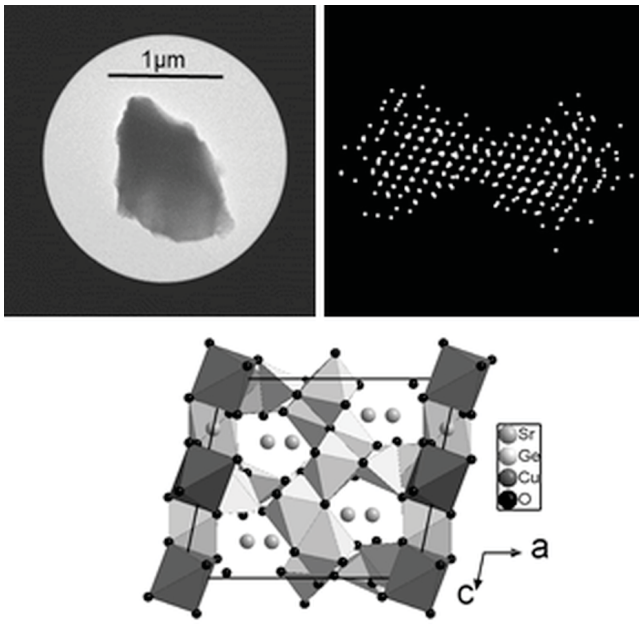

Keywords: low dose, electron diffraction tomography, structure solution

MS23-P07

Applications of the highly efficient low-dose electron diffraction tomography

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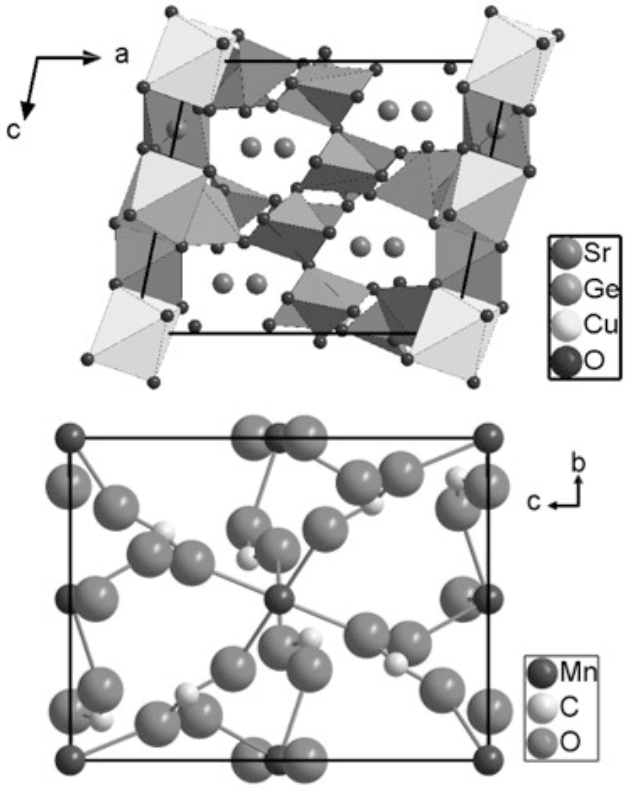
The understanding of the properties of a material comes from the knowledge of its structure. This is true for all materials, and beam sensitive materials don't escape from this rule. However, not only the complex structure of the latter is an issue, but also the difficulty of obtaining sufficient and 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.



Keywords: Low dose, Electron diffraction tomography, Structure solution

MS23-P08

First preliminary results from TAAM-UBDB refinement on electron diffraction data

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Cryo-electron microscopy and electron diffraction methods have made enormous progress in the last years and an increasing number of atomic and near atomic resolution structures are becoming available. At present, interpretation of collected data relay on a very approximate scattering model. The model is based on spherical independent atoms (IAM), ignoring the charge redistribution due to chemical bonding. This approximation may lead to unnecessary loss of information. We propose to base interpretation of data from cryo-electron microscopy and from electron diffraction methods on more realistic electron scattering models.

We are developing Transferable Aspherical Atom Models (TAAMs) from detailed electron densities of molecules and crystals. To build TAAMs we use a databank of aspherical atomic electron densities called UBDB [1]. Currently UBDB allow to reconstruct electron density of any protein, nucleic acid or other biologically important molecule. Thus, it gives also fast access to electrostatic potential.

It has been shown already [2] that replacement of the IAM by TAAM in x-ray crystallography leads to more accurate geometrical information and provide access to quantitative estimation of the electron density distribution and properties derived from it for molecules in a crystalline environment.

Given the fact that electron diffraction is more sensitive to charge density redistribution than x-ray we expect to see even more pronounced improvement after introduction of TAAM to analysis of electron diffraction/scattering.

We will present our first preliminary results of TAAM-UBDB refinements against electron diffraction data collected for paracetamol[3]. The figure illustrates the difference in crystal electrostatic potential (e/bohr) resulting from the differences between the IAM and the TAAM-UBDB electron scattering models.