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LINEAR DETECTOR FOR TIME-RESOLVED EXAFS IN DISPERSIVE MODE

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ABSTRACT

A fast read-out system for a linear photodiode detector has been developed for a time-resolved EXAFS experiment in dispersive mode. A green light-emitting phosphor (Gd_2O_2:Tb) is deposited on a fiber optic faceplate, which is optically coupled with a linear photodiode array with 1024 sensor elements. A 12-bit fast A/D converter is used to process 1024 channel data in 5 msec and store them in memory. Digital data can be routed and stored in a histogram memory having 16 frames with 20 bits. 16 spectra can be measured successively with a time resolution of 5 msec and a variable interval time. An energy resolution of the total system is 5.6 eV at the Fe K-edge for 1200 eV energy range. The application to a kinetic study demonstrates that time-resolved EXAFS or near-edge spectra can be obtained with a time resolution of 25 msec by use of the present detector system.

INTRODUCTION

The development of a fast linear detector with a high spatial resolution is essential in EXAFS experiments in dispersive mode [1] where X-ray absorption spectra are measured as a spatial distribution of dispersed X-ray beam intensity. Since this arrangement is suitable for a time-resolved EXAFS experiment, efforts have been taken to utilize a linear photodiode array as a position-sensitive detector in order to achieve a rapid measurement of transmitted intensity [2]. Phizackerley et al. has used a thin phosphor (YVO_4:Eu) to convert incident X-rays into visible photons to avoid X-ray damage to the silicon p-n junction [2].

Although a linear photodiode array has been recognized as a promising position-sensitive detector with a high spatial resolution (50 μm), the time resolution has been limited by a slow data acquisition process of a read-out circuit which was originally developed for a low count-rate use in X-ray astronomy [3]. Therefore, a fast read-out circuit for a linear photodiode array has been developed for a time-resolved EXAFS experiment. In this paper, the design features and performance of a fast linear detector system are presented.

LINEAR DETECTOR SYSTEM

A linear photodiode array (RETICON RL1024SF) with 1024 sensor elements separated with each other by 25 μm was used. Figure 1 shows the schematic representation of a linear detector. A green light-emitting phosphor (Gd_2O_2:Tb) was uniformly deposited on a fiber optic faceplate, which is optically coupled with sensor elements. The use of phosphor has eliminated the two problems with a direct exposure of photodiode to X-rays, i.e., the rapidly...
decreasing sensitivity in the high energy region (>5 keV) and a radiation damage [4]. Although the radiation damage can be reduced at low temperatures (-90-100 °C) [5], the mechanism of radiation damage is not clear yet.

Special care has been taken to obtain a fine powder of phosher, which was prepared by sedimentation. The thickness of phosher is estimated as about 50-70 μm. The active area of photodiode array is 2.5 mm high and 25.6 mm wide. The spatial resolution of a photodiode array is about 50 μm (2 pixels) for a direct illumination. However, considering the fact that a diameter of optical fiber is 6 μm and the numerical aperture is 1, or incoming visible photons are accepted within an aperture of 45 degrees, an optimum thickness of phosher is estimated to be about 75 μm.

The thermal noise of photodiode can be reduced by cooling. The detector is mounted on a copper block which can be cooled down to -55 °C by a thermoelectric cooler but the noise level at -20 °C is already comparable to that of preamplifier noise. The detector housing with a Kapton window is either evacuated by a mechanical pump or filled with dry nitrogen gas.

Figure 2 shows the block diagram of a fast read-out system. The four-phase clock to operate a linear photodiode array, preamplifiers, and analog multiplexer circuits are installed within the detector housing. The four video lines sampling the output of every fourth pixel are fed into charge-sensitive amplifiers which produce the pulse proportional to the charge depleted in the photodiode p-n junction. Since the overall data acquisition time is often determined by the A/D conversion rate, a fast 12-bit A/D converter (5 μsec conversion) is used to process 1024 channel data in 5 msec. The output signal is digitized and stored in a histogram memory.

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**Fig. 1** Schematic diagram of a linear detector.

**Fig. 2** Block diagram of the fast read-out system for a photodiode array.
Digital data can be routed into 16 frames. Each frame memory consists of 1024 channels with 20 bits. The exposure time and integration number as well as interval time between the frames can be chosen between 1 msec and 999 sec. The entire read-out circuit is controlled by a hardware logic interfaced with a microcomputer (LSI-11/23). The microcomputer system is equipped with a hard disk (20 MB) as well as two floppy disks (2MB) and a main memory of 64 KB with a floating processor. As peripherals, a graphic display with a hardcopy unit and a printer are connected to the microprocessor. The data acquisition software is written in FORTRAN language under RT-I1 operating system. Experimental parameters are easily set up from either the hardware control logic or the microcomputer. Digitized data are displayed by both an oscilloscope and a graphic display terminal. For an experiment which requires a precise timing, a start timing can be accepted.

