MS27-P3 Phase identification and structure determination of crystallites in multiphase powder samples by rotation electron diffraction

Wei Wan[1,2], Yifeng Yun[1,2], Faiz Rabbani[2], Jie Su[1,2], Hongyi Xu[1,2], Sven Hovmöller[1,2], Mats Johnsson[2], Xiaodong Zou[1,2]

1. Berzelii Center EXSELENT on Porous Materials, Stockholm University, SE-10691 Stockholm, Sweden
2. Inorganic and Structural Chemistry, Department of Materials and Environmental Chemistry, Stockholm University, SE-10691 Stockholm, Sweden

email: wei.wan@mmk.su.se

Phase identification and structure determination of submicrometre-sized crystals are important in materials science and crystallography. The most widely used technique for these purposes is powder X-ray diffraction (PXRD). However, the use of PXRD is limited in multiphase samples, especially those containing unknown phases. Electron crystallography has proven a promising alternative for studying individual phases in multiphase crystalline samples containing submicrometre-sized particles. The recently developed rotation electron diffraction (RED) method can be used to collect complete three-dimensional electron diffraction (ED) data from single crystals of submicrometre-sizes in a transmission electron microscope [1,2]. The unit cells and space groups of the crystals can be easily determined and the atomic structures can be solved *ab initio* and refined against the ED data or PXRD data. Here we show a case where RED made it possible to identify as many as four distinct compounds within one sample containing submicrometre-sized crystals in a Ni–Se–O–Cl system [3].

PXRD patterns of the sample could not be indexed using existing known phases (Fig. 1a). Four RED datasets were collected from four crystals with different morphologies and processed using the RED data processing software [2] (Fig. 1b-e). The unit cells were determined and the reflections were indexed. The reflection intensities were extracted and could be used to solve the structures of all four compounds by direct methods using SHELX [4] (Fig. 1f-i). One of the structures was later found in the ICSD database. One of the unknown compound was iso-structural to a known Co-Se-Cl-O phase. The other two structures were new. Nearly all peaks in the PXRD pattern could be indexed using the four phases and the structure models obtained from the RED data were confirmed by Rietveld refinement against the PXRD data.

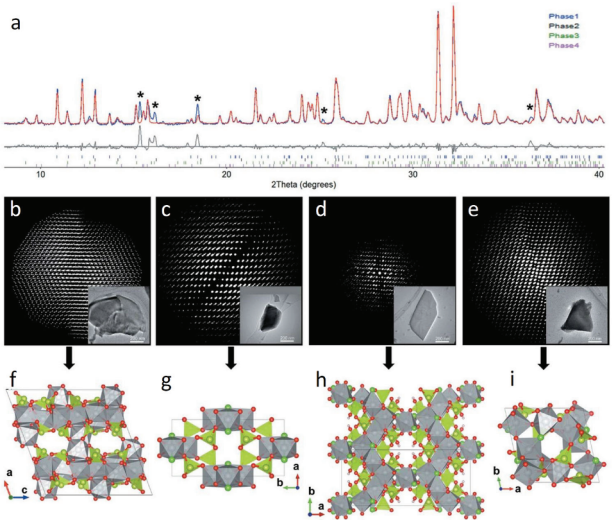

Figure 1. (a) Rietveld refinement against the PXRD pattern using the four phases determined from the RED data. (b)–(e) Reconstructed 3D reciprocal lattices of the four phases with the TEM images inserted. Scale bar = 200 nm. (f)–(i) Structure models of the four phases.

Keywords: electron diffraction, phase identification, structure solution

References

[1] Zhang, D., Oleynikov, P., Hovmöller, S. & Zou, X.D. (2010). Z. Kristallogr. 225, 94.

[2] Wan,W., Sun, J., Su, J., Hovmöller, S. & Zou, X.D. (2013). J. Appl. Cryst. 46, 1863.

[3] Yun, Y.F., Wan, W., Rabbani, F., Su, J., Xu, H.Y., Hovmöller, S., Johnsson, M. & Zou, X.D. (2014). J. Appl. Cryst. 47, 2048.

[4] Sheldrick, G. M. (2008). Acta Cryst. A64, 112.