Electron diffraction tomography for the characterization of sub-micrometric minerals: application to metamict phases

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Single-crystal X-ray diffraction can be performed only on crystalline domains of some cubic microns, while most of hitherto unsolved minerals and many new synthetic phases do not grow in crystals of such dimensions. On the other hand, interpretation of X-ray powder diffraction data may be problematic for polyphasic samples and structures characterized by large cell parameters or pseudo-symmetry. Electron diffraction is able to deliver 3D structural data from single crystallites of few nanometers. This ability derives from the high cross section between electrons and matter and the possibility to focus the electron beam into a nanometric probe. In the last years, electron diffraction tomography (EDT) emerged as an efficient method for acquiring complete and quasi-kinematic data sets for ab-initio structure determination of sub-micrometric phases (Kolb et al., 2011).

The mineral charoite was one of the first structures determined on the basis of EDT data, and still one of the trickiest crystallographic cases faced by electron diffraction. Despite the fact that charoite is a well-known and commercially exploited mineral, its symmetry and structure determination was hampered because two commensurate and pseudo-symmetric polytypes grow together inside fibers less than 1 μm thick (Rozhdestvenskaya et al., 2011). In recent years, tomographic electron diffraction has been used for the characterization of several minerals and products of experimental geology occurring as minor, sub-micrometric phases in poly-mineralogical associations. The porous \((S_2)_{1+x}[^{11}Bi_{9-x}Te_x(OH)_6O_4(SO_4)_2]_2\) was the first natural phase initially recognized, and subsequently structurally determined, by EDT alone (Capitani et al., 2014).

Recently, EDT has been employed for the characterization of metamict phases. Metamict minerals undergo structural changes and amorphization due to the radioactive decay of hosted elements. Phase identification is commonly done on the basis of compositional data alone, or by powder diffraction performed after the sample has been heated in order to produce re-crystallization. Still, different compositional and mineralogical domains may merge in the process. We therefore exploited EDT for the characterization of sub-micrometric crystalline relics in metamict domains, allowing single-crystal ab-initio determination of natural samples without the need of any physical treatment.

