CRYSTAL STRUCTURE DETERMINATION FROM X-RAY POWDER DIFFRACTION DATA

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Crystal structure solution from powder diffraction data is not a straightforward process mainly because of the accuracy of integrated intensity, which is relatively low due to peak overlap, preferred orientation and other factors. On the other hand, single crystals of suitable size for single crystal experiments cannot be prepared for many organic, metal-organic, inorganic and intermetallic samples. Using synchrotron radiation without doubt improves accuracy and resolution, however this source is not as easily accessible as the laboratory X-ray powder diffractometer. Hence structure determination from powder data (SDPD) using laboratory X-ray sources, ab-initio indexing and even Rietveld refinement is always challenging and often can be achieved only with the help of additional information about the sample or the use of other non-crystallographic methods.

In recent years, our group at Binghamton University has been involved in developing new materials that can be used as a cathode in rechargeable lithium batteries. These materials have usually open framework structure, often layered, which along with often relatively low crystallinity and because of that inability to grow crystals provide a lot of challenges for SDPD. Nevertheless, ab-initio crystal structure determination was successfully used to solve the crystal structure of over 15 compounds. The structure of many other compounds, especially intercalates, was completed using Rietveld refinement in parallel with other methods.

The title presentation summarizes the results of solving structures from powder data and discusses problematic cases. Most of the structures were challenging in many different ways: ab-initio indexing in the presence of other phases; powder pattern decomposition with substantial peak broadening at elevated angles; preferred orientation correction (prior to solving structure) and refinement (several different axes). Of course the main challenge was to solve the structure that was done using different methods: direct (automatic) or heavy atom (manual), geometry optimization (DLS) or energy minimization, always involving a lot of chemical and structural knowledge and other analytical data.