Performance of Multilayer Array Analyzer Detector in the Intermediate Energy Region (1 KeV to 3 KeV)

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Abstract. It is natural to use multilayer analyzers as energy resolving x-ray fluorescence detectors in the lower energy region. The analyzer array detectors can afford large detection solid angle, superb energy resolution, and reasonable efficiency. We have constructed a 12-element multilayer detector for operation starting at about one KeV. The detection solid angle at 2 KeV is roughly 3 % of 4π. The FWHM of the detector response curve can reach 30 eV close to 1.2 KeV. The fabrication and testing of the prototype detector will lead to the design of better multilayer array analyzers in this energy region.

Keywords: graded multilayers, analyzer, x-ray fluorescence, intermediate x-ray energy.

INTRODUCTION

We have been developing multilayer array analyzer detectors for x-ray fluorescence [1]. Using laterally graded multilayers, the analyzer/detector can be made tunable over a very large energy region. The primary advantage of such a detector is its high count rate capability. Since the multilayers "select" fluorescence signal based on the diffraction principle, various kinds of detectors can be used for data collection after the analyzers. This includes scintillation counters, ionization chambers and Si photodiodes. It is believed that this kind of detector would be very useful in fluorescence detection with high flux from the third generation synchrotron sources.

Our first multilayer array analyzer detector contains 24 elements [2]. This detector was designed to cover an energy region from 4 KeV and up. At the Ca Kα fluorescence energy the detector collects a total solid angle slightly over 3% of 4π. Tested on both dilute and concentrated Ca samples, the energy resolution of the detector, defined by the FWHM, is roughly 4% of the fluorescence energy, namely 140 eV at 3.5 KeV. The throughput of the system is approximately 30%. Thus the multilayer analyzer detector system would be very promising for working in the intermediate (1 KeV to 3 KeV) and in the low energy region (< 1 KeV). The analyzer system will have a much larger detection solid angle per unit element, while maintaining a good energy resolution. This is coupled with the fact that the energy resolution of solid-state detectors deteriorates with decreasing of photon energy. To explore the opportunity of using the detector working in the lower energy region, we have designed and fabricated a prototype multilayer array analyzer detector working above 1 KeV in fluorescence energy.

DESIGN AND TESTING

The detector contains 12 multilayer elements with a dimension of 100 mm square. The designed d-spacing change is from 24 to 45 Å over a 100 mm distance. This would allow the total coverage of solid angle at 5.5% of 4π at 1 KeV and 2.8% of 4 π at 2 KeV. The multilayers were characterized at synchrotron beam lines to evaluate their reflectivity and bandwidth. At 6.5 KeV, we achieved a reflectivity generally between 55 to 75% with a bandwidth between 2 and 5%.

The 12-element multilayer array analyzer detector is shown in Figure 1. The alignment control of the detector is through two servo motor drivers. One of the drivers determines the orientation of the multilayers to the source, and the other controls the size of the beam entrance slits. An optional third drive may be added to control a set of back slits, which defines the exit window of the beam to the analyzers. The entrance slits is designed with a Venetian blind type of structure. Thus the vertical dimension of the entrance slits can be adjusted from 2 mm to 8 mm.

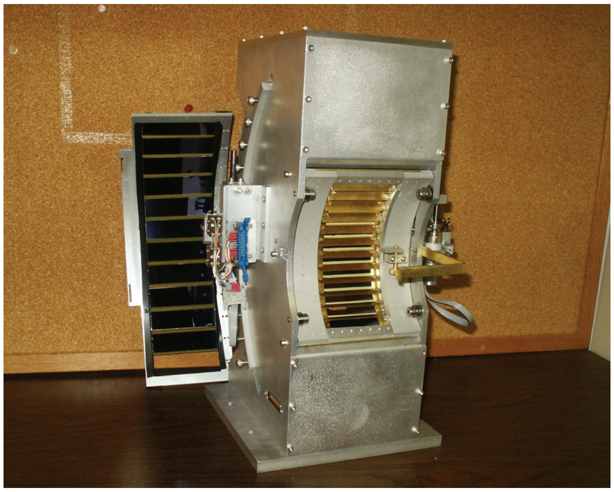

FIGURE 1. 12-element multilayer array analyzer detector.

Showing in the back of the analyzer unit is a ring of Si photo diodes for collecting signal after the multilayers (see Figure 1). The reason for selecting the photo diode with thin oxide dead layer is due to its large linear range, high efficiency, compact size and low cost. However, the detector can be operated only in integration mode, which limits its detection sensitivity.

