

# **Electron Diffraction Tomography: from the original design for FEI Tecnai TEM to the ultra-fast acquisition mode by ZEISS TEM and Medipix detector**

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The idea of collecting step-wise 3D electron diffraction data was implemented for the first time in 2007 by Ute Kolb and her co-workers at the University of Mainz [1]. In 2009, BaSO<sub>4</sub> was the first structure determined ab-initio by electron diffraction tomography (EDT) data [2], and shortly after structures as complex as charoite-90 and ITQ-43 [3-4] could be solved by a straight use of direct methods and kinematical approximation. About ten years later, we can sturdily argue that EDT has been a real turning point for electron crystallography, as it opened the possibility of routine structural characterization of materials too poorly crystalline and too complex for being properly addressed by X-ray methods, either single-crystal or powder. Trusting this view, a number of research groups in the world have pursued the idea of collecting 3D electron diffraction data, often proposing innovative experimental set-ups optimized for different classes of materials.

Since the beginning, EDT method was developed with the aim of collecting more complete data from nano-crystalline beam-sensitive materials, and specifically from organic samples. A semi-automatic acquisition system, initially named Automated Diffraction Tomography (ADT), was designed in collaboration with FEI and implemented in the Tecnai TEM available at the University of Mainz. Crystal position was tracked in STEM mode and electron diffraction data were acquired using a quasi-parallel 50 nm beam. Remarkably, for the first time very beam-sensitive samples like MOF [5] and small molecule organics [6] could be solved ab-initio by direct methods.

Still, further reduction the electron dose is necessary for working with even more beam sensitive materials. Two approaches may be adopted for this goal: speeding the acquisition routines, e.g. working with a continuous rotating sample [7], or/and using more sensitive data recorders [8], able to properly perform with a weaker illumination. In this regard, at CNI@NEST-Pisa we recently set up a modular acquisition system that includes: STEM imaging for crystal search, continuous electron diffraction data acquisition with a rotating sample, cryo-plunging and cryo-transfer sample mounting, beam-precession electron illumination, in-column energy filter for

cutting-out the non-elastic scattered signal and Timepix single-electron detector for data recording. According to the experimental conditions, continuous data acquisitions of up to 80 can be obtained in less than one minute. In this talk we will show our recent achievements for beam sensitive samples, like water-containing porous frameworks, pharmaceuticals and macro-molecules. Additionally, for samples free of defects and structural complications, we were able to spot H atoms ab-initio or after dynamical refinement [9].

## References

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