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Design and testing of a prototype multilayer analyzer X-ray fluorescence detector

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It is a concern that multielement solid state detectors, commonly used for x-ray fluorescence detection on dilute systems, will not be optimal with the third generation sources due to their count rate limitations and the high photon flux from the source. As an alternative, we are developing energy-resolving x-ray fluorescence detectors using synthetic multilayers. In this paper, we present the design and testing of a prototype multilayer array detector that consists of three elements of graded multilayers. The tests show that the elements can be accurately aligned relative to each other, and the instrument can be easily tuned to other fluorescence energies once aligned. A background rejection rate of 40-50 times has been achieved with a throughput of 28-30 percent.

Keywords: X-Ray fluorescence detector, Synthetic multilayers

The unprecedented photon flux and brightness at the third generation synchrotron sources world wide provide real opportunities to widen the horizon of XAFS applications in terms of sensitivity and resolution. However, the currently available solid state fluorescence detectors, such as 13-element Ge detectors (Cramer, et al. 1988), are not optimal due to their count rate limitations of several hundred kilohertz (total counts) per channel. The estimated background and signal photon count rate is in the order of 10^8-10^9 per second into a one steradian solid angle for a typical solution sample at such sources, which is at least one to two orders higher than the pulse processing rate of the Ge detector. Thus we have proposed previously the development of x-ray fluorescence array detector using synthetic multilayers (Zhang, et al. 1998). The advantage of the multilayer array detector is its removal of background with the diffraction of signal from the multilayers, thus avoiding the pulse processing bottleneck of the solid state detectors. This type of detector will have a better detection efficiency than the Ge detector at the third generation synchrotron sources. We have shown that multilayer array detectors can be built with good energy resolution and reasonable solid angle, and is tunable over a wide energy region. In this paper, we report the design and testing of an early prototype detector containing three graded multilayer elements.

We used three linearly graded multilayers for the prototype detector. The d-spacing change in the diffraction plane of the multilayers was from 20 to 34 Å. W/B_4C double layers were deposited on 100 mm X 100 mm Si substrates. The multilayers were characterized at Beam line X18B of National Synchrotron Light Source (NSLS) of Brookhaven National Lab. The measured reflectivity at 22 Å d-spacing is about 45% at 7 KeV, and it increases to 65 to 70% at 32 Å d-spacing.

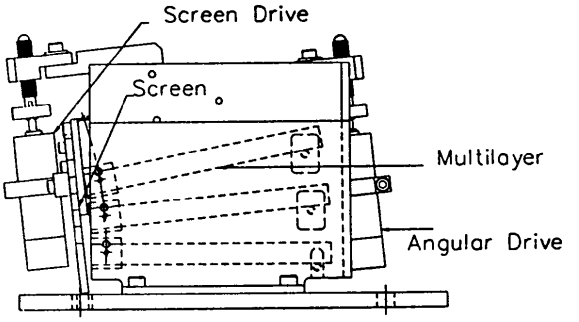

Figure 1. Side view of the design of the prototype detector. The unit consists of 3-multilayer elements. Two servo motors drives are used to control the multilayer rotation and the size of the beam entrance slits.

Figure 1 shows the layout of the prototype detector design. The 3-multilayer elements are mounted vertically. One servo motor and lead screw assembly is used to control the multilayer orientation in the diffraction plane, and another drive assembly is used to move the screen in the vertical direction to control the size of the beam entrance slits. Each drive has an accuracy of much less than a micron in linear motion. Each element can be individually adjusted for the purpose of aligning the elements relative to one another. The designed energy range is from 5 to 10 KeV, and the total detection solid angle for the prototype detector is 0.023 steradians at Fe Kα (6.4 KeV) and 0.017 steradians at Zn Kα (8.6 KeV).

The prototype detector was tested at Beam-lines X18B and X9B of NSLS. The analyzer was placed at 150 cm from the sample in the direction perpendicular to the beam direction. An ionization chamber was placed behind the prototype module to collect the signal. First, the test was done on a concentrated Fe sample. Figure 2 (top portion) sketches out the setup for obtaining a detector calibration curve using fixed beam entrance slits. With the increase of the angle of the multilayers in the diffraction plane, the detector elements started to block the direct beam (which is the portion directly from the sample and without interception by the multilayers) from entering the ionization chamber. This decline of direct beam signal was followed by a small peak and a large peak from the reflection of the multilayers. The small peak corresponds to the diffraction of Kβ fluorescence and the large peak, Kα. With the reference to the Kα peak, individual elements can be aligned relative to each other using the fine adjustment mechanism. Figure 2 shows the normalized detector calibration curve relative to total incoming beam with all the multilayer elements aligned. The Kα peak is located at about 45 milliradians. The height of the peak, which is the integrated throughput, is approximately 28%. Once aligned, the detector was calibrated on Co and Zn concentrated samples. At both fluorescence energies, the measured throughput of the detector was approximately 27 to 28%. Thus it shows that the energy change of the detector can be easily accomplished by rotating all elements to appropriate orientations.

