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Serial electron diffraction for phase analysis and structure determination

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Can electron diffraction be used to perform serial snapshot crystallography? In an attempt to answer this question, we have developed a strategy to collect and process such data to characterize the structures of polycrystalline materials, particularly those that only form nano-sized crystals and are sensitive to radiation damage. These materials are difficult to measure using the conventional methods that rely on long exposure of the same crystal. In a serial electron crystallography experiment, each crystal is exposed only for a fraction of a second to avoid beam damage, but by combining snapshots from a large number of randomly oriented crystals, a complete data set can be assembled.

The advantages of using an electron microscope for serial crystallography are threefold: (1) electrons diffract much more strongly than X-rays, so that high-resolution diffraction data can be collected on nano-sized crystals, (2) crystals can be observed directly in imaging mode, eliminating the randomness typically associated with serial crystallography experiments, and (3) modern electron microscopes are computer controlled, such that the entire data collection process can be fully automated. To do so, our strategy combines goniometer translation with electron beam shift, which allows the entire sample stage to be probed. At each position of the sample stage, the locations of the crystals are identified using image recognition techniques. Diffraction data are then collected on each crystal using a quasi-parallel focused beam with a pre-defined size (usually 200-1000 nm). On our JEOL 2100-LaB6 microscope, equipped with a fast TimePix direct electron detector [1], approximately 1000-2000 diffraction patterns can be collected per hour. Data acquisition has been automated entirely, and can run unsupervised for hours after initial setup and calibration of the beam position.

Our tests show that these electron diffraction snapshots can be indexed reliably using a modified version of our previously developed algorithm to process serial snapshot data collected with a broad-bandpass beam [2]. The large number of crystals enables quantitative phase analysis and automatic screening of materials for known and unknown phases. Snapshots from crystals with the same lattice parameters can be combined and used for structure determination. For this purpose, we developed a merging routine based on rank aggregation, which is robust when merging inaccurate data (e.g. because of dynamical scattering) and avoids the problem of scaling each frame individually. Although our interest is in inorganic materials, these methods may also be applied to other beam-sensitive compounds such as pharmaceuticals and proteins.

[1] van Genderen et al. (2016) Acta Cryst. A, 72, 236
[2] Dejoie et al. (2015) IUCrJ, 2, 361

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