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The Revolutionary Multigrain Crystallography Method for High-Pressure X-ray Diffraction

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For a century, x-ray crystallography has been conducted with one of the two extreme sample conditions: either a single crystal which produces a single set of diffraction spots directly corresponding to the geometric orientation of the crystal, or a powder sample which comprises a huge number of crystals so numerous that the diffraction spots overlap and merge into smooth rings while the geometric relation is completely lost and only the d-spacings information of diffraction planes is observed. High-pressure DAC is not optimized for either condition. Powder x-ray diffraction has the intrinsic limitations when the sample consists of multiple different phases with low symmetry. Diffraction rings cover most of the detector area, and only a couple of low angle diffraction rings are unique to a phase; most other rings overlap with one another and cannot be used for unique identification or accurate determination of crystallographic parameters. New structures and minor phases are often overshadowed by the diffraction of major phases and are impossible to find or identify. Advanced crystallographic software, such as Rietveld and LeBail refinement methods, may get most out of a powder pattern, but cannot overcome the intrinsic limitations. In addition, the common practice of integrating the 2D ring into a 1D peak plot throws away the valuable information of a whole dimension, such as the azimuthal angle of diffraction spot around the ring.

Single-crystal XRD contains orientation and geometrical relationship in addition to d-spacings and thus provides a definitive characterization of the unit cell and symmetry. Unless the crystal is orientated exactly relative to the incident monochromatic x-ray beam to satisfy the Bragg relation, the crystal gives no signal, and the detector remains empty. Rotating the crystal around the w-axis (perpendicular to the incident x-ray beam) can bring the crystal to Bragg condition and occasionally obtain single diffraction spots at a given angle and scanning step. In spite of the advantages, single crystal cannot be sustained through phase transitions and often breaks down into multiple crystals and generate spotty XRD patterns.

Spottiness, that is generally regarded as a flaw in powder XRD, can be turned into great advantages if we can separate individual crystallites and handle them as individual single crystals. The high-brilliance x-ray beam available at synchrotron facilities has made it possible to collect diffraction spots in a powder sample comprised of up to hundreds of submicron crystallites. The newly developed Multigrain Crystallography (MGC) package, which is a suite of programs used for processing and indexing diffraction spots, has been developed to separate and identify the crystallographic orientation of each individual crystallite in the aggregate of hundreds of crystallites. Once separated, the data set for each crystallite can be handled with the standard single-crystal refinement program identical to a stand-alone single crystal, resulting in excellent statistics in refinement and full coverage of the reciprocal space. The MGC method is very powerful in unequivocal determination of symmetry and unit cells, testing different indexing models, picking out minor phases, and resolving strain of individual crystallites, and will very likely replace the powder and single-crystal x-ray diffraction methods as the dominant crystallographic tool for future high pressure-temperature studies in DAC.

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