Time-resolved pump-probe-probe diffraction experiment using the large area XSPA 500k detector at Synchrotron SOLEIL

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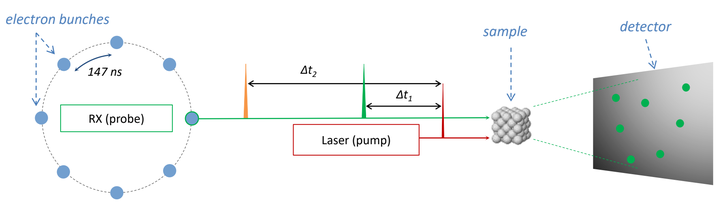
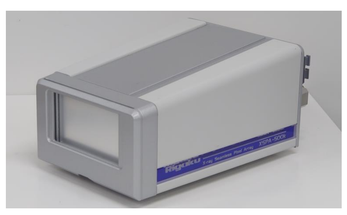
Introduction – scientific motivation

The XSPA 500k [1] is an X-ray single photon counting hybrid pixel detector based on UFXC32k readout chips [2], that has been developed by Rigaku Corporation.

The detector offers several unique features such as a seamless array of uniform pixels of 76 × 76 µm², very high-count rate (>2 × 10^6 cps/pixel), very fast readout (up to 56 kfps/2-bit per pixel) , and an ultra-short multi-gating operation (down to 40 ns). Recently the new multi-gating operation mode [3] has been verified experimentally in a time resolved pump-probe-probe diffraction experiment at CRISTAL beamline at Synchrotron SOLEIL and demonstrated its very good performance.



Scheme of the pump-probe-probe experiment



The XSPA-500k detector

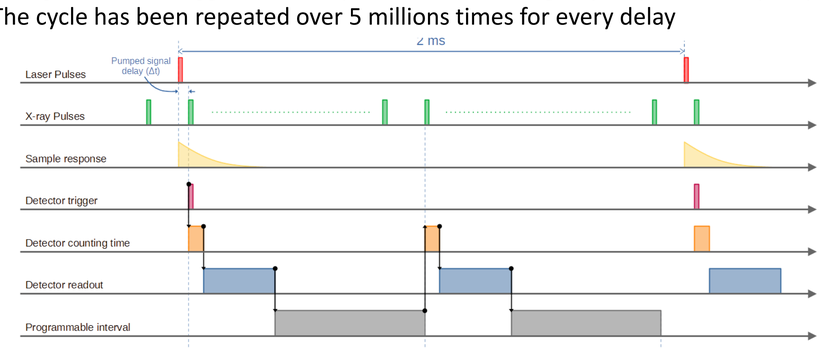
Pixel size	76 µm × 76 µm
Active area	77.8 mm × 38.9 mm
Number of pixels	1024 × 512 pixels (no inter-chips gaps)
Energy selection	two thresholds
Pixel counter depth	2 × 14 bits 1 × 28 bits (long counter mode, only one threshold)
Photons count rate	linear up to 2×10^6 cps/pixel
Max. framerate	56 kfps @ 2-bit/pixel (ZeroDead), 33 kfps @ 4-bit/pixel (ZeroDead) 17 kfps @ 8-bit/pixel (ZeroDead), 8.5 kfps @ 14-bit/pixel (ZeroDead) 970 kfps @2-bits/pixel (BurstMode, duty ratio 1.12%)
Min. counting time	~40 ns (multi-probe readout mode)

The pump-probe-probe measurements

Measurement principle

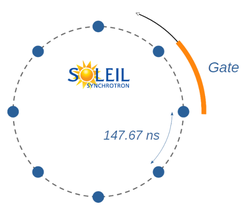
Chronogram of the pump-probe-probe measurements with XSPA detector:

