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Improve the spatial resolution of a soft X-ray Charge Coupled Device used for Resonant Inelastic X-ray Scattering


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ABSTRACT: The Super Advanced X-ray Emission Spectrometer (SAXES) at the Advanced Resonant Scattering (ADRESS) beamline of the Swiss Light Source is a high-resolution X-ray spectrometer used as an end station for Resonant Inelastic X-ray Scattering from 400 eV to 1600 eV. Through the dispersion of photons across a CCD, the energy of scattered photons may be determined by their detected spatial position. The limiting factor of the energy resolution is currently the spatial resolution achieved with the CCD, reported at 24 µm FWHM.

For this energy range the electron clouds are formed by interactions in the ‘field free’ region of the back-illuminated CCD. These clouds diffuse in all directions whilst being attracted to the electrodes, leading to events that are made up of signals in multiple pixels. The spreading of the charge allows centroiding techniques to be used to improve the CCD spatial resolution and therefore improve the energy resolution of SAXES.

The PolLux microscopy beamline at the SLS produces an X-ray beam with a diameter of 20 nm. The images produced from scanning the narrow beam across CCD pixels (13.5 × 13.5 µm²) can aid in the production of event recognition algorithms, allowing the matching of event profiles to photon interactions in a specific region of a pixel. Through the use of this information software analysis can be refined with the aim of improving the energy resolution.

KEYWORDS: Data processing methods; Solid state detectors; X-ray detectors; Spectrometers

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1 Introduction

The Advanced Resonant Spectroscopies (ADRESS) beamline is a high resolution soft X-ray beamline at the Swiss Light Source [1]. The beamline has been optimized for a Resonant Inelastic X-ray Scattering (RIXS) spectrometer, the Super Advanced X-ray Emission Spectrometer (SAXES) [2]. The instrument is capable of investigating excitations lower than 0.1 eV at incident energies between 400 eV and 1600 eV with low count rates, leading to a design that balances the flux with a high energy resolution. The work presented here continues the study devoted to improving the energy resolving power.

2 Resonant Inelastic X-ray Scattering at the Swiss Light Source

RIXS is a photon-in photon-out technique used to investigate low energy electronic and magnetic excitations within materials [3]. The change in energy and momenta of scattered photons provides information about the chemical environment in the bulk of samples. X-rays at energies between 400 eV and 1600 eV can be delivered to samples in SAXES allowing the ingoing photon energy to be tuned to the resonance of a selected element to study its environment. The soft X-ray energy range is important for studying materials which contain transition metals or rare earth atoms, for instance present in material systems such as heavy fermion systems and superconductors [4, 5].
2.1 The Super Advanced X-ray Emission Spectrometer

SAXES was designed and built with the goal of achieving an energy resolution of \( \frac{E}{\Delta E} > 12000 \) at 930 eV [2]. A variable line spacing grating disperses X-rays across a Charge Coupled Device (CCD). The CCD is mounted at a grazing incidence of approximately 20° to increase the effective pixel density by a factor of approximately 2.9. The distance between the detector and sample is the maximum in the space available for the equipment at approximately 5 m. Images taken have interaction events from X-rays with equal energy dispersed in straight lines across the CCD. The total signal level along each isoenergetic line is summed to convert the two dimensional image into a spectrum.

3 Detector resolution

The current CCD in SAXES is an e2v technologies plc. back illuminated CCD42-40, with 2048 \( \times \) 2048 pixels, each 13.5 \( \mu \)m square. Supplied as part of a Princeton Instruments (Roper Scientific Inc.) special package [6], it is cooled using liquid nitrogen to \(-110^\circ\text{C}\) to suppress dark current generation. The spatial resolution has previously been shown to be 24 \( \mu \)m FWHM [2]. In an earlier study, Dinardo et al. [7] published spatial resolution measurements of thinned back illuminated CCDs (~16 \( \mu \)m detection thickness), with pixel sizes of 13.5 \( \mu \)m and 20 \( \mu \)m. In these cases it was shown that the spatial resolution was independent of the pixel size due to charge splitting.

3.1 Charge splitting

X-ray photons interacting in the active silicon of a CCD pixel produce a cloud of signal electrons. Furthest from the electrodes in the device there is a region with virtually no electric field — the ‘field free’ region. Electrons formed here diffuse in a stochastic and isotropic manner with most entering the depletion region and being attracted to their closest pixels. When interactions occur near a pixel boundary, or where the pixels are sufficiently small, the diffusion of the electrons form a distribution of signal across the nearby pixels. In SAXES the CCD has 13.5 \( \mu \)m square pixels and the majority of interactions by 400 eV to 1600 eV photons form events that appear in an image with signal split across multiple adjacent pixels.

3.2 Improved spatial resolution with centroiding

Due to the isotropic nature of the charge spreading in the field free region the spatial resolution of the CCD in SAXES is limited by the dimensions of the charge cloud rather than the dimensions of the pixel. Centroiding an isolated event can remove the charge spread contribution to the PSF resulting in an improved spatial resolution.

