

Electron response in windowless Si(Li), SDD and PIN diode photodetectors

Christopher E. Cox, Stephen J. Asztalos, Wolfgang Hennig, William K. Warburton

Abstract—The photon response of small volume, low capacitance semiconductor X-ray detectors is widely reported and is characterized by the ability to resolve closely spaced X-ray lines. But the response to electrons is less well known. We present spectra from commercially available high-resolution X-ray detectors irradiated with mono-energetic internal conversion electrons with energies from 45 keV to 300 keV. Many radioisotopes emit conversion electrons in this range, and electron spectroscopy offers an additional tool for detection and identification. One application reported in this work is the detection of trace amounts of radioactive Xenon gas, where the simultaneous measurement of high-resolution X-rays and conversion electrons in Silicon X-ray detectors offers an improvement in the Xenon detection limit compared to scintillator-based systems. Future plans for improvements by the use of multiple detector arrays and coincidence gating are briefly discussed.

I. INTRODUCTION

A. Detector Structure.

HIGH resolution silicon X-ray detectors, for example the Lithium drifted Silicon detector (Si(Li)), the Silicon Drift Detector (SDD) and the PIN diode are established tools for X-ray Microanalysis, X-ray Fluorescence and other energy dispersive applications that require closely spaced X-ray lines to be resolved. Fig. 1 is a simplified schematic cross-section of a small-volume detector used for X-ray spectroscopy, in this case a silicon PIN diode. The bulk of the detector is near-intrinsic lightly doped p-type Silicon, and a Phosphorous implant forms the p-n junction at the front surface. A positive operating bias of about 200V depletes the active volume and creates the electric field for collecting electrons and holes. Typical dimensions for a high-resolution PIN diode are from 6 to 25 sq.mm surface area and 0.5 mm thick.

In the case of an SDD, negative bias is applied to a Boron implanted p-type contact, which forms the entrance window for radiation. The rear contact structure is complex, but the detector's response to X-rays and electrons is determined by

the absorption characteristic of the entrance window, which is similar to the PIN diode. The Si(Li) detector uses a negatively biased Schottky Barrier p-type contact at the front, which gives similar absorption characteristics to the other two detectors.

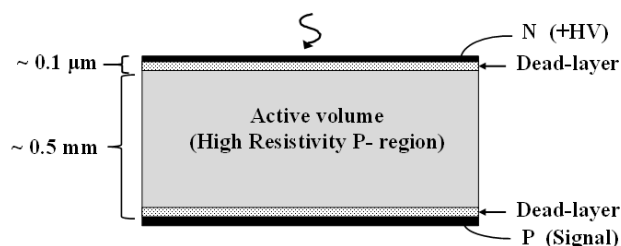


Fig. 1. Simplified cross-section of a silicon PIN diode.

For all three detector types, an unavoidable consequence of the semiconductor manufacturing process is the formation of a dead-layer between the contact and the bulk Silicon, that is, a low field region where the X-ray or electron energy can be partially or wholly absorbed without contributing to the output signal. This region of Silicon is much thicker than the contact itself and is the dominant source of X-ray and electron absorption at the entrance window. Improvements in detector technology over the last two decades have led to a reduction of the dead-layer from about 0.3 μm to 0.1 μm , resulting in a reduction of background counts from partial absorption, improved sensitivity to very low energy X-rays [1], [2], and an expected improvement in the collection efficiency of electrons. There have been earlier studies [3]-[7] irradiating Silicon detectors with internal conversion electrons. The current work aims to evaluate the latest Silicon X-ray detector technology for performing electron spectroscopy at energies as low as 20 keV.

B. Electron Penetration and application to Radioisotope Identification

Fig. 2 shows the approximate penetration depth of electrons in Silicon as a function of electron energy. For the PIN diode structure shown in fig. 1 electrons are fully absorbed in the energy range from a few keV to 500keV. Many radioisotopes emit mono-energetic conversion electrons in this range, with an electron energy equal to a Gamma-ray energy less the electron binding energy. In some applications where the radioisotope activity is very weak compared to background, it is desirable to both resolve closely spaced X-ray lines and collect Gamma emissions in the same detector. But due to the

Manuscript received November 2, 2011. This work was supported by the U.S. Department of Energy under Award No. DE-SC0004272.

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constraints of the Silicon material and the small detector size, it is not possible to efficiently detect Gamma rays in a high-resolution X-ray detector. However, conversion electron spectroscopy offers an alternative method to achieve the same goal, especially when the radioisotope under investigation is in the form of a gas, so that the self-absorption of conversion electrons in the source material is minimized.

