific commentaries
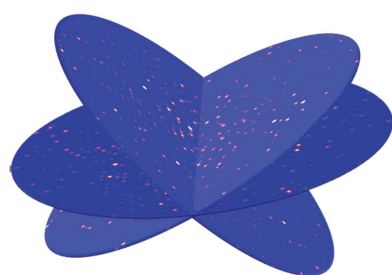
has long been regarded as a difficult, if not impossible, task. Zhang *et al.*'s proof-of-principle study has demonstrated that with the latest XFEL and sample delivery technology, single-crystal diffraction patterns can be collected from multiphase polycrystalline samples, processed, and then the molecular structures can be solved *ab initio*. This technique promises to open up new avenues for the study of many important polycrystalline materials that cannot be analysed by conventional X-ray powder diffraction methods.

References

Chapman, H. N., Fromme, P., Barty, A., White, T. A., Kirian, R. A., Aquila, A., Hunter, M. S., Schulz, J., DePonte, D. P., Weierstall, U., Doak, R. B., Maia, F. R. N. C., Martin, A. V., Schlichting, I., Lomb, L., Coppola, N., Shoeman, R. L., Epp, S. W., Hartmann, R., Rolles, D., Rudenko, A., Foucar, L., Kimmel, N., Weidenspointner, G., Holl, P., Liang, M., Barthelmess, M., Caleman, C., Boutet, S., Bogan, M. J., Krzywinski, J., Bostedt, C., Bajt, S., Gumprecht, L., Rudek, B., Erk, B., Schmidt, C., Hömke, A., Reich, C., Pietschner, D., Strüder, L., Hauser, G., Gorke, H., Ullrich, J., Herrmann, S., Schaller, G., Schopper, F., Soltau, H., Kühnel, K. U., Messerschmidt, M., Bozek, J. D., Hau-Riege, S. P., Frank, M., Hampton, C. Y., Sierra, R. G., Starodub, D., Williams, G. J., Hajdu, J., Timneanu, N., Seibert, M. M., Andreasson, J., Rocker, A., Jönsson, O., Svenda, M., Stern, S., Nass, K., Andritschke, R., Schröter, C. D., Krasniqi, F., Bott, M., Schmidt, K. E., Wang, X., Grotjohann, I., Holton, J. M., Barends, T. R. M., Neutze, R., Marchesini, S., Fromme, R., Schorb, S., Rupp, D., Adolph, M., Gorkhover, T., Andersson, I., Hirsemann, H., Potdevin, G., Graafsma, H., Nilsson, B. & Spence, J. C. H. (2011). *Nature*, **470**, 73–77.

Gramm, F., Baerlocher, C., McCusker, L. B., Warrender, S. J., Wright, P. A., Han, B., Hong, S. B., Liu, Z., Ohsuna, T. & Terasaki, O. (2006). *Nature*, **444**, 79–81.

Hong, S. B., Min, H. K., Shin, C. H., Cox, P. A., Warrender, S. J. & Wright, P. A. (2007). *J. Am. Chem. Soc.* **129**, 10870–10885.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spence, J. C., Weierstall, U. & Chapman, H. N. (2012). *Rep. Prog. Phys.* **75**, 102601.

White, T. A., Kirian, R. A., Martin, A. V., Aquila, A., Nass, K., Barty, A. & Chapman, H. N. (2012). *J. Appl. Cryst.* **45**, 335–341.

Zhang, T., Jin, S., Gu, Y. X., He, Y., Li, M., Li, Y. & Fan, H. F. (2015). *IUCrJ*, **2**, 322–326.