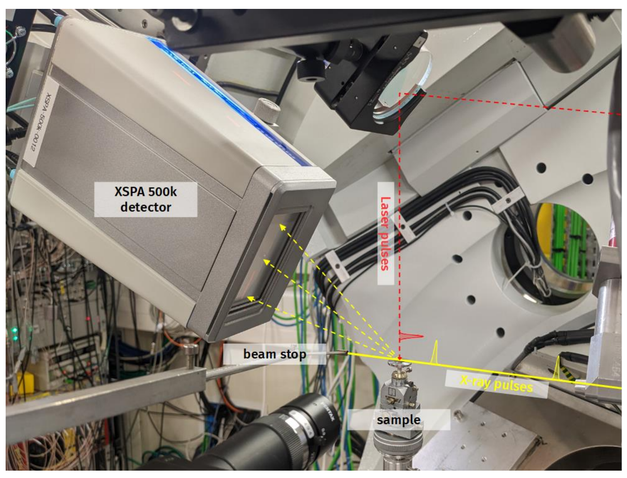
- A first image (pumped) is acquired at time Δt after the femtosecond laser pulse (sample excitation)
- A second image (unpumped) is acquired when the sample has returned to the relaxed state (1 ms after pumped image in the case of our experiment)
- A single external trigger starts acquisition of two images, pumped and unpumped
- The cycle has been repeated over 5 millions times for every delay



Experimental setup

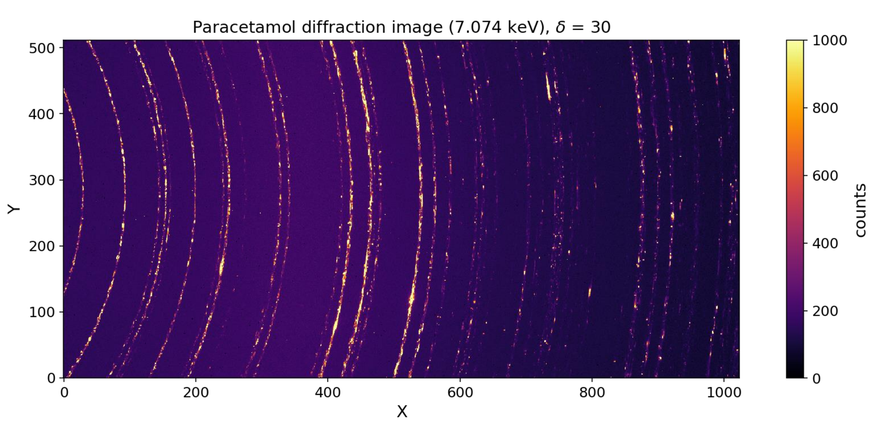
- X-ray energy : 7.078 keV
- Sample : Ti_3O_5
- Measurements done at four different pumped delays : -500 ps, 100 ps, 200 ps and 500 ps
- Machine filling mode : 8 bunch (bunch separation ≈147.67 ns)
- Detector gating time = 40 ns





Ti_3O_5 powder sample

Example of the diffraction image obtained from a reference paracetamol sample used for precise calibration of the detector-sample distance.