PERFORMANCE
The performance of this linear detector was evaluated by comparing EXAFS and near-edge spectra for various materials obtained by a dispersive and point-by-point methods. A linearity of photodiode output was measured by monitoring the transmitted X-ray beam intensity with various thicknesses of absorber (aluminum foil). The dynamic range of photodiode array for the X-rays at 9 keV (Cu K-edge) is estimated as $10^9$. Absorption spectra are obtained by measuring the intensity of incident and transmitted X-rays and taking a logarithm of $(I_0/I)$. All measurements were carried out when the storage ring was operated at 2.5 GeV. Higher harmonics are negligible due to a platinum coated mirror placed between the polychromator and a linear detector.

The energy resolution of the spectrometer is given as a convolution of the spatial resolution of a linear detector and a geometrical resolution [1]. The total resolution was measured by placing a narrow slit (20 μm) at 50 mm from a photodiode array. The FWHM (full width at half maximum) of a sharp peak arising from X-rays escaped from the slit was 5.6 eV at the Fe K-edge (7.1 keV) when an energy range of 1200 eV is covered. With $R=2460$ mm, a geometrical energy resolution is estimated as 1.73 eV at the Fe K-edge. Since the slit width corresponds to 0.98 eV in energy, the spatial resolution of a linear detector is a decisive factor of the total resolution. A better energy resolution (2 eV at 9 keV) is achieved either by a direct exposure of photodiode or by use of silicon (311) crystal.

Fig. 3 Fe K-EXAFS oscillations of iron foil.

Fig. 4 Fourier transform of Fe K-EXAFS.
Figure 3 shows the Fe K-EXAFS oscillations of iron foil measured by (a) a point-by-point method, (b) energy-dispersive mode with 3.5 sec integration, and (c) energy-dispersive mode with 35 msec integration. A cylindrically bent triangular silicon (111) crystal was used as a polychromator in a dispersive experiment while a silicon (311) channel-cut monochromator was used in a point-by-point measurement. The energy scale in a dispersive mode was calibrated by an interpolation between the Fe K-edge (7.114 keV) of iron foil and Ni K-edge (8.334 keV) of copper foil which were measured simultaneously.

The EXAFS oscillations obtained by the two methods agree well each other both in position and magnitude. The magnitude of oscillation in (b) is smaller than (a) by 20 % in the low k range (k<6.0 Å⁻¹) whereas the dispersive mode gives the same magnitude of oscillation with that of a point-by-point method in the high k range (k>6.0 Å⁻¹). These results suggest that a difference in the magnitude is due to the lower energy resolution of dispersive mode which averages out sharp structures at k=4-5 Å⁻¹.

Figure 4 shows the results of Fourier transform of the data shown in Fig. 3. The positions of a prominent peak located at 2.2 Å due to the contribution of the nearest neighbors agree with each other within an experimental error (0.01 Å). The positions of more distant shells located at 3.6 Å and 4.5 Å in (b) and (c) also agree those of a point-by-point method. The magnitude of Fourier transform for the EXAFS data in a dispersive mode is smaller than that of point-by-point mode by 10-15 %. It should be noted that the data taken in 35 msec gives essentially the same features in the radial function with the data integrated over 100 times. These results suggest that the noise in EXAFS data taken in 35 msec by an energy dispersive spectrometer is statistical since a high frequency random noise is canceled out during the Fourier transform.

A new type of photodiode array (Hamamatsu S2301-512Q) with 512 sensor elements separated by 50 μm has been tested in a direct exposure mode. This photodiode array has superior signal-to-noise ratio because of a low-noise three-phase switching register constructed by a Plasma-Coupled Device (PCD). Preliminary experiments indicated that the EXAFS spectra extending over 300 eV with an energy resolution of 2 eV can be obtained in less than 10 msec.

The present linear detector system has been applied to kinetic studies of chemical reaction by Matsushita et al. [6], which demonstrates that the near-edge spectra can be recorded with a time resolution of 25 msec using a stopped-flow apparatus. The time resolution is now limited by the signal-to-noise ratio at the preamplifier partly due to a low quantum efficiency of phosphor. Further improvement of time resolution is possible if the incident photon flux can be increased by two orders of magnitude. The use of a secondary electron multiplier such as a microchannel plate (MCP) as an image intensifier is expected to realize a time resolution of a few msec. A new intensified photodiode detector has been built and the details will appear elsewhere.

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