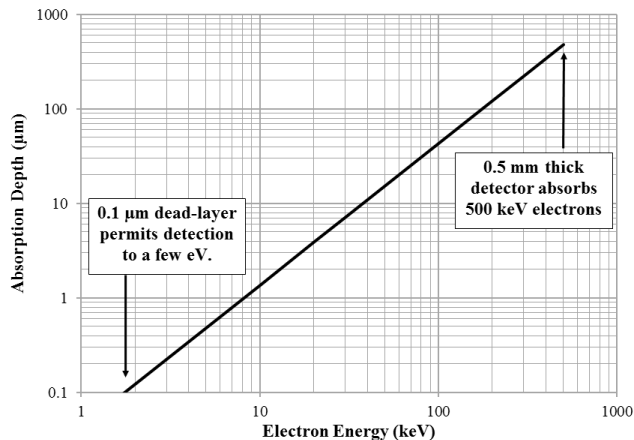


Fig. 2. Approximate absorption depth of electrons in Silicon [8]

We present results of simultaneous X-ray and conversion electron spectroscopy oriented towards the detection of trace amounts of radioactive Xenon gas, in support of the Comprehensive Test Ban Treaty Organization (CTBTO) monitoring program. The initial proof of principle was performed with a Liquid Nitrogen cooled Si(Li) detector. It is expected that the final development will involve the construction of an array of 24 25-sq.mm detectors mounted in a small cube structure for near 4π geometry. For this reason, subsequent measurements have focused on the compact and relatively low-cost PIN diode. X-ray and electron spectroscopy results are presented from a windowless PIN diode using two types of open, but solid, radioisotope sources, where inevitably some absorption of electrons takes place in the source. Finally, a more realistic test was performed with a mixture of ^{133}Xe and $^{133\text{m}}\text{Xe}$ gas where self-absorption in the source is greatly reduced.

II. INITIAL PROOF OF PRINCIPLE

Table I shows the radioactive sources used for this work, and the associated X-ray, Gamma-ray and conversion electron energies. Note that the complete conversion electron decay scheme typically has fine structure lines around the main peaks, but the spread in energy is usually less than the resolution of the detector, so the fine structure energies are not listed. Peaks with very low probability are also omitted. ^{133}Ba and ^{133}Xe both decay to ^{133}Cs and therefore have similar radiation emissions. $^{133\text{m}}\text{Xe}$ results from the ^{133}Xe decay scheme. An important specification for the detector in our CTBTO application is to resolve the $K\alpha$ lines at 30 keV and 31 keV from $^{133\text{m}}\text{Xe}$ and ^{133}Xe respectively, which requires about 500 eV FWHM resolution at these energies.

TABLE I. LIST OF RADIOACTIVE SOURCES USED FOR THIS STUDY.

Isotope	X-ray	Gamma-ray	Conversion Electron
^{109}Cd (solid)	$K\alpha$: 22 keV $K\beta$: 25 keV	88 keV	K : 62 keV L : 84 keV M : 87 keV
^{133}Ba (solid)	$K\alpha$: 31 keV $K\beta$: 35 keV	81 keV 276 keV, 303 keV 356 keV, 384 keV	K : 45 keV L : 75 keV M : 80 keV
^{133}Xe (gas)	$K\alpha$: 31 keV $K\beta$: 35 keV	81 keV	K : 45 keV L : 75 keV M : 80 keV
$^{133\text{m}}\text{Xe}$ (gas)	$K\alpha$: 30 keV $K\beta$: 34 keV	233 keV	K : 199 keV L : 228 keV M : 232 keV

The relative position of the Si(Li) detector and radioactive source used for the initial tests is shown in fig. 3. Spectrum Techniques supplied an “open” source that consisted of a needle coated with ^{109}Cd in solution, which was let to dry. The intention was to minimize the absorption of the conversion electrons in the source. For this test we were interested in observing the electron absorption in the air gap between the source and the detector, and the effect of the detector’s thin vacuum window. Fig. 4 shows the resulting multiple ^{109}Cd spectra for different air gap values, with the source outside the vacuum window.

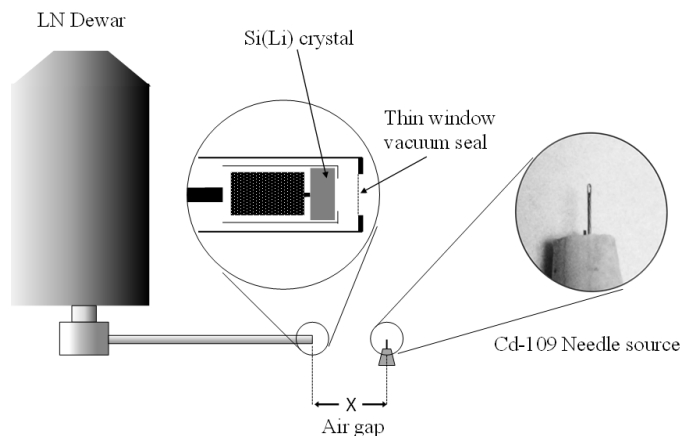


Fig. 3. Experimental set-up for measurements with a Si(Li) detector for the initial proof of principle.

