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XSTRIP—a silicon microstrip-based X-ray detector for ultra-fast X-ray spectroscopy studies

Jon Headspith[a,*], G. Salvini[a], S.L. Thomas[b], G. Derbyshire[b], A. Dent[a], T. Rayment[c], J. Evans[d], R. Farrow[a], C. Anderson[a], J. Cliche[b], B.R. Dobson[a]

[a] *CLRC Daresbury Laboratory, Daresbury, Warrington WA44AD, UK*
[b] *CLRC Rutherford Appleton Laboratory,Chilton, Didcot, Oxon OX11 0QX, UK*
[c] *University of Cambridge, Cambridge CB2 1TN, UK*
[d] *University of Southampton. Highfield, Southampton SO17 1BJ, UK*

Abstract

For a number of years, an exciting and important area of synchrotron radiation science has been X-ray absorption spectroscopy fine structure studies of dynamically changing samples on the sub-second time-scales. By utilizing this technique, precise measurement of detailed structural changes can be investigated during a chemical or phase change reaction without the need for repeated experiments or expensive stopped flow techniques. Until recently, instrumentation to facilitate these studies has been based on commercially available detectors developed predominantly for other applications. Whilst these systems have yielded quality science, they have been subject to a number of fundamental limitations, particularly their speed, linearity and dynamic range. We have developed a new detector, XSTRIP, to overcome some of these. This new instrument marries dedicated silicon microstrip technology with specialist low noise, custom developed, fast readout integrated circuits, to yield an instrument that will unlock whole new areas of science to researchers. This paper will discuss some of the drawbacks of historical systems, give details of the XSTRIP system and also present the operating parameters of the system. In addition, some of the initial scientific experimental results will also be presented.

1. XAFS theory

X-ray absorption spectroscopy (XAS) studies are generally made in the range 200–35,000 eV. In this energy range, at and above a threshold energy characteristic of the atomic number of the target atom, the incoming X-ray photon is absorbed, ejecting a core photoelectron from the absorbing atom leaving behind a core hole. This photoelectron is ejected with an energy equal to the energy of the incoming photon less the binding energy of the electron. The outgoing photoelectron can be considered as a spherical wave and will interact with the electrons surrounding neighboring atoms, which will reflect some of the photoelectron wave back to the absorbing centre. The outgoing and reflected waves interfere at the absorbing atom, modifying the absorption cross-section. As the incoming X-ray energy is increased, the interference changes, leading to oscillations of the

*Corresponding author. Tel.: +44-1925-603624; fax: +44-1925-603618.
E-mail address: j.headspith@dl.ac.uk (J. Headspith).

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X-ray cross-section with energy. These oscillations gradually die away, but can extend to over 1000 eV above the absorption threshold and so are called extended X-ray absorption fine structure (EXAFS).

The electron wave back-scattering amplitudes and phase are dependent on the atomic number of the neighboring atoms, their coordination and the distance they are from the central atom, hence information regarding the co-ordination environment of the absorbing atom can be obtained by analyzing the EXAFS.

2. Time resolved EXAFS

In conventional X-ray spectroscopy experiments, the minimum time to record a spectrum is usually a few minutes, mainly determined by the speed at which it is possible to scan a monochromator. However, since many, if not most, chemical reactions take place over a much shorter time-scale and frequently involve short-lived intermediate species, it is not possible to follow the structural evolution of reactions in real time.

The goal of time-resolved XAS is to determine the structure of these transient molecular species. An effective way to begin to achieve this on a time-scale of 10^{-3}–10^{-6} s is to measure the whole spectrum simultaneously via the technique of energy-dispersive EXAFS (EDE). In this technique, the sample is irradiated with a range of X-ray energies linearly dispersed in angle and focused on the sample. If the X-rays transmitted by the sample are then measured with a position sensitive detector placed behind the sample and after the focused beam has linearly dispersed (see Fig. 1), it is possible to record the entire absorption spectrum in a single shot.

Station 9.3 on the Synchrotron Radiation Source (SRS) at Daresbury Laboratory was designed with an EDE facility and has been used regularly for some years now. The original detector systems we used were based upon commercially available photodiode arrays (PDA) with custom read-out electronics that offer modest speed and linearity [1–4]. These systems have been developed over a 10-year period, culminating in the PDA system at the SRS, which can record data with 5–100 ms time resolution. This PDA system suffers low efficiency, it is a single low-resistivity die solution, has a high susceptibility to radiation damage coupled with high dark current, low readout speed, limited well depth and poor linearity. In addition the reset and read-out electronics were not ideal.

