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Nuclear Instruments and Methods in Physics Research A 570 (2007) 176–181

NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH
Section A

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Gaining efficiency and resolution in soft X-ray emission spectrometers thanks to directly illuminated CCD detectors

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Received 5 September 2006; received in revised form 3 October 2006; accepted 5 October 2006
Available online 7 November 2006

Abstract

The back-illuminated charge coupled devices (CCD) are suitable for soft X-ray photon detection. Their nominal performances suggest that they can boost both efficiency and resolving power of X-ray spectrometers based on diffraction gratings and two-dimensional position sensitive detectors. We tested the performances of two commercially available CCDs, intended to replace a more traditional microchannel plate (MCP) detector. Our tests show that the devices have excellent performances in terms of dark current, response linearity, detection efficiency and spatial resolution. We observed that the CCDs have better efficiency (more than 10 times) and better resolution (~3 times) than the MCP. Moreover we found an intrinsic limit for the spatial resolution, which is almost independent of the detector pixel size and is estimated around 25 μm.

PACS: 78.70.En; 07.85.Nc; 07.85.Qe; 07.85.Fv

Keywords: X-ray emission spectroscopy; CCD detector; X-ray spectrometer

1. Introduction

The recent advances of synchrotron radiation sources have opened the way to new X-ray based spectroscopic techniques of high interest for the study of electronic states in solids and molecules. Among those techniques resonant inelastic X-ray scattering (RIXS) and X-ray emission spectroscopy (XES) [1–5] in the soft X-ray range (30–1500 eV) are extremely photon hungry due to the intrinsic low cross-section of the process and to the unavoidable limitations in the angular acceptance of the instrumentation. In fact soft X-ray RIXS and XES are measured with spectrometers based on grazing incidence (2–4°) gratings ruled on X-ray quality mirrors. In order to achieve high energy resolution the source of those optical elements, which is the spot illuminated on the sample, has to be as small as possible (5–30 μm), the footprint on the grating cannot be too extended (30–70 mm) to avoid excessive optical aberrations, and the detector has to provide a good spatial resolution. In Fig. 1 we show the optical scheme of our spectrometer, called advanced X-ray emission spectrograph (AXES) [6], currently installed at the beam line ID08 of the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The general layout is shared by the majority of the soft X-ray spectrometers [7–10]: the radiation coming from the sample (illuminated by monochromatic X-ray beam) is dispersed in energy by the grating and a two-dimensional position sensitive detector allows the parallel acquisition of the emitted spectrum during periods of time of tens to hundreds of seconds. In some cases [10] one or two mirrors are also used to refocus the radiation before the detector, but this detail does not change the way the detector is used. During the acquisition everything remains fixed, included the incident photon energy. The ultimate resolving power of the spectrometer is thus dictated by the source size along the dispersion direction (in many cases it is determined by

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doi:10.1016/j.nima.2006.10.024

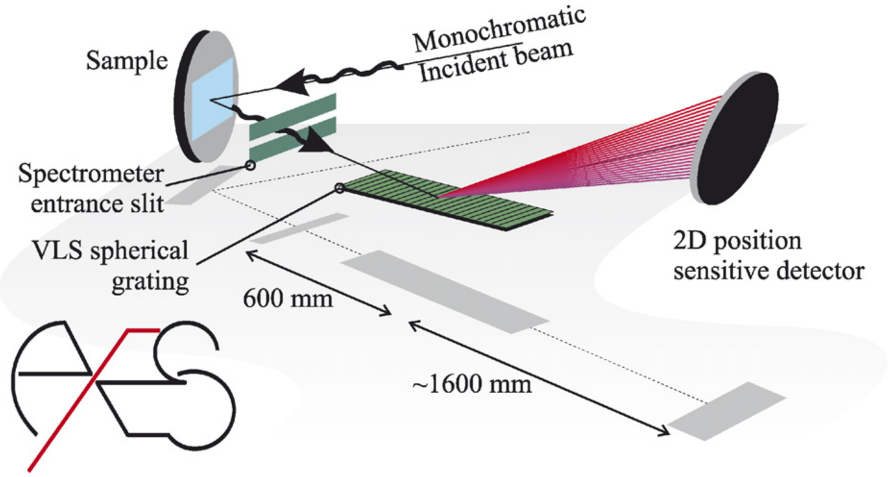

Fig. 1. Schematic representation of the AXES soft X-ray spectrometer, based on a variable line spacing (VLS) spherical grating and a two-dimensional position sensitive detector. The angle between the incident beam and the spectrometer optical axis is 70°. The sample is 20–30 mm before the entrance slit. The entrance slit width can be regulated between 5 and 100 μm.