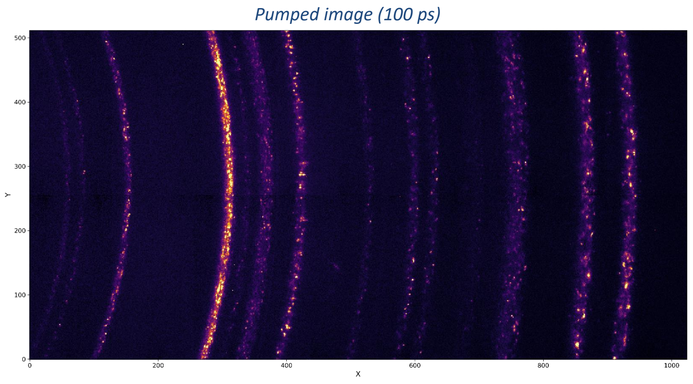


Very good uniformity of the image over a large area

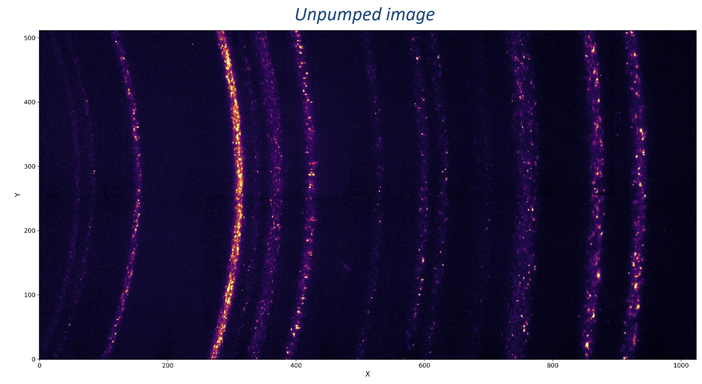
Results of the pump-probe-probe experiment

Example of the diffraction rings from Ti_3O_5 samples on the unpumped and pumped images. The pumped delay is 100 ps.

Pumped image (100 ps)



Unpumped image

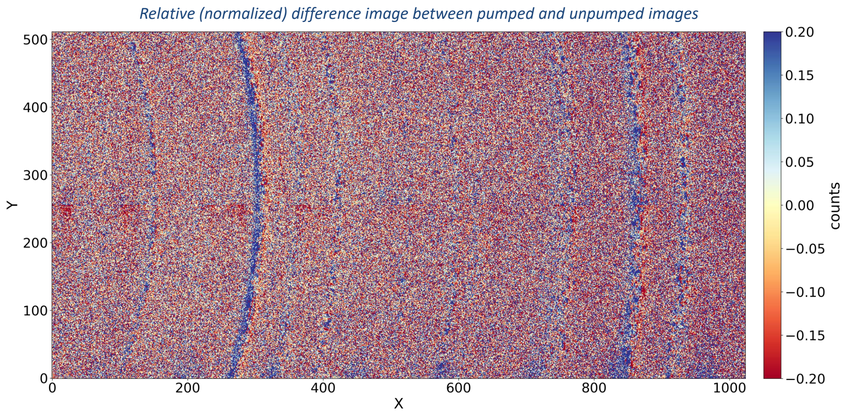


Relative (normalized) difference image between pumped and unpumped images

Relative difference image:

$$\frac{pumped_{\Delta t} - unpumped_{avg}}{unpumped_{avg}}$$

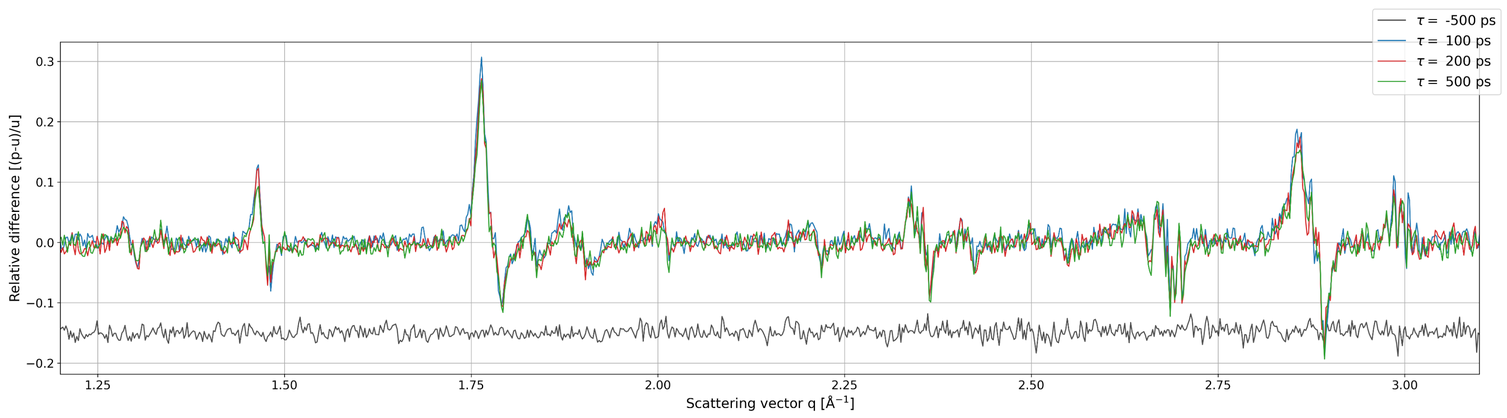
Where $pumped_{\Delta t}$ is pumped image at a given delay, and $unpumped_{avg}$ is the averaged unpumped image (from all tested delays)



