Tests of a multilayer analyzer x-ray fluorescence array detector

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Introduction

It is a concern that multi-element solid-state detectors commonly used in x-ray fluorescence detection for dilute systems will not be suitable at the third-generation sources such as the Advanced Photon Source (APS), Argonne National Laboratory. Indeed, with the more than 10–13 photon flux available at the Bio-CAT insertion device (ID) beamline, a solid state detector (e.g., the Canberra 13-element Ge detector) will not work efficiently because of count rate limitations. It is estimated that only a few percent of the beam can be utilized by such a detector. In other words, elements in the order of thousands are needed on the detector to use the whole beam effectively. Thus, we have proposed the development of x-ray fluorescence detectors using multilayers [1, 2]. Acting as an analyzer, the mutilayers select x-ray fluorescence signals from background photons when the Bragg condition is met at the fluorescence energy. The data can be collected using a nonenergy-resolving detector. Thus, this type of detectors essentially has no count rate limitations.

Methods and Materials

A 20-element multilayer array detector using graded multilayers was designed. The smallest d-spacing of the multilayers is 20 Å with a grading ratio of 1.7. The dimensions of the multilayers are 100 mm perpendicular to the diffraction plane and 80 mm in the plane. The analyzer array has a total solid angle of 0.21 steradian at Mn Kα and 0.14 at Zn Kα energies. The detector is shown in Figure 1. Two driving systems are used to control the detector: one controls the size of the entrance slits and the other controls the rotation of 20 multilayers. The orientation of individual elements can be adjusted relative to each other in order to align the analyzer.

The tests of the detector were performed at the Bio-CAT beamline, 18-ID. The beamline was operating with a double-crystal monochromator with Si(111) crystals. The x-ray beam was focused horizontally and vertically. The beam was further restricted by Huber slits to a spot of 0.3 mm horizontal and 0.1 mm vertical.
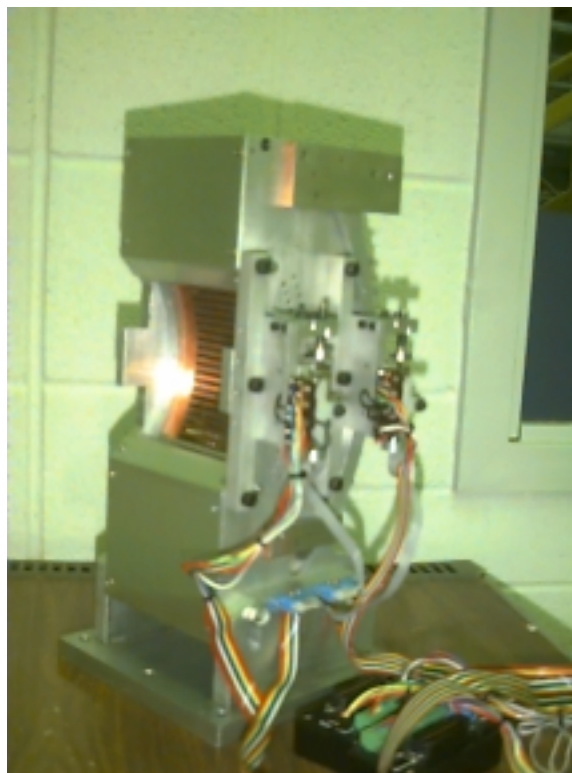

Figure 1: Photo of the 20-element multilayer analyzer unit.

Results and Discussion

Initial tests of the detector unit were done on concentrated metal complexes. Individual elements were characterized for their reflection at the Fe Kα fluorescence energy. The elements were aligned relative to each other using the fine adjustment mechanism. The detector calibration curve was obtained by rotating all the elements in the diffraction plane with a fixed beam entrance slit. Figure 2 shows the calibration at the Fe Kα energy. As can be seen from the calibration curve, the throughput of the detector is approximately 26%. Similar throughput of the detector was obtained when calibrated at other fluorescence energies (e.g., Mn, Co, Cu, and Zn). This shows that the alignment will be maintained for various energies.
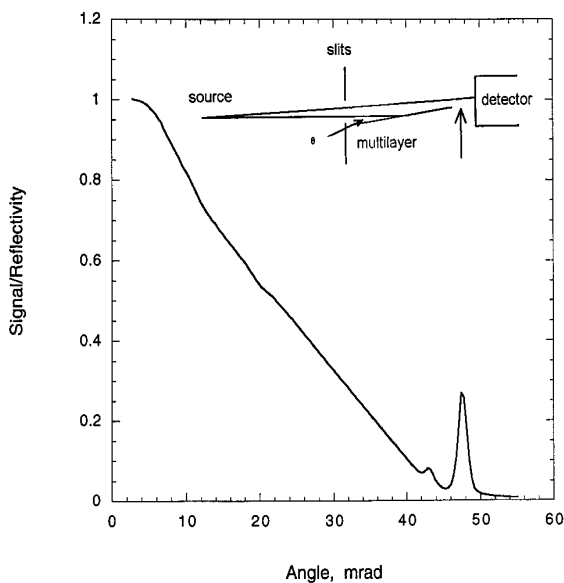
Figure 2: Calibration of the multilayer array analyzer detector (MAAD) at the Fe Kα using a fixed entrance slit.

The cause of the low throughput of the detector (26%) was revealed by measuring the reflection properties of the multilayer elements. The reflectivity is more than 70% at the large d-spacing side and falls off substantially below 23–24 Å d-spacing (data not shown). The full width at half maximum (FWHM) increases dramatically from about 60 eV at 21 Å to 190 eV at 33 Å, which is consistent with simulation results. Thus, the low throughput of the detector can be attributed in part to the low reflectivity at the low d-spacing region. The detector was tested on dilute biological systems. Figure 3 shows a single x-ray absorption fine structure (XAFS) scan when measured at the analyzer angle corresponding to the Mn fluorescence energy on a 2 mM Mn solution sample. The signal-to-background ratio is approximately 5:1, while the signal-to-background ratio without the analyzer is 1:3. Thus, the total rejection rate is approximately 15 times.
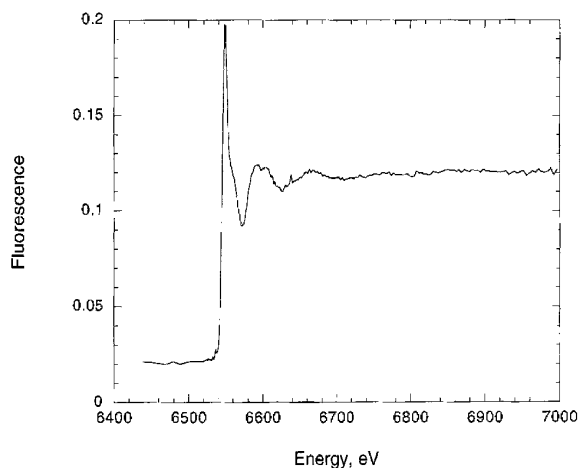

Figure 3: XAFS single scan measured with MAAD.

References

[1]   K. Zhang, G. Rosenbaum, and G. Bunker, *J. Synchrotron Rad.* **5**, 1227–1234, (1998).
[2]   K. Zhang, G. Rosenbaum, and G. Bunker, *J. Synchrotron Rad.* **6**, 220–221, (1999).