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The impacts of advanced electron crystallographic techniques on structure determination and phase analysis of nanoporous materials

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Abstract Text: Nanoporous materials are often synthesised in polycrystalline and multiphasic forms, which makes their phase analysis and structure determination by X-ray diffraction difficult. Electron crystallography has unique advantages for studying such materials¹⁻⁵. During the past decade, several important developments have been achieved in electron crystallography, which made large impacts in the structure elucidation of nanoporous materials.

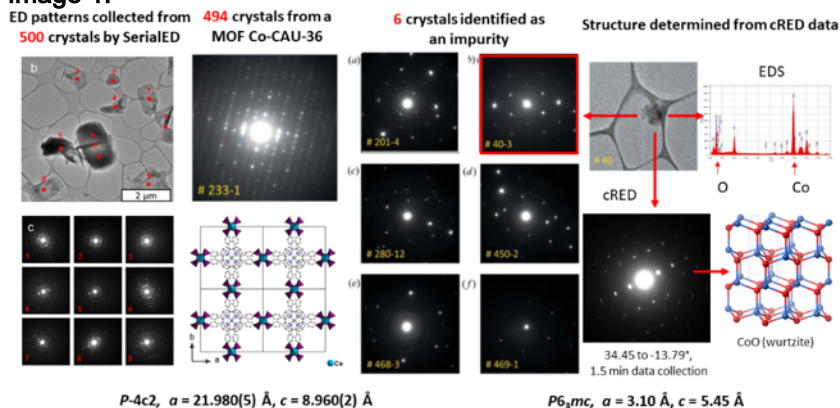
Here I will present the latest developments of 3D electron diffraction techniques, especially the fast and high-throughput data collection by continuous rotation electron diffraction (cRED).⁴ Today a complete cRED data can be collected in a few minutes or less, which allows high quality atomic resolution data to be collected on electron beam-sensitive materials such as zeolites, metal-organic frameworks (MOFs) and porous coordination polymers. I will show that the structure determination by cRED is as feasible and accurate as that by single crystal X-ray diffraction.⁴⁻⁶

To further speed up and automate data collection, we have developed serial electron diffraction (SerialED), which can automatically screen > 3500 particles/hour.⁷ This provides new possibilities for studying very beam sensitive crystals. We also combined data collection by SerialED with cRED and developed SerialRED to perform fully automated data collection and data analysis for 3D electron diffraction.⁸ The large number of particles makes it possible for phase analysis, and for detection of minor impurity phases that may not be detectable even by X-ray diffraction (Fig. 1). I will show their impacts in structure determination and phase analysis of multiphasic samples.

The rapid development of new electron diffraction techniques offers fast and accurate structure determination of nanoporous materials. We expect that the SerialRED in combination with hierarchical cluster analysis will make 3D ED a promising technique for high throughput phase analysis and *ab initio* structure determination.

Image 1. High throughput 3D electron diffraction and its application on *ab initio* structure determination and phase analysis. The method can screen hundreds of crystals/hour, and perform cRED data collection and analysis. Minor phases can be detected and the structure can be determined. An example on a Co-CAU-36 sample is shown.^{4,8}

Image 1:



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