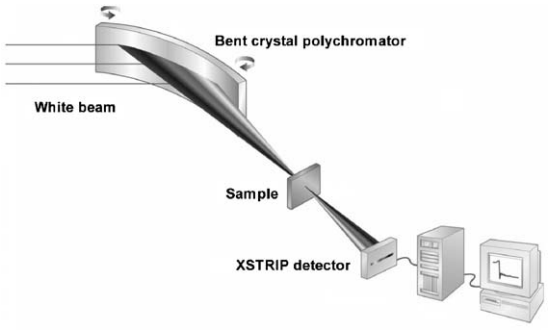

Fig. 1. The layout of a typical EDE experiment.

An alternative approach for an EDE detector is the CCD system in use at the European Synchrotron Radiation Facility (ESRF) in Grenoble, which can collect data quickly (100 μs). However, the poor duty cycle, low X-ray efficiency and limited linearity of the ESRF system still make it possible to collect better quality data at the SRS using the PDA system.

Despite these early efforts, it was apparent that the vast majority of chemical reactions lay beyond the capability of existing detectors: obviously structural reaction studies required a new generation of detectors.

3. Prototype XSTRIP

We were able to obtain internal funding to draw on the background within CLRC in particle physics instrumentation and in collaboration with Imperial College, London, to develop a silicon microstrip based system [5].

A custom detector was designed and interfaced to existing hardware present within CLRC which had been used previously for high energy physics experiments (see Fig. 2).

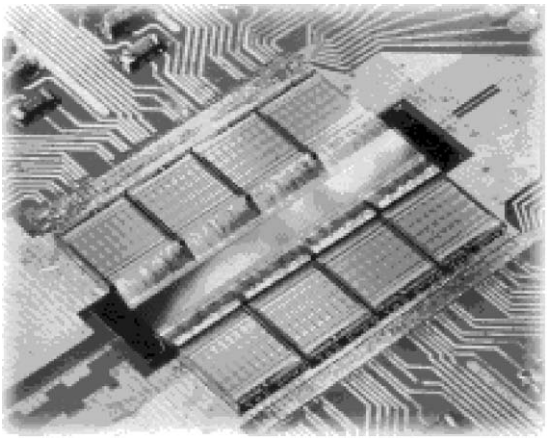

Fig. 2. The prototype multi-chip module.

Initial testing of the prototype proved successful and the system was tested on station 9.3 at the SRS.

A few problems became apparent with the prototype: thermal stability, charge latency and linearity issues meant that it was unsuitable for quantitative measurements, but it did prove the principle of using silicon microstrip detectors and we could envisage ways to overcome the remaining problems.

4. XSTRIP description

After successful testing of the prototype a full development program was initiated (EPSRC GR/M63751, GR/M58627 & GR/M55825). The detector system was stringently specified to allow the system to be capable of handling the flux not only from the SRS, but also from much more intense third generation sources such as the ESRF.

The important features of the specification are detailed in Table 1.

4.1. The system

The system was designed where possible to take advantage of commercially available data acquisition equipment. As such it was decided to base the architecture of the data acquisition system on the PC platform.

Table 1
Specification parameters for the XSTRIP system

Useable energy range	5–25 keV
Minimum integration time	10 μs
Maximum integration time	1 s
Integral non-linearity	<0.2%
System deadtime per integration	1 μs
Efficiency[a] of the entire system at 15 keV	>50%
Dark current contribution per integration	<10% of full dynamic range
Maximum charge handled per integration	10 pC

[a] Efficiency of the entire system takes into account all loss components, the Si detector DQE, transmission of the cryostat Be window and the system live time.

4.2. Detector

The detector is fabricated from 500 μm thick high-resistivity silicon. It contains 1024 diodes arranged in a one-dimensional array. The diodes are 4 mm high and 15 μm wide, spaced at 25 μm intervals and are bonded out alternately top and bottom of the array to on a 50 μm bond pitch.

The detector was fabricated by CSNV Belgium to a CLRC specification.

4.3. Detector readout chip, XCHIP

The XCHIP is a 0.5 μm, full custom, mixed-signal ASIC containing 128 charge integrating preamplifiers with the ability to sample and hold the preamplifier output voltage. The stored voltages are read out sequentially, via multiplexers. The chip is divided into blocks of 64 channels whose integration time can be set independently. Each of these blocks is further subdivided into two blocks of 32 channels, each with its own multiplexer, to enable readout speed specifications to be met. For each channel the output of the integrating amplifier is sampled at the beginning and end of the integration period on one pair of capacitors. At the end of the integration period the voltages stored on these capacitors are readout via the multiplexer, while another integration sampling voltages onto a third storage capacitor starts a new cycle. The XCHIP was been tested on the

bench and achieved a non-linearity of 0.035%. During bench testing it was discovered that if the supply voltage was raised to 4 V, the non-linearity could be improved to 0.0036%. The reasons for this improvement in linearity are still being investigated.

