

Hybrid Photon Counting detectors for X-ray spectroscopy applications

Dr. Dubravka Šišak Jung & Dr. Nicolas Pilet, DECTRIS Ltd.

Introduction

From the early advent of the Hybrid Photon Counting (HPC) technology, X-ray spectroscopists have recognized the advantages of HPC-detectors. The first spectroscopic experiments with the PILATUS detector were carried out more than a decade ago [1, 2]. Ever since, HPC detectors are continuously changing the way we understand and practice X-ray spectroscopy at synchrotron beamlines and in laboratory spectrometers.

Laboratory spectrometers benefit from the large detection area that allows for collecting a wide energy spectrum in a single shot. Noise-free detection and small pixel (strip) size yield high resolution data even at long exposure times. This report presents two new spectrometers, one WDXRF and the other interchangeable WDXRF-XAS spectrometer with the following highlights:

- EXAFS and XANES experiments in a lab
- Energy resolution as high as 2 eV
- No moving parts for collecting the spectrum up to 1.24 keV

HPC-detectors are widely used at synchrotron beamlines, mostly for applications that require high time-resolution or single-photon sensitivity. This report presents XAFS, XRF-CT and RIXS experiments carried out under challenging conditions:

- Extremely short exposure times
- Sample heterogeneity
- High background signal
- Limited absorbed dose
- Single-shot data collection

¹ https://www1.psf.mit.edu/research/alcator/pubs/research_highlights/APS_2007_PRs/cmod_APS_2007.pdf

² Will, K.W. et al. (2008) Rev.Sci. Instrum. 79, 10E320.

XAFS: X-ray Absorption Fine Structure, XANES: X-ray Absorption Near-Edge Structure, XPCS: X-ray Photon Correlation Spectroscopy, WDXRF: Wavelength Dispersive X-Ray Fluorescence, RIXS: Resonant Inelastic X-ray Scattering (RIXS).

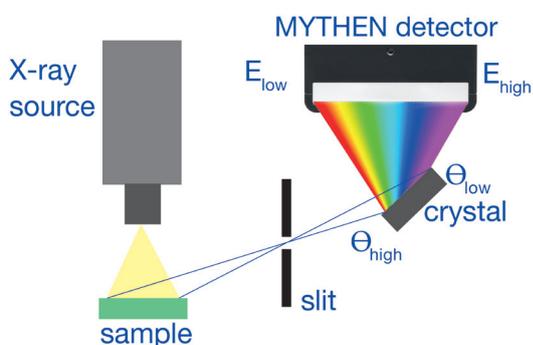
MYTHEN2 R strip detectors



- 50 μm strip
- 1280 or 640 strips per module
- 24-bit dynamic range
- Maintenance-free
- 3-year warranty

Collecting a wide energy spectrum without using mechanical moving parts was so far possible only at synchrotron sources. However, two recent developments based on the MYTHEN 1K detector show that this is also possible in laboratory spectrometers [1, 2]. MYTHEN's detection area allowed for collecting a spectrum up to 1.24 keV in a single shot, while its strip size pushed the energy resolution down to 2 eV per strip. This not only enables time-resolved high-resolution WDXRF studies but also EXAFS studies to be carried out in the lab.

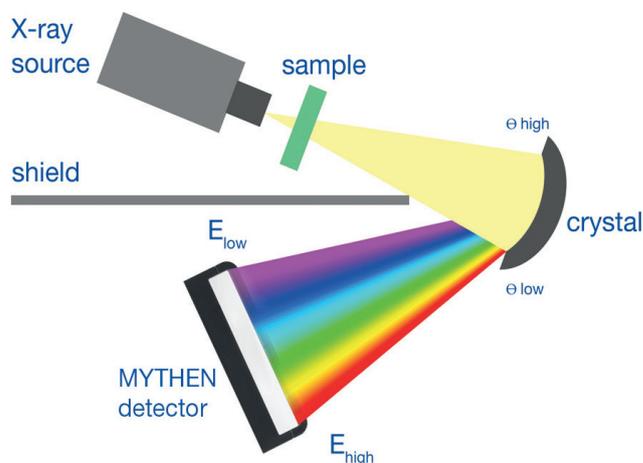
WD-XRF in a lab¹



This system was developed for analysis of 3d elements. The first results show that it is possible to distinguish Mn(VII) from Mn(II), making the system suitable for chemical state analysis of transition metals for *in-situ* studies.