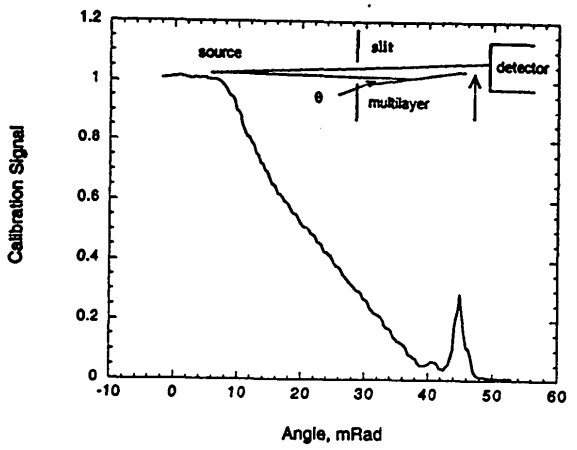

Figure 2. Prototype detector calibration on a concentrated Fe sample. The experimental configuration for the calibration is also shown. The throughput of the detector is approximately 28 percent.

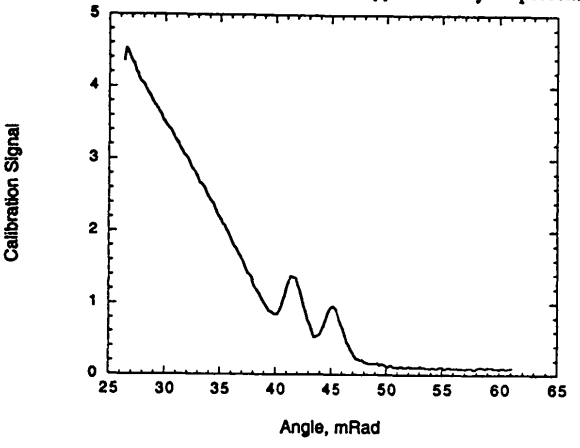

Figure 3. Prototype detector calibration on a 4 mM iron solution sample. The two peas located at 42.5 and 45 mRad are due to the diffraction of background and fluorescence, respectively.

Tests were also performed on solution samples with various metal concentrations. Figure 3 shows the detector calibration curve collected on a 4 mM iron solution sample. With the rotation of the detector elements in the diffraction plane, two diffraction peaks are observed. The first peak corresponds to the background photons having a larger energy, and the second peak located at 45.5 mRad corresponds to the fluorescence photons. The detector was then set at the angle corresponding to the peak of the iron fluorescence, and x-ray absorption near edge spectra were collected. Figure 4 A and 4 B show the spectra with the orientations of the multilayer elements at the fluorescence peak and at 9 mRad to allow for the measurement of signal directly. The signal to background ratio without using the analyzer is (Fig 4 B) 0.37, and it increases to 4.7 when analyzer was used. This gives a background rejection rate of 12.7 times. In this case, the effective photon counts, which is the square of signal counts divided by the total counts, are not improved by using the analyzer. The multilayer detector will become better statistically when the background is 4 to 5 times larger than the signal. When the background is much larger than the signal, the effective counts with the analyzer will improve by 3.5 times approximately.

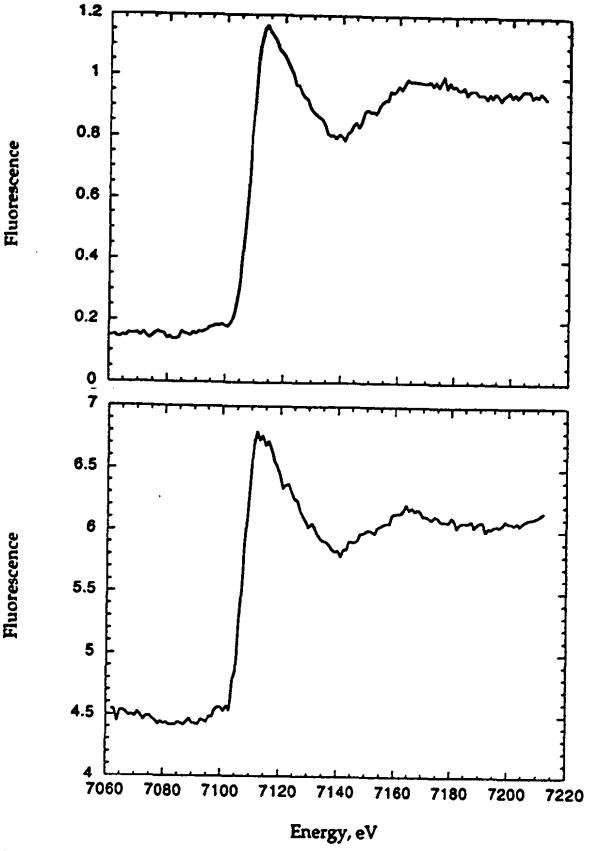

Figure 4 X-ray near edge spectra collected with the prototype detector (top) and without the prototype detector (bottom). In the later case, the multilayer elements were set at 9 mRad in the diffraction plane.

To summarize, we have constructed a prototype multilayer array detector with three graded multilayer elements. Tests of the prototype detector at synchrotron beamlines show that individual elements can be aligned accurately relative to each other, and energy changes can be accomplished easily by simply changing the elements orientation. The throughput of the prototype detector was 28%. Tests on dilute samples showed a background rejection ratio of 12.7 time, which is equivalent to a background rejection rate of 45 times. We are now constructing a 20 element multilayer detector unit for further evaluation.

References

Cramer, S. P., Tench, O., Yocurn, M. &George, G. N. (1988), Nucl. Instrum. Methods. A266, 586-591.
K. Zhang, G. Rosenbaum, and G Bunker, J. Synchrotron Rad. (1998) 5, 1227-1234.

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