The detector has been commissioned and tested at the 2-ID-B and 9-BM beamlines at the Advanced Photon Source, Argonne National Lab. First the elements were aligned relative to each other. This procedure requires fine adjustment of the individual multilayer orientation. After this procedure, the detector is ready for operation. The first test was done on a concentrated Al sample. With a fixed entrance slits, the detector was calibrated by rotating the multilayer elements by gradually intercepting the direct beam from the fluorescence source. In this manner, the reflection efficiency can be obtained by evaluation of the Kα fluorescence peak relative to the signal of the direct beam. From this calibration curve (data not shown), the reflective efficiency and its energy resolution could be obtained, which were approximately 23% and 40 eV, respectively.

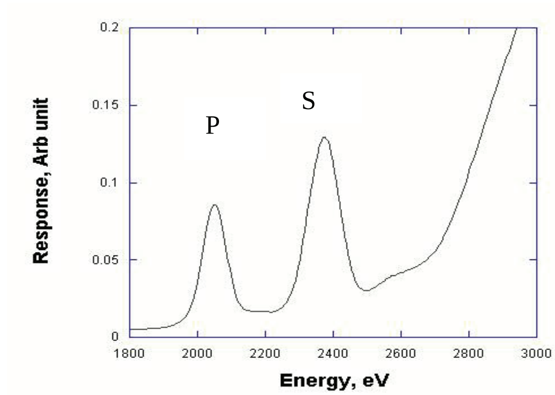

FIGURE 2. Fluorescence response on a Bovine liver sample. Note that the multilayer rocking curve has been converted into energy space.

Further tests were performed on a NIST standard of Bovine liver, which contains 0.7 weight percent of sulfur and 1.1 weight percent of phosphorous. Setting the photon energy at 2.54 KeV, we measured the detector calibration curve on the sample, which is shown in Figure 2. As can be seen from the figure, both S and P Kα are shown, while their Kβ fluorescence show as shoulders at higher energies, respectively. Measured by the Full-Width-at-Half-Maximum (FWHM) of the Kα fluorescence peaks, the analyzer resolution is about 100 eV at the S fluorescence energy, and 80 eV at the P fluorescence energy.

Detector calibration was also done on a Ge sample, which is shown in Figure 3. In this case, the beam energy was set at 1.83 KeV. The plot shows the detector response (arbitrary unit) vs. the energy. Ge Lα and Lβ fluorescence lines are located at 1188 eV and 1218 eV, a 30 eV separation. As shown in the calibration curve, the peaks are very well resolved. The detector response curve can be easily fit with two Gaussian distributions. The FWHM of the two distributions are 30 and 28 eV, respectively.

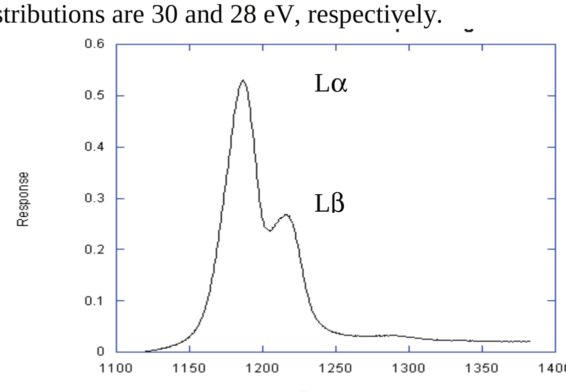

FIGURE 3. Detector response at Ge L fluorescence.

SUMMARY

Fluorescence detection on dilute systems in the intermediate to lower energy region is challenging due to lower fluorescence yield and the requirement for much better energy resolving detector systems. For instance, the ratio of energy separation between the edge energy and emission energy to the edge energy is generally larger than 10% for the K-edges of transition metal elements, while the ratio is reduced to 8.6% for Ca, 5.1% for S and 3.9% for Mg K edges. This is coupled with the fact that the fluorescence lines are densely distributed in this energy region. The energy resolution of widely used 13-element Ge detectors and Si drift detectors [3], which is at 3-4% at 6 KeV, would be 5% or greater when working in the intermediate to lower energy region. Thus an energy resolving detector with better energy resolution would be most desirable for fluorescence detection in this region.

We have constructed a prototype multilayer array analyzer detector for operation at intermediate to lower energies. With linearly graded d-spacing in the diffraction plane, the analyzer is tunable in a large energy region, and covers reasonable solid angle. Tested at 2.5 KeV to 1 KeV range, we obtained an energy resolution close to 3% with a throughput over 20%. In addition, the analyzer/detector can handle large count rates encountered at third generation synchrotron sources. Further development of such detectors should produce better analyzer/detector systems suitable for this energy region.

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