Oral Contributions

[MS32 - 05] Laboratory data collection using a pixel detector: application to ms time resolved crystallography

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X ray diffraction and scattering techniques are unique tools for studying the structural properties of crystals. Thanks to their unprecedented properties, the new generation of X-ray detectors, the hybrid pixel array detectors or 'pixel detectors' based on single-photon counting processes, are the future for accurate and time resolved diffraction experiments: high dynamic range and linearity, no readout noise, high signal-to-noise ratio, fast readout time and high frame rates are among their intrinsic characteristics which render them so attractive. First used on synchrotron beam lines, these detectors are also very promising at laboratory sources, in particular for pump-probe or quasi-static experiments and accurate electron density measurements as explained in this presentation. An original laboratory diffractometer made from a Nonius Mach3 goniometer equipped with an IμS Incoated Mo micro source and an XPAD [1] pixel array detector has been assembled at the CRM2 laboratory. Our XPAD detector has also been installed at the CRISTAL Soleil synchrotron beamline (mounted on a 6 circles goniometer for very precise Bragg peak angle shifts measurements). Among the many possible applications, we first have focused on three applications; diffraction of piezoelectrics under electric field [2] (CRM2 and Soleil), photo-crystallography (CRM2) and measurement of accurate structure factors for charge density analysis (CRM2). Here will be presented our new experimental approach to perform in-situ electric field diffraction on single crystals using the field-switching method with a synchrotron or a laboratory X-ray source. Taking advantages of the fast readout of the XPAD hybrid pixel 2D detector and its programmable electronic functionalities, the operation mode of the detector has been customized to significantly increase the efficiency of the method. The very weak electric field induced structural response of a piezoelectric crystal (Δ(I)/I about %) can be accurately measured. This also allows the piezoelectric tensor to be accurately obtained from Δq shifts whereas the structural variations can be modeled using a full set of ΔI/I data. This experimental method and methodology will be detailed as a case study on piezoelectric single crystals belonging to the a-quartz family. Using the two developed scan modes, we demonstrate that tiny Bragg angle shifts can be measured as well as small field induced Bragg intensities variations (< 1%). The relevance of measurements performed with our X-ray laboratory diffractometer will be evidenced: partial data sets collected at synchrotrons can be completed, but more interestingly, most of the data can now be performed in the laboratory (medium to strong intensity reflections) in a comparable data collection time [3]. To stress the quality of the laboratory data the room temperature Ylid[4] structure will also be discussed compared to that obtained from both Agilent Supernova with Atlas CCD and Bruker-AXS D8 Venture with PHOTON100 CMOS diffractometers.

[1] imXPAD SAS - Espace Mistral, Athélia IV, 297 avenue du Mistral, 13600 La Ciotat - www.imxpad.com

[2] Studies of electric field induced structural and electron-density modifications by X-ray diffraction: N. K. Hansen, P. Fertey and R. Guillot, *Acta Cryst. (2004). A60, 465-471*

[3] Diffraction studies under in-situ electric field using A 2D hybrid pixel XPAD detector : Fertey

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[4] The crystal structure of 2-dimethylsulfuranylidene-1,3-indanedione : A. T. Christensen and E. Thom, *Acta Cryst. (1971). B27, 581-586*

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