- Spectrum of 1.24 keV covered in one shot
- Data acquisition 30 s
- Sampling width 0.98 eV per strip
- Energy resolution 3.9 eV

XAS-XES in a lab^{2,3}



This van Hámós spectrometer allows for XRF, XANES and EXAFS experiments to be carried out in a lab. The two setups are easily exchanged. The first results show that the system is suitable for both short and long-duration experiments.

- Energy resolution 2 eV
- LOD (Limit of detection) 0.33% for 1h exposure time
- Long exposure times for following chemical reactions

¹Sato, K. et al. (2017) *X-Ray Spectrom.* **46**, 330-335, <https://doi.org/10.1002/xrs.2797>

²Németh, Z. et al. (2016) *Rev.Sci. Instrum.* **87**, 103105, <https://doi.org/10.1063/1.4964098>

³Baynóczi, E. et al. (2017) *Inorg. Chem.* **56**(22), 14220-14226., <https://pubs.acs.org/doi/10.1021/acs.inorgchem.7b02311>

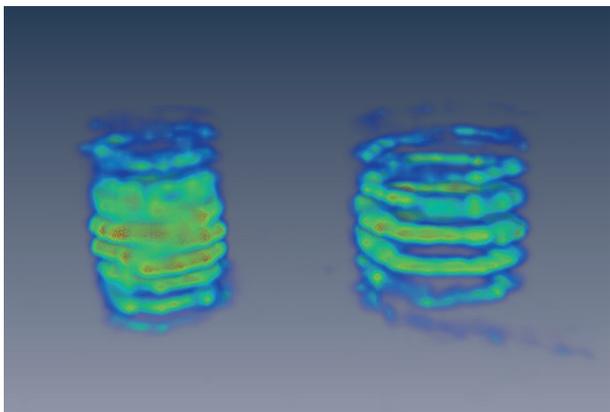
PILATUS3 X pixel detectors



- Large-area detectors up to 6M pixels
- Instant retrigger technology
- Gating and triggering options
- Vacuum option
- Frame rate up to 500 Hz
- Optional energy threshold 1600 eV

Large detection area, high dynamic range and no-noise performance of the PILATUS have resolved several factors limiting the speed and the quality of X-ray spectroscopy studies at synchrotron sources [4]. For EXAFS experiments that meant pushing the temporal resolution to sub-second range, even for inhomogeneous samples. These detector features have also set path to new approaches, such as *in vivo* 3D imaging using the XRF computed tomography.

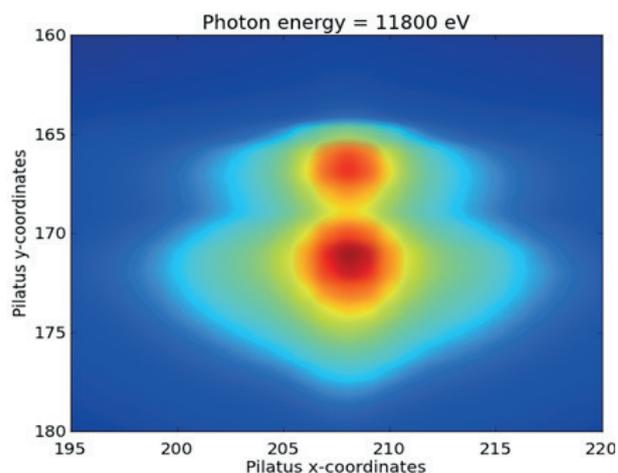
XRF-CT at synchrotrons⁵



The combination of a multi-pinhole and a large area detector reduces the time necessary to perform XRF-CT measurements without compromising the data quality. The reduced absorbed dose could pave a way to *in vivo* imaging.

- 3D imaging of 15 x 15 x 15 mm³ objects
- Spatial resolution < 0.35 mm
- LOD 38 µg/ml
- Exposure time 1 min

EXAFS at synchrotrons^{6,7}



In the transmission geometry, the detector enables the spatially-resolved monitoring of a reaction in real time.

In grazing incidence setup, the detector allowed for the simultaneous measurement of the complete scattering pattern of Bi/Au-bilayer sample (figure).

