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DEVELOPMENT OF MERCURIC IODIDE DETECTORS FOR XAS & XRD MEASUREMENTS

WILLIAM K. WARBURTON, JAN S. IWANCZYK, and ANDRZEJ J. DABROWSKI

Institute of Physics, University of Southern California, 4676 Admiralty Way

#932, Marina del Rey, CA 90292, USA

AT03-77EV72031

BRITT HEDMAN, JAMES E. PENNER-HAHN, A. LAWRENCE ROE, and KEITH O. HODGSON

Stanford Synchrotron Radiation Laboratory, SLAC Bln 69, P.O. Box 4349,

Stanford, CA 94305, USA and Department of Chemistry, Stanford University,

Stanford, CA 94305, USA

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ALBERT BEYERLE

DE86 011965

EG&G Inc., 130 Robin Hill Road, Goleta, CA 93017, USA

A prototype element for an energy dispersive detector (EDD) array was constructed using a Mercuric Iodide detector. Both detector and front end FET could be thermoelectrically cooled. Tested at SSRL, the detector had 250 eV electronic noise and 315 eV resolution at 5.9 keV. K line fluorescence spectra were collected for selected elements between Cl (2622 eV) and Zn (8638 eV). Count rate capability to 60,000 cps was demonstrated. Several detector parameters were measured, including energy linearity, resolution vs. shaping time, and detector dead time. An EXAFS (extended x-ray absorption fine structure) spectrum was recorded and compared to simultaneously collected transmission data.

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1. Introduction

There is general interest in the synchrotron radiation community in arrays composed of energy dispersive detector (EDD) elements. Such arrays have potential applications in many areas, including fluorescence EXAFS, anomalous diffraction, and energy dispersive diffraction. Mercuric Iodide x-ray detectors are receiving serious consideration in this context for several reasons. First, due to its large bandgap, (2.2 eV, with picoamp room temperature leakage currents) it can be made into high quality, room temperature (RT) x-ray detectors [1]. Currently RT resolution of 290 eV for Fe-55 is obtainable and principally limited by preamplifier noise. Spectra have been obtained at energies as low as O-K (523 eV) and Fe-L (704 eV) [2]. Second, because RT operation eliminates both cryogenic and vacuum packaging, HgI₂ claims an important advantage over both Si and Ge in the construction of rugged, compact, low power EDD arrays. HgI₂ is a new material, however, requiring some novel fabrication and electronic techniques, so there are many questions which must be addressed before a HgI₂ array can be commissioned.

We therefore began this process by fabricating a single HgI₂ detector channel, as a prototype element in an eventual array, and performing various tests on it at SSRL. Among our goals were the evaluation of count rate capability, resolution, robustness, compatibility with array solid angle needs, special electronic requirements, and unusual packaging problems.

2. Detector System Design and Experimental Setup

The detector (see Figure 1) was constructed as a small, hand portable unit consisting of three attached parts: an unencumbered HgI₂ detector probe, a pulsed optical feedback preamplifier, and a battery powered, high voltage supply. The probe dimensions are 19 mm dia by 50 mm long. The other two parts are packaged in small component boxes 42 mm x 68 mm x 75 mm.

The probe is an Al tube with a 50 μ m Be window (50% cutoff at 2500 eV) holding the HgI₂ detector, the front end FET, and two thermoelectric coolers mounted on a Cu support structure. The detector is a 400 μ m thick slice of HgI₂, with painted-on, carbon emulsion contacts of 4 mm² area containing embedded lead wires. Excepting the active front contact, this assembly is embedded in plastic on a BeO substrate attached to a thermoelectric cooler. The collecting lead connects directly to the front-end FET's gate, which is also cooled thermoelectrically via a BeO stand-off post. The probe was backfilled with dry nitrogen gas and hermetically sealed after assembly. In operation, the detector is cooled to about +5 °C and the FET to about -15 °C. The total cooling power is about 0.25 W.

The preamplifier is of the charge-sensitive, pulsed optical feedback variety, which was found to perform best for HgI₂ and near RT components [2]. The preamplifier chassis is used to mount the detector probe. The high voltage supply is a small DC-DC converter adjusted to provide the optimum voltage for the detector. It operates for one year on a 4.5 V camera battery.

The complete spectrometry system also included a Canberra 2020 amplifier, Ortec 550 SCA, Ortec 419 pulser, and Tracor Northern TN-1710 MCA. Both SCA and MCA were connected via CAMAC modules to a PDP 11/34 computer. Input x-ray intensities were monitored using a standard SSRL short ion chamber system. All data were taken at SSRL BL II-3, unfocussed, under dedicated conditions (3.0 GeV, 50 mA) using a Si[111] double-crystal monochromator.