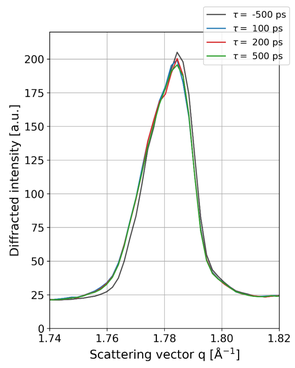
Visible background inhomogeneities are due to the threshold set to 5 keV (>70% of the incident energy) in order to avoid Ti fluorescence from the sample (4.5 keV)

Structural changes in the sample after laser excitation appear as shifts in the diffraction rings visible in the blue and red regions of the image

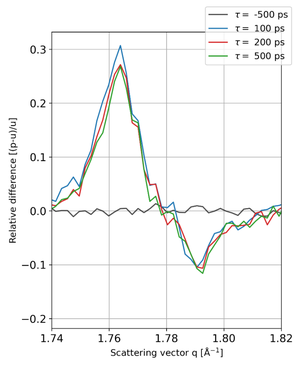
Relative diffraction diagrams after azimuthal integration for several laser delays



Intensity profile of the strongest diffraction peak



Relative difference profile of the strongest diffraction peak



The plot at -500 ps has been shifted manually on the Y-axis better visibility

Within the tested pumped delay range (0 ps to 500 ps), the sample's response is quasi-identical (no relaxation phase observed)

The measurements confirm that the pump-probe-probe acquisition mode is implemented efficiently in the detector, as required for our experiment

Conclusions

- Very good performance of the detector in gated mode operation (gate = 40 ns), no issues in separating signal from 8-bunch filling mode at SOLEIL (147.67 ns bunch separation)
- Very good imaging performance of the detector with the seamless pixel array
- Successfully performed pump-probe-probe measurements with Ti3O5 sample, the structural response of the sample was monitored with two consecutive diffraction images (pumped and unpumped) demonstrating correct implementation and operation of this mode in the XSPA detectors

[1] Y. Nakaye, T. Sakumura, Y. Sakuma, S. Mikusu, A. Dawiec, F. Orsini, P. Grybos, R. Szczygiel, P. Maj, et al., Characterization and performance evaluation of the XSPA-500k detector using synchrotron radiation, J. Synchrotron Radiat. 28, International Union of Crystallography, (2021).
[2] P. Grybos, P. Kmon, P. Maj and R. Szczygiel, 32k Channel Readout IC for Single Photon Counting Pixel Detectors with Pitch, Dead Time of 85 ns, Offset Spread and 2% rms Gain Spread, IEEE Trans. Nucl. Sci. 63, (2016) 1155.
[3] D. Bachiller-Perea, Y.-M. Abiven, J. Bisou, P. Fertey, P. Grybos, A. Jarnac, B. Kanouté, A. Koziol, F. Langlois, et al., First pump–probe–probe hard X-ray diffraction experiments with a 2D hybrid pixel detector developed at the SOLEIL synchrotron, J. Synchrotron Radiat. 27, International Union of Crystallography, (2020) 340.