Dynamical refinement of beam sensitive crystals: going below a total dose of 0.1 $e^-/Å^2$

H. Klein, E. Yörük, S. Kodjikian

Institut Néel, Université Grenoble Alpes, CNRS, 38000 Grenoble, France

holger.klein@neel.cnrs.fr

There is a wealth of materials that are beam sensitive and many of them only exist in nanometric crystals, because the growth of bigger crystals is either impossible or so complicated that it is not reasonable to spend enough time and resources to grow big crystals before knowing their potential for research or applications. This difficulty is encountered in minerals, zeolites, metal-organic frameworks or molecular crystals, including pharmaceuticals and biological crystals.

In order to study these crystals and potentially discover highly interesting materials a structure determination method that can deal with beam sensitive crystals of nanometric size is needed. The nanometric size makes them destined for electron diffraction, since electrons interact much more strongly with matter than X-rays or neutrons. In addition, for the same amount of beam damage, electron diffraction yields more information than X-rays [1].

The recently developed low-dose electron diffraction tomography (LD-EDT) [2] not only combines the advantages inherent in electron diffraction, but is also optimized for minimizing the electron dose used for the data collection. While using only minimal dose, the data quality is still high, allowing not only the solution of complex unknown structures, but also their refinement taking into account dynamical diffraction effects.

In this contribution we present several examples of crystals solved and refined by this method. The range of the crystals presented includes a synthetic oxide ($Sr_5CuGe_9O_{24}$), a natural mineral (bulachite) and a metal organic framework (Mn-formiate). The dynamical refinement can be successfully performed on data sets that needed less than 0.1 $e^-/Å^2$ for the entire data set.

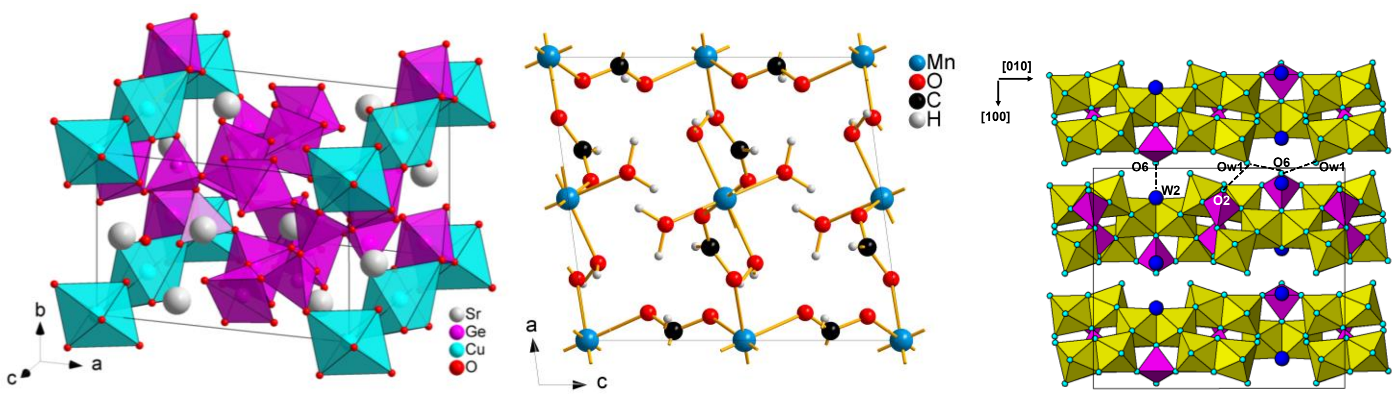

Figure 1. Projected structures of $Sr_5CuGe_9O_{24}$ (left), Mn-formaite (center) and bulachite (right)

[1] R. Henderson, Quarterly *Reviews of Biophysics* **28**, 2 (1995), 171-193

[2] S. Kodjikian and H. Klein, *Ultramicroscopy*, 2019, **200**, 12-19

Keywords: electron crystallography; beam sensitive materials; structure solution; structure refinement; minerals; MOF

The authors are grateful to the following colleagues for providing the samples used in this study: L. Ding (ISIS Facility, United Kingdom) for $Sr_5CuGe_9O_{24}$, D. Luneau (LMI, Lyon, France) for Mn-formiate and I. Grey (CSIRO Mineral Resources, Clayton South, Australia) for bulachite.