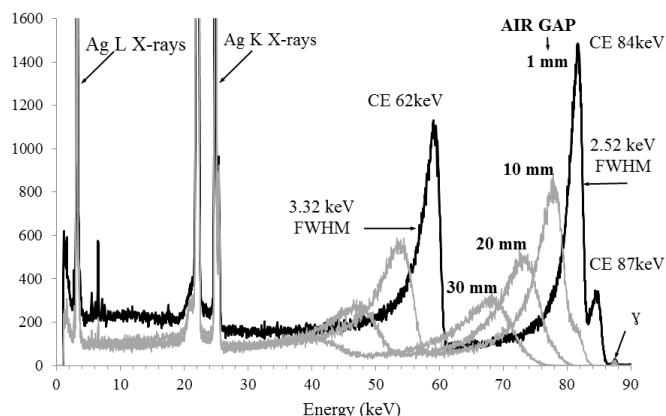


Fig. 4. ^{109}Cd spectrum from Si(Li) detector, showing the effect of the air gap between the source and detector vacuum window.

As the air gap is increased the conversion electron peak centroids shift to lower energies and a low energy tail develops due to partial absorption of electrons in the air. As expected, the absorption effect worsens for lower electron energies. The residual peak shift at the smallest air-gap (1 mm) is due to a combination of self-absorption in the source, the remaining air, the detector window and the detector dead-layer. For a gaseous source, this result illustrates the importance of using a small volume of gas to restrict the electron path lengths.

The next test involved mounting the ^{109}Cd source inside the detector vacuum to eliminate the absorption effect of the thin vacuum window, which was constructed from 300 nm polyimide film coated in Aluminum. Fig. 5 shows the ^{109}Cd spectrum with the source inside the detector vacuum, where the conversion electron peak shift and tailing is caused only by the detector dead-layer and self-absorption in the source. The resulting energy resolution of the electron peaks is significantly improved. Note also that even the relatively low energy Auger electron peaks are visible.

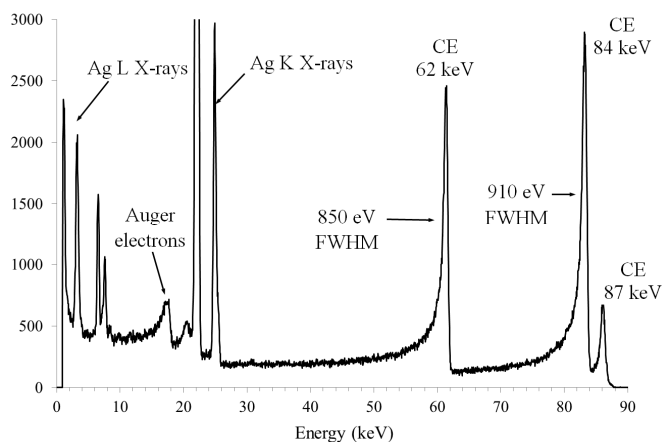


Fig. 5. ^{109}Cd spectra from Si(Li) detector showing the effect of moving the source inside the detector vacuum.

III. MEASUREMENTS WITH WINDOWLESS PIN DIODE.

A. Solid Sources: ^{109}Cd and ^{133}Ba

Fig. 5 demonstrates the importance of using a windowless detector to achieve the best energy resolution from the conversion electron peaks. The next set of data was therefore collected with an Amptek 6-sq.mm x 0.5 mm PIN diode with the Beryllium window removed. In this configuration the Silicon crystal is exposed to the radioisotope under test without the additional barrier of the vacuum window. This detector also has the advantage of being compact and relatively inexpensive.

The experimental set up is shown in fig. 6. For the initial test with ^{109}Cd the needle source was placed as close as possible to the detector, but still leaving an unwanted air gap of about 1mm. The detector was operated at room temperature to avoid the possibility of water vapor condensation on the sensitive Silicon surface. Fig. 7 is the resulting spectrum with this arrangement.