- Data acquisition down to sub-ms
- Spatially-resolved data can be obtained
- Different surfaces and interfaces can be simultaneously studied

⁴Micelli, A. (2009) *JINST* **4** P03024, <https://doi.org/10.1088/1748-0221/4/03/P03024>

⁵Sasaya, T. et al. (2017) *Scientific Reports*. **7**(1), 5742., <https://www.nature.com/articles/s41598-017-05179-2#rightslink>

⁶Lützenkirchen-Hecht, D. et al. (2016) *J. Phys.* **712**, 012017., <http://iopscience.iop.org/article/10.1088/1742-6596/712/1/012017>

⁷Tanida, H. et al. (2011) *J. Synch. Rad.* **18**, 919-922., <https://doi.org/10.1107/S0909049511031232>

EIGER X pixel detectors

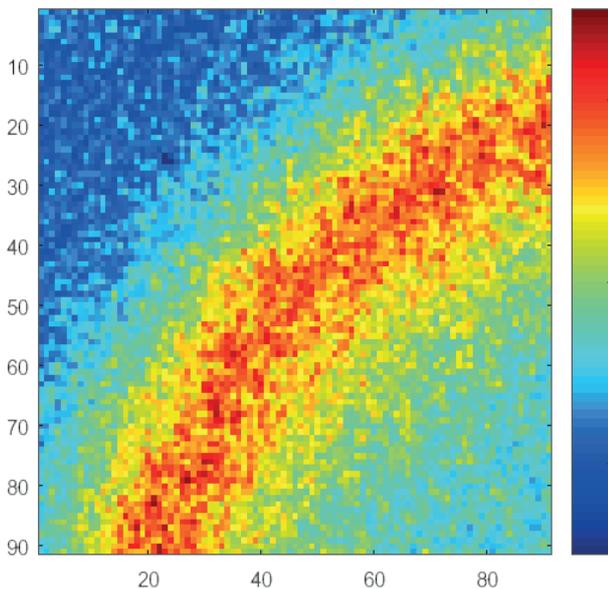


- Small pixel size 75 μm
- Up to 9000 Hz, continuous readout
- Image depth 32 bit
- 2×10^6 counts per pixel per second
- Vacuum option

EIGER detectors feature very small pixels, high count rates and image depth. This makes them ideal for the most demanding synchrotron applications. X-ray Photon Correlation Spectroscopy (XPCS) benefits from the single-photon sensitivity of the detector and the extreme frame rates of the detector.

In the other example, MYTHEN2 microstrip detector was used as a part of the high-resolution RIXS setup, optimized for low-lying magnetic dynamics of iridates.

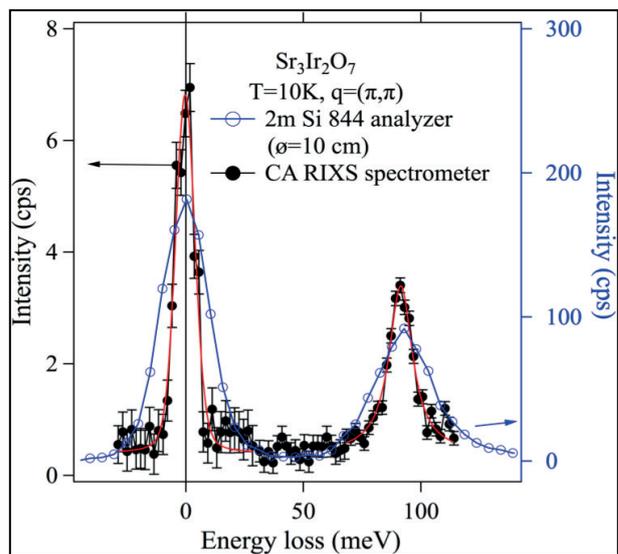
XPCS at synchrotrons⁸



Using the EIGER detectors, the distribution of the temporal fluctuations can be measured in the sub-second regime. Moreover, each pixel of the detector is an individual detector, so pixel-to-pixel intensity cross-correlations can be established.

- Single-photon sensitivity of the detector
- Pixel-to-pixel intensity cross-correlations
- Possibility to collect the SAXS and XPCS data simultaneously
- Sub-ms time resolution available

RIXS at synchrotron with MYTHEN2⁹



Comparison of the magnon spectrum of $\text{Sr}_3\text{Ir}_2\text{O}_7$ at the magnetic zone center measured by the flat-crystal RIXS spectrometer (black) and a standard RIXS spectrometer (blue).

- Sub-10 meV energy resolution at the Ir L3 edge
- Efficient polarization analysis without loss of energy resolution

⁸Courtesy of F. Westermeier, DESY.

⁹Kim, J. *et al.* (2018) Scientific Reports **8**, 1958 <https://www.nature.com/articles/s41598-018-20396-z#rightslink>