4.4. Detector head

The detector and readout chips are mounted onto a ceramic multi-chip module that provides the detector head with a high thermal conductivity substrate allowing efficient cooling to reduce the leakage (dark) current. Fig. 3 shows a photo of the detector and bonded XCHIPS. The detector head and local Peltier thermo-electric coolers are mounted within a vacuum cryostat chamber. The vessel was designed to be compatible with a moderate vacuum (10^{-4} mbar). However, trials of the system have demonstrated that back-filling the vessel with nitrogen gas at atmospheric pressure provides an adequate thermal barrier when the detector is cooled to -30°C. This has a number of advantages: the system only requires electrical power and no cryogenic liquids are required.

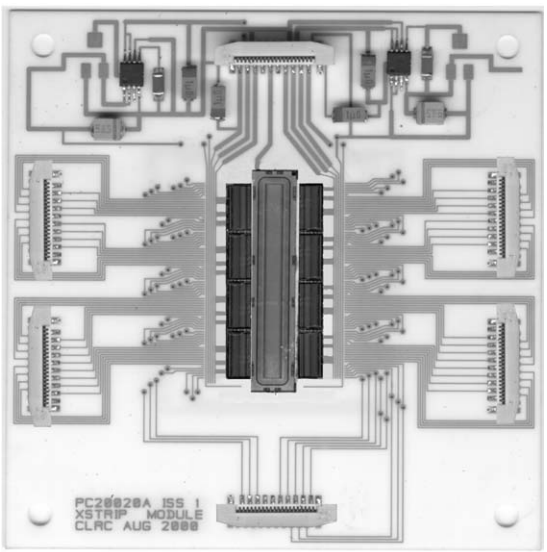

Fig. 3. A photograph of the multi-chip module, showing the central microstrip detector bonded out to the surrounding XCHIP devices.

4.5. Signal transmission system

The high linearity output signals of the XCHIP are transmitted to the PC-based data acquisition (DAQ) system by a high linearity, differential, analogue signaling system. This transmission system allows the use of a 2 m cable length between the detector and the PC DAQ system and hence simplifies detector and equipment placement.

4.5.1. Data acquisition system

The DAQ system hardware (provided by Sundance Multiprocessor Technology Ltd) comprises two PCI carrier cards, each card containing 16 14bit, 5 MHz, analogue to digital converters (ADC), one 400,000-gate field programmable gate array (FPGA), 8 Mbytes of fast static ram (SRAM) and one fast digital signal processor (DSP). The ADCs are used to convert the 32 channels of incoming analogue signals into digital data streams. The data streams are then handled by the two FPGAs, which execute the accumulation and data framing algorithms, developed by CLRC. A DSP processor and a set of libraries provided by 3L Ltd handle communication with the host processor.

4.6. Graphical user interface

The user interface has been written in Visual Basic® and makes use of available plug-in components where possible. The user interface has a remote TCP/IP interface to provide simple implementation to other station computing systems and to permit the data collected using XSTRIP to be archived according to the normal SRS protocols.

4.7. Detector power supply

The detector power supply has been designed for low noise and safety. In the event of unforeseen electrical problems a safe shutdown is initiated. One of the main features of the power supply is to ensure that the detector bias voltage is only applied when the power to the XCHIPs has been applied.

4.8. XSTRIP timing system

For time-resolved studies issues of timing are of paramount importance not only for control of the detector but also to allow co-ordination with other equipment. Furthermore, for the fastest experiments the whole experimental system has to be phase locked to the pulse structure of the synchrotron itself. Getting this timing wrong can produce up to 20% errors in the data and thereby render the data utterly useless. The timing card produces all of the timing pulses for the experiment and makes available input and output control signals for users' equipment.

5. XSTRIP parameters

The data acquisition system specification is summarized in Table 2.

This gives the system the flexibility and the memory depth to handle today's dynamic EXAFS experiments. The system has been tested and meets all its design requirements.

6. Test results

The detector was initially tested on station 9.3 at the SRS, when we recorded a spectrum of platinum foil on the first day of the commissioning time. Once initial commissioning was completed, we were able to demonstrate that it was possible to discern an absorption edge in a single 90 μs scan. Moreover, it was feasible to record data of analyzable quality in 350 μs. Due to the extremely good linearity, measured at 0.03%, and low dark current of the micro-strip, measured at 132 ADU/s, it was clear that, at the SRS, the signal is simply photon flux limited.