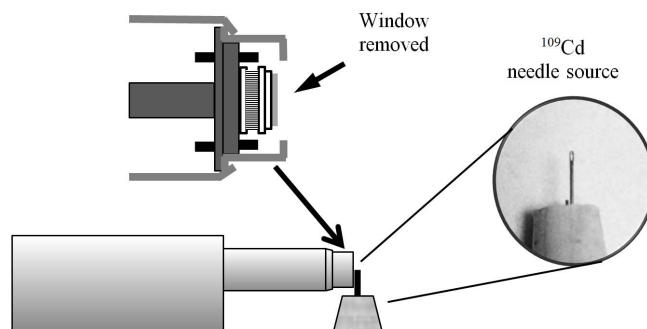


Fig. 6. Experimental set up for ^{109}Cd measurement with windowless PIN Diode. The air gap between the source and Si crystal is about 1mm.

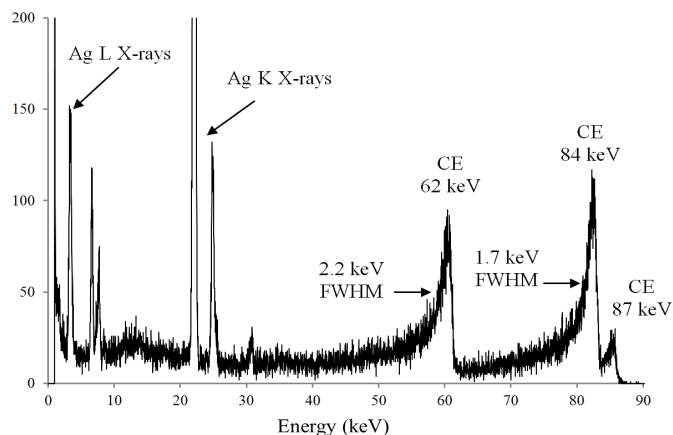


Fig. 7. Spectrum from ^{109}Cd using a windowless PIN diode in the configuration shown in fig. 6.

The electron peaks have improved resolution compared to the 1 mm air gap results from the Si(Li) detector, (fig. 4). The centroid shift and tailing is now from the combination of self-absorption in the source, the 1 mm air gap and the detector dead-layer, but there is no absorption from the window.

^{133}Ba , which simulates the X-ray and conversion electron peaks from ^{133}Xe , was the next source to be tested. This time the radioisotope solution was dried onto a small plate made from pure Silver, which provided improved geometry and collection efficiency compared to the needle source. The resulting spectrum is shown in fig. 8

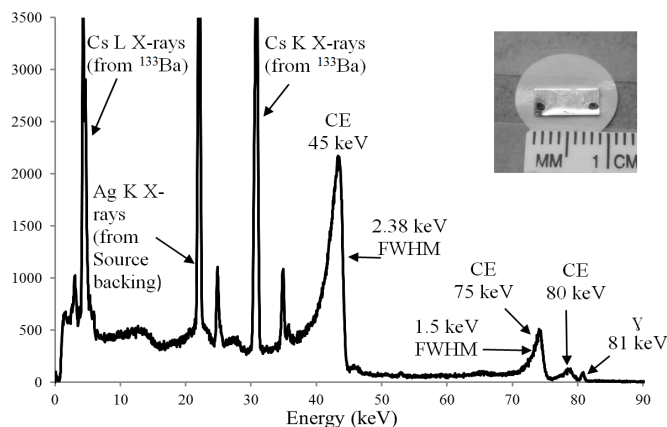


Fig. 8. Spectrum from ^{133}Ba "plate" source with the windowless PIN diode

^{133}Ba (and ^{133}Xe) emits a conversion electron at 45 keV, which is somewhat lower energy than the peaks from ^{109}Cd , but it is still well defined and easily observable, as shown in fig. 8. With the 6-sq.mm PIN diode operating at room temperature, the resolution of the Cs $K\alpha_1$ line at 31 keV was 474 eV FWHM, which is just sufficient to resolve the ^{133}Xe and $^{133\text{m}}\text{Xe}$ lines in the gaseous Xenon sample. However, for PIN diodes with a larger surface area, (e.g., 25-sq.mm), moderate cooling may be required to achieve the necessary X-ray energy resolution.

Figs. 9a and 9b are a comparison of ^{133}Ba spectra collected with an SDD and a PIN diode. The scale has been expanded to show the weak high energy conversion electrons around 300 keV. Unfortunately, it was not possible to obtain an SDD without a protective sealed window, and both spectra were therefore collected using detectors sealed with 25 μm thick Beryllium. The spectra are essentially identical, although the lower energy electron peaks at 45 and 75 keV are not well defined due to absorption in the window. Nevertheless, the result demonstrates the ability of the detectors to measure relatively high energy conversion electrons.