In order to apply centroiding algorithms to an image, X-ray events which are split over several pixels must not share pixels with other nearby X-ray events (i.e. pileup). The standard integration time of the SAXES CCD is 10 minutes during which time X-ray events in spectral lines pile up. The long integration time minimises the proportion of time during which the CCD is reading out, maximising the duty cycle of the spectrometer. The throughput of the spectrometer could not be maintained if the integration time was to be reduced sufficiently to allow the use of centroiding.
techniques. However, a high spatial resolution, short integration time mode incorporating a centroiding algorithm would be desirable. To formulate and integrate this algorithm an understanding of the accuracy of centroiding methods applied to events across the area of a pixel is required.

4 Probing the device at a sub-pixel level

PolLux is a soft X-ray microscopy beamline at the Swiss Light Source [8]. Monochromatic X-rays with energies between 200 eV and 1400 eV are focussed onto a sample with a spot size down to 20 nm in diameter using a Fresnel Zone Plate configuration. Sample stages with submicron translational movement relative to the beam allow scanning of the spot across the pixels of a device. A back-illuminated uncoated e2v CCD42-10 was investigated (2048 × 512 pixels, 13.5 µm square). The design of this device is the same as the e2v CCD42-40 currently used in SAXES, but the latter has a larger imaging area with more pixels.

An interfacing setup was made to place the device in the PolLux microscope, attaching it to the translational stages used for sample movement, figure 1. A CCD drive system from XCAM Ltd. [9] was used to supply input voltages and provide the analogue to digital signal processing, using a dual slope correlated double sampling routine. The CCD was cooled using a copper braid coupled to a Polycold PCC Compact Cooler to reduce dark current generation and operated between −10°C and −20°C.

Figure 1. Photograph of the setup interfacing in the PolLux experimental hutch.
5 Sub-pixel imaging

X-rays at 1000 eV were focussed to a spot size less than 0.1 µm. The setup and flux were adjusted to allow single photon counting with a noise of 4 to 6 electrons rms. The spot was tracked linearly along the centre of a row of the CCD42-10 pixels (13.5 µm square). The locations of the 13 positions a-m are shown in figure 2i. Images of split events due to X-ray interactions at these positions were taken.

The signal in the pixels around which the spot was focussed is averaged across all frames for the first position (a), shown in figure 2ii. The pixel signals from row 2 are shown for position a in figure 2iii alongside the mean signals from the same pixels when the beam was focussed at the other positions (b-m). The pixel with highest signal shifts from column 4 to 2 as the beam spot is moved by an average of approximately 1.4 µm in each step. The signal in column 4 decreases as the distance to the X-ray interaction location increases. At position f the spot is focussed close to the centre of the pixel. At this location, signal charge is still seen in neighbouring pixels, confirming that the PSF of the charge cloud is larger than the pixel width (13.5 µm).

Figure 2. i: positions (a-m) in the pixels at which the X-ray spot was focussed. ii: the average signals from the currently used “integrating mode” from the pixels around the 1000 eV X-ray interaction position for spot position a. iii: the signal from pixels shown in i as the 1000 eV spot position was moved across the pixels from slices a-m. Signal is seen to spread from the main pixel into the adjacent pixels. iv: the image profile accumulated in “photon counting mode” for each spot position after the X-ray events have been centred using a simple “centre of gravity” algorithm. The centre of the spread moves across the pixels with the spot position and the photon counting mode demonstrates greatly improved resolution.
Each image with an event containing the signal from a single photon interaction was centroided. A centre of mass algorithm across an area of 9 pixels around the spot (3 × 3 pixels) was used, after the zero-level offset and background were removed. Histograms of the resulting locations are shown in the figure 2iv. The mean centroid location follows the stepped scanning pattern expected from the mean signals in figure 2iii.

The spread of the centroided locations (figure 2iv) is determined through the fitting of a Gaussian profile. The results for 1000 eV are shown in the left panel of figure 3. The spatial resolution is best when the interaction occurs close to the centre of the pixel. The trend continues when the spot scans into the next pixel at positions k, l and m.

The spread of the centroided locations was determined for each spot position in the energy data sets. The mean FWHM of the Gaussian profile fits is shown in right panel of figure 3 for each energy. As the X-ray energy increases the spatial resolution from centroiding accuracy improves. A proposed cause is the improved signal to noise ratio from higher energy photons in the outer pixels of a split event.

**Figure 3.** *Left Panel*: the binned centroid locations are fit by a Gaussian profile to determine a spatial resolution for each position across the pixel at 1000 eV. The approximate edges of the main pixel are shown. *Right Panel*: the mean spatial resolution from centroiding interactions at positions across the pixel. Improved resolution at higher energy is due to the higher signal to noise ratio.
6 Conclusions

The PolLux microscopy beamline has been used to focus soft X-rays to a spot size less than 0.1 µm and scanned across the 13.5 µm square pixels of a CCD42-10 (a smaller area version of the CCD42-40 currently used in SAXES). Single photon split events have been centroided using a centre of mass style algorithm improving the spatial resolution for interactions in the centre of the pixel to an average of 4.7 µm, 4.6 µm, 3.7 µm and 3.0 µm FWHM for photons of energy 530 eV, 680 eV, 850 eV and 1000 eV respectively. This is over a 5 fold increase from the electron cloud size limitation of 24 µm FWHM previously reported.

Based on these measurements it can be predicted that the spatial resolution of the CCD of SAXES could be improved to below 5 µm FWHM for isoenergetic RIXS spectral lines across its energy range of 400 eV to 1600 eV. A higher energy resolution mode of operation for SAXES will prove a useful tool for users to investigate systems with RIXS.

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