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Possibility of Improved Phasing Method for MicroED – experimental aspects

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The authors introduce some experimental data in support of a potential new way of capturing diffraction in a transmission electron microscope. There is a possibility to improve the phasing process in protein crystallography (Micro-ED) with this approach. To date, we have acquired several diffraction data sets from MgO nanocubes (as a test specimen), along with supporting conventional HRTEM and far-field diffraction data, for reference purposes. Data has been acquired in an FEI Titan microscope, operated at 300kV, as well as in a JEOL ARM-200F microscope, operated at 200kV. We have also acquired preliminary data on carbamazepine and lysozyme nanocrystals.

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Low-dose electron diffraction tomography

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New materials are invented every day, to respond to ever more complex societal and economic issues (pharmaceuticals, soil decontamination, energy conversion etc. ...). The understanding and development of these materials goes inevitably through a stage of structures determination. In the research on nanomaterials, transmission electron microscopy is proving to be a particularly suitable technique, since it exploits the property of electrons to interact strongly with matter - an extraordinary advantage for studying nano-sized materials.

However, important classes of materials like metal-organic frameworks (MOFs), zeolites and biological crystals are sensitive to the electron beam. Classical investigation techniques that necessitate a too high electron dose for the acquisition of the data then become powerless. New experimental methods are needed.

We present here an innovative method to study the atomic structure of sensitive materials by electron diffraction. Our method opens the field of application of electron crystallography to beam sensitive materials, such as MOFs and biological crystals. It requires only a standard TEM, without any particular equipment, except for a fast camera with a high sensitivity and a beam precession system.

Each aspect of low-dose electron diffraction tomography has been optimized to limit the total irradiation time of the sample: no preliminary crystalline orientation is necessary and the beam is systematically blanked between two successive exposures. Thus the crystal is irradiated only during the acquisition of diffraction patterns, i.e. only when the irradiation is useful in terms of diffraction.

The quality of the recorded data was tested by the resolution of two complex structures: $Sr_5CuGe_9O_{24}$ and manganese formiate $[Mn(HCOO)_2(H_2O)_2]_\infty$, the latter of which is beam sensitive. Both structures are obtained with great precision. We evaluated the number of frames needed to obtain reliable structural models.

Low-dose electron diffraction tomography has three main advantages:

- It provides a set of data suitable for structural resolution with an extremely low cumulative electron dose of 0.27 $e^-/Å^2$, ten times less than the dose usually used in cryo-TEM structural determinations.
- Because of its speed and the simplicity of its implementation, it can be used routinely for the study of a great number of particles in a powder sample.
- It only requires equipment that is standard in a large number of laboratories.