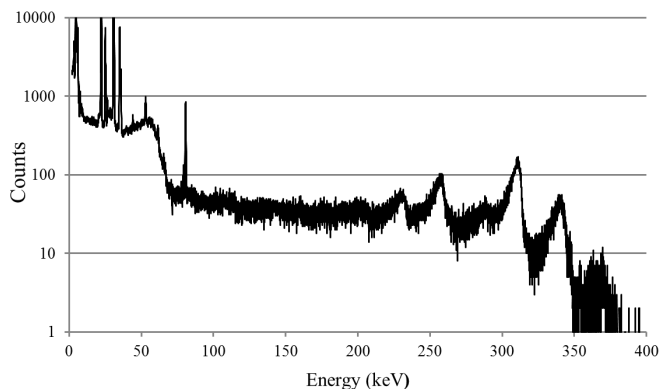


Fig. 9a. ^{133}Ba spectrum from a PIN diode with a 25 μm Be window.

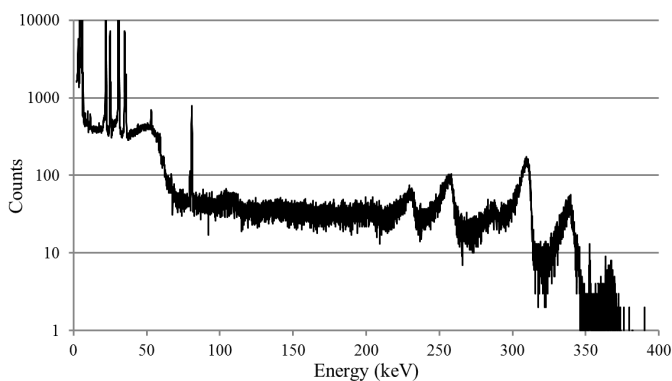


Fig. 9b. ^{133}Ba spectrum from an SDD with a 25 μm Be window, showing a close similarity with fig. 9a.

B. Gas source: ^{133}Xe and $^{133\text{m}}\text{Xe}$.

The final set of measurements for the windowless PIN diode was performed with Xenon gas, using the arrangement shown in fig. 10. Three 70 mm Conflat flanges, sealed with Copper rings, formed an internal chamber of about 4 cm^3 . A Swagelok fitting provided the gas inlet. The detector was sealed on the

surface of its end-cap with a small diameter O-ring. The “open” end of the chamber was sealed with a 25 μm thick Stainless Steel foil which, if required, allowed a second detector to measure coincidence Gammas, (but not conversion electrons). Not shown are the valves and vacuum pump to evacuate and purge the chamber of water vapor prior to introducing the Xenon.

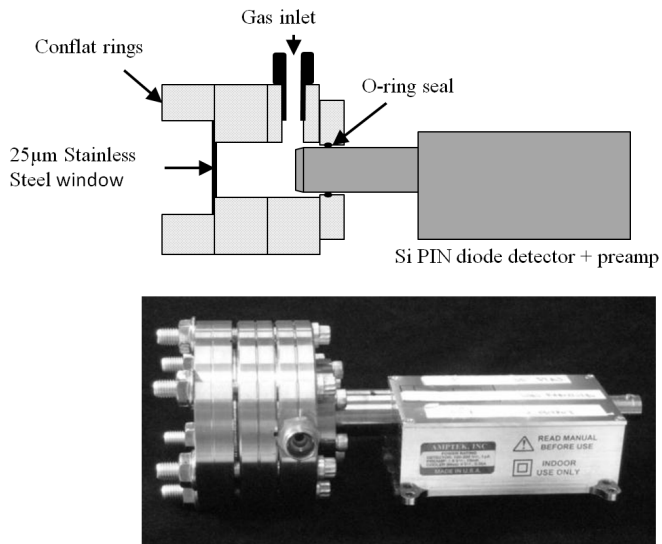


Fig. 10. Experimental set-up for measuring Xenon gas with the windowless PIN diode.

^{132}Xe was irradiated at the University of Texas research reactor to produce the short-lived isotopes ^{133}Xe and $^{133\text{m}}\text{Xe}$, (see Table I). Spectra were collected on site at UT, and an example of the results is shown in fig. 11. Of particular interest is the near-Gaussian shape of the conversion electron peaks. In the case of a gaseous radioisotope, self-absorption of electrons in the source is much reduced, and the dominant source of electron absorption is assumed to be the detector dead-layer. A comparison with the ^{133}Ba electron peaks in figs. 12 and 13 shows the significant improvement with Xenon gas compared to the solid ^{133}Ba source, indicating that there was considerable self-absorption of electrons in the both the needle and plate sources. This result gives strong support to the concept of using a solid state X-ray detector for Xenon detection.