an adjustable width entrance slit), by the grating groove density and surface quality, by the distance of the detector from the source and by the effective spatial resolution of the detector. On the other hand, often a reduction of the intensity reaching the detector is the price to pay for improving the energy resolution: for instance a smaller entrance slit and a higher grating groove density increase the resolving power but entail an intensity loss.

With the given constraints, the spectacular increase of the useful flux on the sample provided by the modern beam lines and the development of new two-dimensional detectors with high efficiency and high spatial resolution have been the major steps towards high resolution RIXS and XES. In fact more brilliant synchrotron sources allow to better focus the highly monochromatic incident beam onto the sample surface, providing an intrinsically small source to the X-ray spectrometer. The small-pixel/big-size detectors allow to spatially resolve photons having closer energies without extending excessively the spectrometer exit arm [11,12]. We present here the remarkable improvement in the performances of our spectrometer obtained by replacing the original detector (triple micro-channel plate with resisitive anode encoder) with a directly illuminated charge coupled device (CCD) specially built for soft X-ray applications in high vacuum environment.

We present results of two commercial back-illuminated Si wafer CCD cameras by Roper Scientific: Princeton PI-SX 1300 (CCD1) and Princeton PI-SX 2 k (CCD2), with 20 and 13.5 μm pixel size, 1300×1340 pixels ($26.0 \times 26.8\,\mathrm{mm}^2$) and 512×2048 pixels ($6.9 \times 27.6\,\mathrm{mm}^2$) active area, respectively [13]. For both detectors we performed preliminary tests on the dark-current level, on the response linearity and on the spatial resolution when detecting soft X-rays. We optimized the procedure to pass from the raw images to the RIXS/XES spectra and we estimated the actual gain in efficiency and resolving power coming from the new detectors. Having taken advantage also from other modifications of the dedicated monochromator [14], the AXES spectrometer is now working with a measured combined resolving power better than 1500 from TiL$_3$ ($E = 460\,\mathrm{eV}$, $\Delta E = 0.250\,\mathrm{eV}$) to CuL$_3$ ($E = 930\,\mathrm{eV}$, $\Delta E = 0.62\,\mathrm{eV}$), with the best measured performances at the MnL$_{2,3}$ ($E = 640\,\mathrm{eV}$, $\Delta E = 0.32\,\mathrm{eV}$). These performances are obtained with an entrance slit of 10 μm, the original variable line spacing spherical grating (2400 grooves/mm), entrance arm 600 mm, average exit arm ~1600 mm, CCD2 detector cooled at −70 °C and mounted at 45° incidence. In Fig. 2 we show typical images obtained in 300 s with AXES: we can recognize the iso-energetic horizontal lines and, in the blowups, the white spots corresponding to single photons.

A crucial point in all the work presented in this publication is the use of commercial CCD detectors, although designed and built for soft X-ray applications. We will not take into consideration special devices developed for astronomy or advanced projects, whose price is currently at least an order of magnitude higher than that of our CCDs. Thus we will make no specific references to the vast literature on X-ray CCDs [15–21], because our point of view is that of users interested in the optimal use of CCD detectors in the field of soft X-ray spectroscopy at the synchrotron radiation facilities. We are not going to discuss the physics and the technology of CCD devices per se.

2. Preliminary tests

Before installing the CCD cameras on the AXES spectrometer, we made offline tests of dark current and response linearity in order to check for the effective performances and compliance with our requirements.

In our case both cameras are cooled with a cascade of several Peltier cells using forced air as hot sink, and the temperature is kept stable with a precision of ±0.05 °C.