Table 2
Summary of the design specification for the XSTRIP data acquisition system

Parameter	Value
Readout time for the entire array of 1024 pixels	10 μs
Integration times available	1 μs–1 s
Accumulated readouts available per frame[a]	1–1,000,000
Storage space for	1700 frames

[a] A frame is an individual readout, or pixel by pixel accumulation of readouts which are stored to system memory as one discrete data set.

Fig. 4 shows a comparison of the best data previously available using the PDA detector and data collected using XSTRIP for a 5 μm nickel foil. The nickel foil data shows that data from a 600 μs scan with XSTRIP are substantially better than a 2 ms scan using the PDA. This indicates that XSTRIP offers not only a factor of 50 increase in speed over the photodiode array, but also a substantial increase in the data quality for comparable integration times.

These initial comparisons are encouraging, but further measurements are required, since it transpires the stability of the X-ray beam was somewhat better for the second set of beam time. Whatever the outcome of these studies it is remarkable that a recognizable nickel spectrum can be collected in 60 μs.

Many samples that researchers wish to study are considerably dilute; thus the performance of the system with such samples was of great interest. Fig. 5 shows data collected using a benchmark standard solution of 100mM Ni^{2+} in water (typical of concentrations used in stopped flow kinetics experiments) for a variety of different integration times. There is a similar level of improvement compared to similar data [6,7]. The data show that it is perfectly feasible now to record

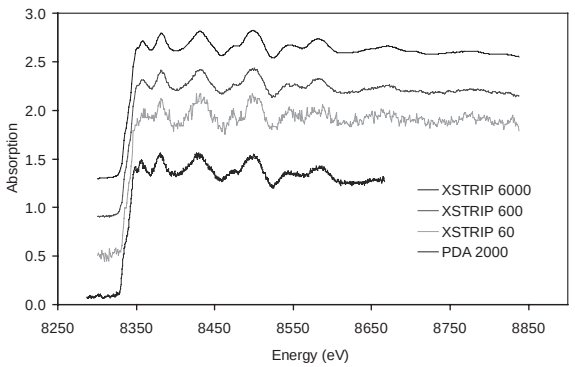

Fig. 4. A comparison of spectra from 5 μm Ni foil recorded with XSTRIP at varying integration times (from top downwards, 6000, 600, 60 μs) with data from the PDA system (bottom trace, 2000 μs).

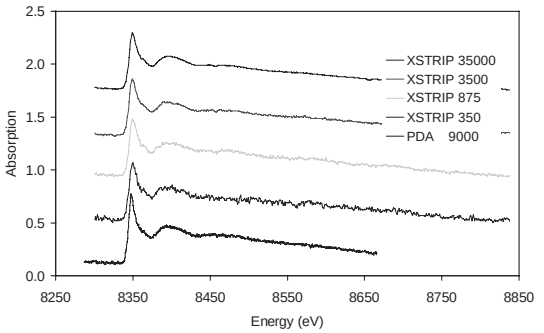

Fig. 5. A comparison of spectra from 100mM Ni^{2+} in water recorded with XSTRIP at varying integration times (from top downwards, 35,000, 3500, 875, 350 μs) with data from the PDA system (bottom trace, 9000 μs).

spectra in 350 μs, which can be quantitatively interpreted with regard to the edge height and specific features on the absorption profile. (The small sharp dips apparent in all the spectra are not due to the detector but due to a feature of the monochromator.) It is clear that if an experiment is designed such that it may be cycled repetitively to improve statistics, then the way is open for experiment in the 10 μs regime even on a second-generation source such as the SRS.

The initial tests during the commissioning of the system have been followed by runs by a number of research groups. The success of these experiments has now made XSTRIP the detector of choice for EDE experiments performed on station 9.3 at the SRS.

7. Future

Whilst the XSTRIP system is in use for research studies, work continues to fully optimize the system. Also, the quality of the detector system has highlighted shortcomings in other aspects of the experimental set-up which are also being addressed.

Although the current XSTRIP detector has not yet been utilized to its fullest capabilities, there is already a scientific push to enhance the system. We can envisage a system with increased linearity and readout speed.

8. Conclusion

A silicon microstrip-based detector system for time-resolved XAFS measurements has been designed, built and tested. The system demonstrates a significant improvement in speed: up to 100 times faster in some circumstances. The improved linearity of the read-out electronics has also improved the data quality compared to previous detector systems. This detector system will allow new experiments at the SRS and at more modern, more intense synchrotron sources.

Acknowledgements

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