MS24-2-6 Dynamical refinement of 3D ED data: comparison of CCD and HPD data
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Abstract
Electron crystallography has gained a significant impact and interest over the last few years due to the development of 3D data acquisition techniques and analysis of the obtained 3D Electron diffraction data. The introduction of new, direct detectors provided a significant boost to the speed and data quality of 3D ED data. However, direct comparison of the data quality coming from these new detectors and from more traditional detectors has rarely, if ever, been done on real-life data. Therefore, we decided to profit from our microscope setup which allows easy switching between two detectors and compare the data collection, and structure refinement from both under otherwise identical conditions
The analysis was done on a TEM FEI Tecnai G2 20 with an accelerating voltage of 200 kV, equipped with a CCD detector Olympus SIS Veleta, dynamic range 14 bit, and a hybrid pixel detector Medipix 3 ASI Cheetah. Both detectors are retractable, mounted at the same height at opposite side-entry ports of the microscope, having an almost identical field of view. Therefore, data can be collected on both without changing any alignment settings of the microscope.
Three data sets were collected on a selected crystal of Lutetium Aluminium Garnet ($Lu_3Al_5O_{12}$, LAG) in continuous rotation mode, with varying exposure times.
All the data sets were processed with PETS2[1] software, and the processing parameters like the rocking curve width and mosaicity were kept constant for all data sets. The structure was then solved and refined in JANA2020[2] using the dynamical refinement. Even though results indicate that after kinematical and dynamical refinement, data collected from the CCD detector (C1) has very good R-values, the number of observed reflections was low compared to the other two datasets (H1 and C2). The obtained results depict some differences in the R-factors, Fourier maps, and atomic displacement parameters of the dynamical refinement between the detector systems, but they are still inconclusive to identify the best among the two.

References
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2. Petrícek, Václav & Dusek, Michal & Palatinus, Lukáš. (2014). Crystallographic Computing System JANA2006: General features. Zeitschrift für Kristallographie - Crystalline Materials. 229. 10.1515/zkri-2014-1737.

Comparison of R-factors between HPD and CPD

DETECTORS	HPD	CCD	
	H1	C1	C2
Exposure time	500	1000	2828
KINEMATIC REFINEMENT			
N(obs)	310	231	310
N(all)	310	310	310
R(obs)	30.16	24.34	32.88
wR(obs)	40.64	25.71	46.12
R(all)	30.16	26.85	32.33
wR(all)	40.64	25.89	46.12
DYNAMIC REFINEMENT			
N(obs)	3758	1560	3687
N(all)	3814	3953	3704
R(obs)	9.89	7.88	10.24
wR(obs)	11.12	7.81	11.71
R(all)	9.94	13.48	10.24
wR(all)	11.13	8.22	11.71