3. Experimental Results:

Resolution versus energy with 12 μ sec shaping time was determined in two different ways. First, monoenergetic spectra were taken at 1 keV intervals between 4 and 10 keV of x-rays scattered off Kapton tape. Second, samples of CsCl, Ti, Cr, Fe, Cu, and Zn were irradiated and their fluorescence spectra recorded. Characteristic of this series, the Cl, Fe, and Zn spectra are shown in Figure 2. Resolution values in the range of 295 to 350 eV were obtained at 500 cps. Peak positions were found to be linear in energy, as expected.

Resolution was also measured as a function of amplifier shaping times at 4, 7, and 10 keV at 500 cps and found to be essentially independent of shaping time between 4 and 12 μ sec, but degraded considerably for 1 or 2 μ sec shaping times.

Work was also attempted to determine count rate capability and resolution at high counting rates, but met with mixed and irreproducible results. For example, using 3 μ sec shaping time at 5000 cps, the resolution

ΔE was 470 eV. Remeasurement at 8000 cps following a count rate increase to 12,000 cps gave ΔE of 380 eV, still at 3 μ sec. These results seemed to be caused by random baseline fluctuations which we believe arose from contact charging and injection at the higher currents associated with higher count rates. An ad hoc solution adopted to minimize the influence of this low frequency noise was to employ an ultrashort shaping time of 0.25 μ sec. This degraded resolution to 910 eV, but the detector was able to function successfully at 60,000 cps, a factor of 6 higher than ever previously reported [3]. This poor resolution was found to be count rate independent, being determined entirely by electronic noise.

A detector deadtime measurement was made by measuring Fe-K α count rate R_d in the HgI₂ detector, recorded as a function of intensity I_0 in the x-ray beam used to excite an Fe foil. I_0 was measured with an ion chamber. Deadtime was determined from a linear fit to a plot of R_d/I_0 vs. I_0 and found to be 2.1 μ sec for 0.25 μ sec shaping time operation.

Finally, using the HgI₂ detector with 0.25 μ sec shaping time, an EXAFS spectrum was taken at the Fe edge from a sample of synthetic magnetoplumbite. The sample had been prepared for transmission EXAFS and was about 2 absorption lengths thick. I_0 and transmitted intensity I , were monitored using N₂ filled ion chambers to obtain a transmission EXAFS spectrum simultaneously. Both curves are presented in Figure 3. To our knowledge, this represents the first use of HgI₂ as a fluorescence EXAFS detector. An examination of the curves shows that, with 60,000 peak counts/sec, we are able to obtain data with comparable amplitudes and phases to the simultaneously recorded transmission data. Exact amplitude equality

is not expected, since the sample is too concentrated for precision fluorescence work. The fluorescence spectrum also shows a higher noise level than the transmission spectrum, but no higher than can reasonably be attributed to counting statistics. An attempt to collect data with a longer shaping time at 5000 cps proved unprofitable due to extremely poor counting statistics. Similarly, we were unable to work either with dilute samples or at low energies (the Cl edge) simply because the detector area (4 mm^2) was too small to intercept acceptable count rates. This is not unexpected, since this area limits solid collection angles to about $\pi/1000$.

4. Discussion and Conclusions

A prototype EDD array element using a HgI₂ detector element was transported to SSRL and found to be robust, giving resolutions and count rate behavior equivalent to laboratory results. Resolutions were found to be between 300 and 350 eV between 2.6 and 10.0 keV, which is acceptable for array requirements. For the first time the HgI₂ detector system was operated up to 60,000 cps count rate. The need for further contact development and larger solid detection angles was established. The detector was also demonstrated to be capable of obtaining reasonable EXAFS spectra.

We are now able to consider what the next steps should be in developing HgI₂ detectors for EDD array elements. These include:

1: Detector development: needs were clearly established for more reliable contacts and larger area detectors. The latter requirement implies a need for higher quality detector material, so that both detector areas and

thicknesses can be increased while maintaining required capacitance values. Some research is also needed to establish how much areas can be increased without exceeding array resolution requirements.

2: Optimized electronics: the current electronics were optimized for high resolution, not high data rate applications. Other preamplifier and amplifier configurations may prove superior in array applications. Pulsed optical feedback systems, for example, may be a source of serious crosstalk between array elements. Miniaturization using hybridized or integrated circuitry will also be a necessary step for multielement array construction.

3: Small test arrays: construction of an array of 4 to 9 elements is the next logical step, as it will provide both working criteria to build and a mechanism to test most of the subsystems and components needed to build a successful EDD array composed of a large number of elements.

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Figure Captions

Figure 1: Photograph of the HgI₂ detector-preamplifier package.

Figure 2: Excitation spectra of Cl, Fe, and Zn recorded with HgI₂ prototype EDD array element.

Figure 3: Fe edge EXAFS spectra of magnetoplumbite recorded simultaneously in transmission and in fluorescence using HgI₂ prototype EDD array element.

Fig. 1

Fig. 2

Fig. 3