

Received 24 February 2015

Accepted 26 February 2015

**Keywords:** serial crystallography; polycrystalline samples; multiphase.

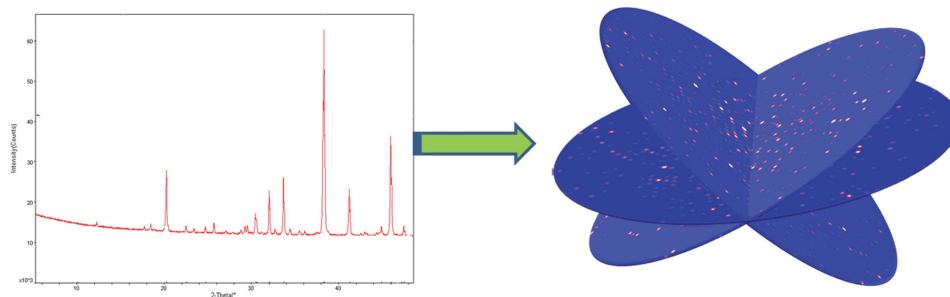
## Powder to become crystal clear

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.

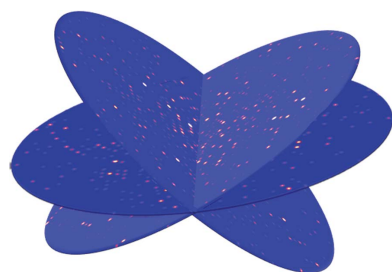


**Figure 1**

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



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.

### Acknowledgements

Dr Tao Zhang is thanked for help with preparing the figure.

### 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.