MS23-P05

Possibility of Improved Phasing Method for MicroED – experimental aspects

Cathal Cassidy[1], Tsumoru Shintake[1]

1. Okinawa Institute of Science & Technology, Onna, Japan

email: c.cassidy@oist.jp

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.

Keywords: electron diffraction, nanocrystal, phasing

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Low-dose electron diffraction tomography

Holger Klein[1], Stéphanie Kodjikian[1]

1. Institut Néel, Univ. Grenoble Alpes and CNRS, Grenoble, France

email: holger.klein@neel.cnrs.fr

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.

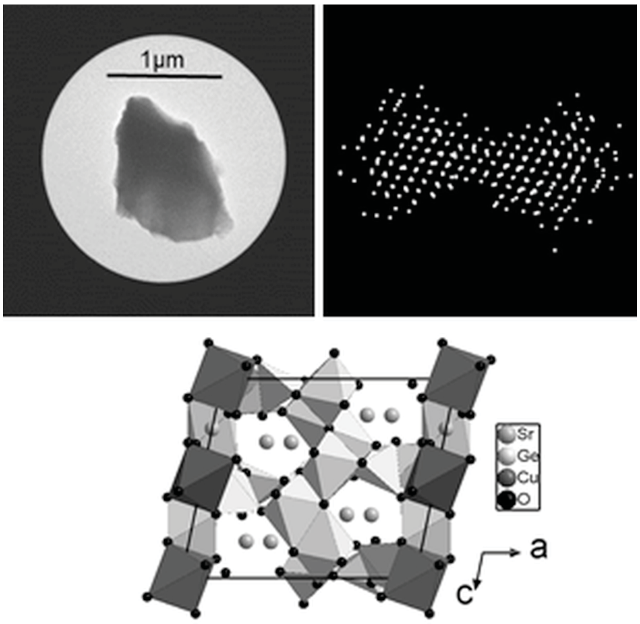
We present here an innovative method to study the atomic structure of sensitive materials by electron diffraction. Our method opens the field of application of electron crystallography to beam sensitive materials, such as MOFs and biological crystals. It requires only a standard TEM, without any particular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



Keywords: low dose, electron diffraction tomography, structure solution

MS23-P07

Applications of the highly efficient low-dose electron diffraction tomography

Stéphanie Kodjikian[1], Holger Klein[1]

1. Institut Néel, CNRS and Université Grenoble-Alpes, Grenoble, France

email: stephanie.kodjikian@neel.cnrs.fr

The understanding of the properties of a material comes from the knowledge of its structure. This is true for all materials, and beam sensitive materials don't escape from this rule. However, not only the complex structure of the latter is an issue, but also the difficulty of obtaining sufficient and reliable data before the sample is destroyed by the beam. With this in mind we have developed a new and highly efficient method of electron diffraction data acquisition: low-dose electron diffraction tomography (LD-EDT) that we apply here to two different materials.

A first test of the performance of this method was the solution of the complex monoclinic oxide $Sr_5CuGe_9O_{24}$ from a data set of 100 frames. PETS and Superflip in JANA2006 yielded a structural model containing all 22 independent atom positions. A comparison with the X-ray refined structure shows the high precision of our solution.

The successful application of the LD-EDT to beam sensitive materials depends on its efficient use of the electron dose. The total dose depends on the exposure time for each frame and the number of frames in the data set. As the completeness and the redundancy of the data have been shown to be decisive parameters in electron crystallography, a large number of recorded frames is an asset for the structure solution, while each additional frame adds some dose to the crystal.

We have therefore tested the influence of the number of LD-EDT frames in the data set on the structure solutions. The complete data set was reduced by using only 50, 40, 30 or 20 frames in the structure solution procedure. Even though the data set completeness and redundancy are much lower than what is usually necessary for precise structure solutions, they are sufficient when the data is acquired by LD-EDT. One reason for the tolerance towards smaller data sets might be the higher data quality due to the fact that the beam is larger than the sample and therefore the diffracting volume is the same for each frame during the acquisition.

Very short exposure times of the individual frames are also sufficient for the data acquisition. As an example the structure of a beam sensitive metal-organic framework (MOF), manganese formiate $[Mn(HCOO)_2(H_2O)_2]_\infty$, has been solved. Exposure times of 0.2 s in a very weak beam (total dose of 0.27 $e^-/Å^2$) yielded the complete structure to a high degree of precision.