IV. TOWARDS A FIELD-DEPLOYABLE DETECTOR

The work reported so far has demonstrated the capability of solid state X-ray detectors to identify and measure radioactive Xenon gas using X-rays and conversion electrons. A practical, field deployable detector must maximize collection efficiency to reduce the lower limit of detection of Xenon. To this end, a new detector development is under way that will implement the following modifications to the design:

- 1) Confine the gas to a small volume, e.g. 1-2 cm^3 , to reduce the electron path length before interaction with a detector.

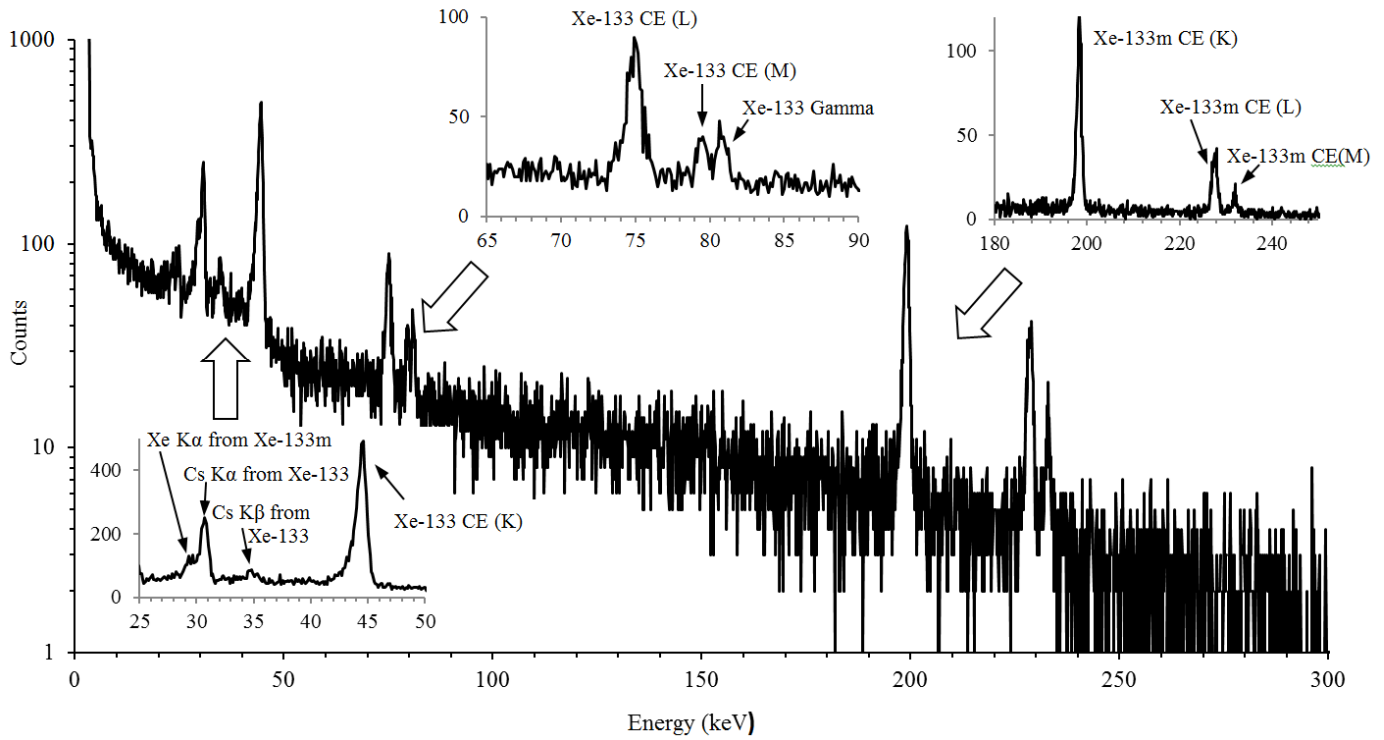


Fig. 11. ^{133}Xe and $^{133\text{m}}\text{Xe}$ spectrum from windowless PIN diode. 3000 sec count.

