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TEM Random & Ultra-fast Precession ED Tomography for analysis of nm crystals

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Since the invention of Precession Electron Diffraction (PED) in Transmission Electron Microscopy (TEM) by Vincent & Midgley [1] in 1994 and mainly after the introduction of dedicated PED devices to different TEM, the structure of various nano-sized crystals have been solved by Electron Crystalography. The most popular technique that was recently developed based on beam precession is the 3D Precession Diffraction Tomography (PEDT) [2]. A series of ED patterns are collected every 1° while the sample is tilted around the goniometer axis. By the automatic measurement of ED intensities (ADT 3D software), the unit cell, crystal symmetry and the detailed crystal structure can be determined. A large number of crystal structures, such as complex metals, alloys, organic pigments, MOF, catalysts etc., have been solved by the 3D PEDT technique. A drawback of 3D PEDT (especially for beam sensitive materials) is the long acquisition times (45–120 min), due to the time consuming step of tracking the crystal under the beam during tilting. To deal with this problem, we have developed two novel approaches: the Random Electron Diffraction Tomography (rPEDT) technique and the Ultra-Fast 3D diffraction tomography (UF PEDT) [3]. By rPEDT technique, a sample area (few microns), where several crystals in different (random) orientations are present, is scanned rapidly using an ASTAR precession device (NanoMEGAS SPRL). PED patterns of all scanned crystals are collected by a fast speed CCD camera (up to 120 frames/sec; 8/12 bit). Concerning UF PEDT, the data acquisition time can be 10-20 times faster compared to hitherto 3D PEDT procedure. UF PEDT can be applied when the crystal shift is stable and reproducible during tilting the sample for a specific tilt range. Thus, such crystals can be tracked by shifting the beam following the crystal displacement during tilting (using ASTAR beam scanning). Obtained PED patterns can be recorded with a fast CCD camera, while crystal is tilted. As a conclusion, rPEDT and UF-PEDT can be considered as breakthrough techniques in electron crystallography as they can be performed in any commercial TEM. Both techniques reduce considerable 3D intensity data acquisition time, and allow the analysis of unknown compounds, including beam sensitive organic crystals, as fast techniques prevents crystal beam damage. The authors acknowledge financial support from EU ESTEEM-2 project (European Network for Electron Microscopy www.esteem2.eu).

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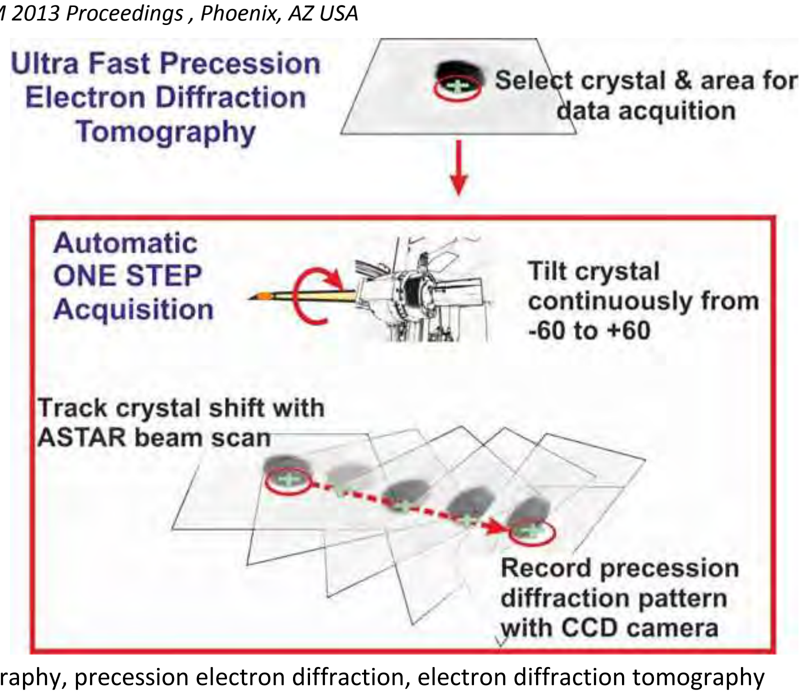

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