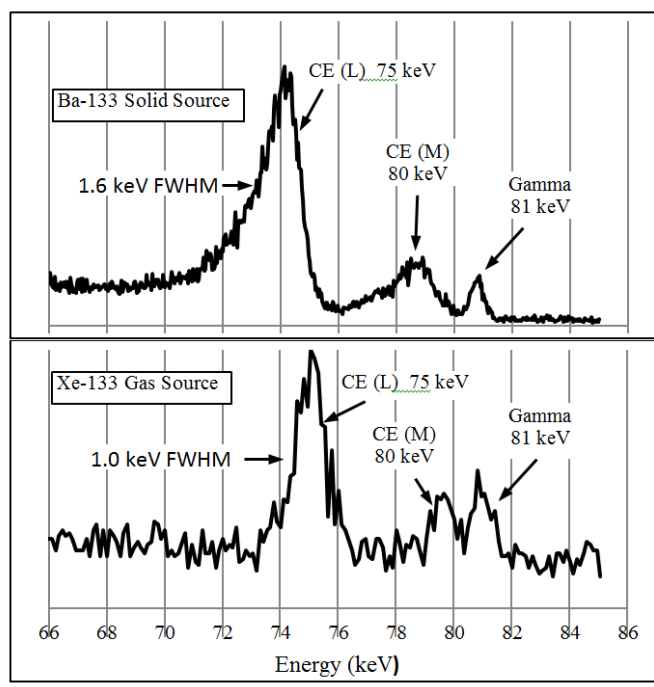


Fig. 12. Comparison of conversion electron peaks around 75 keV from ^{133}Ba solid source and ^{133}Xe gas, using a windowless PIN diode.

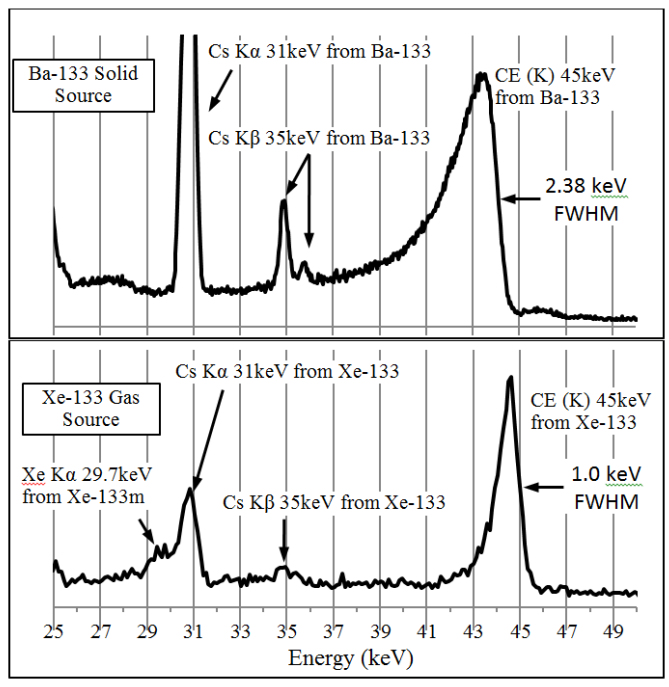


Fig. 13. Comparison of conversion electron peaks around 45 keV from ^{133}Ba solid source and ^{133}Xe gas, using a windowless PIN diode.

- 2). Cover as much of the internal surface area of the trapped volume as possible with closely-packed multiple detectors to provide a solid angle collection efficiency approaching 4π sr.
- 3). Implement coincidence gating between detectors to reduce unwanted counts from background radiation.

Commercially available high resolution PIN diodes are typically packaged on a TO8 header together with the thermoelectric cooler, temperature sensor, Field Effect Transistor (FET) and feedback components. Wire bonds from the header pins to the Silicon chip provide the power and signal connections. This is a convenient package for single detectors, but does not allow multiple detectors to be closely packed together. Therefore, in cooperation with a leading PIN diode manufacturer, a new package is being developed that will incorporate four 25-sq.mm PIN diode chips on a common support approximately 13 mm square. The concept is illustrated in fig. 14. Electrical connections will be made such that the FET and other components are mounted on the rear of the detector. Tests with a standard 25-sq.mm PIN diode indicate that maintaining the detector temperature at 290K will be sufficient to achieve 500 eV FWHM resolution at 30 keV, thereby resolving the 30 and 31 keV peaks from ^{133}Xe and $^{133\text{m}}\text{Xe}$. Since only minimal cooling is required, the temperature of the entire detector assembly can be easily controlled with a thermoelectric Peltier cooler.

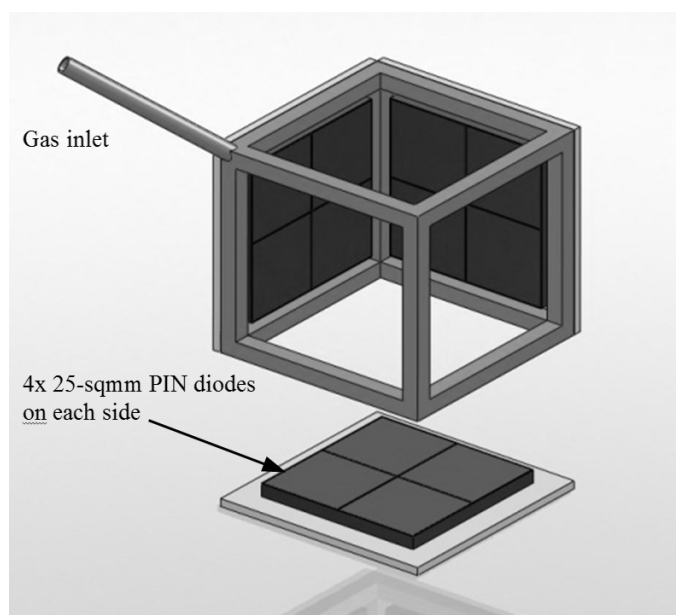


Fig. 14. Concept of 4π geometry multi-element detector for Xenon detection.

The 24-detector array is well suited for the implementation of coincidence gating between the individual detectors. The X-rays and conversion electrons are emitted simultaneously for each event. An X-ray collected in one detector can be gated

against a conversion electron in another, further reducing unwanted background counts. The effectiveness of the gating process increases with the resolution of the peaks.

The perceived advantages of the 24 detector PIN diode array compared to existing Xenon detection systems are as follows:

- Simpler cooling requirements than HPGe detectors.
- Improved energy resolution for K X-ray lines and ability to use additional L lines for Xenon identification.
- Ability to detect ^{37}Ar , which only emits L X-rays, in the same detector.
- Low background due to low detector mass; no shielding required.
- Coincidence gating on highly resolved X-ray and conversion electron peaks reduces the background even further.
- Thin detector entrance window gives greatly improved energy resolution of mono-energetic conversion electrons.
- Silicon is a non-porous metal (oxide), so no memory effect from absorbed Xenon, unlike scintillator detectors.

Fig. 15 demonstrates the energy resolution advantage over an existing room temperature Xenon detection system that uses scintillator detectors. The broad unresolved X-ray and gamma peaks from the scintillator are compared to the highly resolved X-ray and conversion electron peaks from the PIN diode. The disadvantage of the X-ray detector is the loss of gamma-ray detection. Although a detailed analysis is beyond the scope of this work, by calculation [9] it is estimated that the 24-detector system will improve the Xenon detection sensitivity by a factor of two compared to existing room temperature scintillator-based systems.

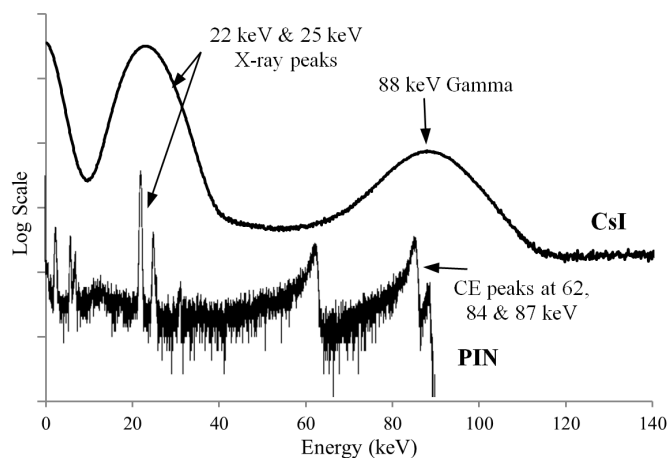


Fig. 15. Comparison of ^{109}Cd spectra from a windowless a PIN diode and a CsI detector used in an existing Xenon detection system.

V. CONCLUSIONS

Modern, state-of-the-art silicon X-ray detectors, even relatively inexpensive PIN diodes, have sufficiently thin dead-layer for energy dispersive spectroscopy with conversion electrons below 100 keV. Using these detectors for the simultaneous collection of high resolution X-rays and mono-energetic electrons offers advantages in the detection of trace amounts of radioisotopes in some applications. The detection of Xenon gas in support of the Comprehensive Test Ban Treaty Organization monitoring program is one such case. We proved the principle of the technique with “open” solid sources, and a subsequent test with Xenon gas gave better energy resolution results than we expected. The design of further detectors is underway, including a cube of 24 PIN diodes that will allow coincidence gating between the detectors to enhance the detection efficiency compared to existing room temperature detector systems.

ACKNOWLEDGMENT

The authors are very grateful to S. Biegalski and T. Tipping at the University of Texas, Austin, for accommodating XIA in the collection of Xe spectra, and to Princeton Gamma-Tech Instruments and Amptek Inc for the provision of windowless Si(Li) and PIN diode detectors, respectively.

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