Development of Compound Semiconductors for Planetary and Astrophysics Space Missions

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ABSTRACT

We discuss the observational requirements for future X-ray planetary and astrophysics missions and present preliminary laboratory results from our compound semiconductor program. The detectors used in the tests were simple monolithic devices, which are used in conjunction with a detailed material science and technology development program intended to produce near Fano limited, pixilated hard X-ray detectors. In practical terms, this means producing active arrays, comprised of over 10^3 pixels each being of order 100 microns in size, with spectral resolving powers, $E/\Delta E > 20$ at 10 keV and high quantum efficiencies over the energy range 1 to 200 keV. Four materials are currently under study - GaAs, HgI₂, TlBr and CdZnTe. In the cases of GaAs and CdZnTe, the detector energy resolution functions are approaching the Fano limit.

Keywords: Compound semiconductors, CdZnTe, GaAs, HgI₂, TlBr, X-ray astronomy

1. INTRODUCTION

With the recent launch of the Chandra X-ray advanced astrophysics facility (formerly AXAF) and the Newton X-ray Multi Mirror Mission (formerly XMM), X-ray astronomy has emerged as a fully developed field of observational astronomy. While it still lags far behind optical astronomy (in fact, in terms of collecting power and spatial resolution X-ray telescopes are still only comparable with late 19th century optical instruments) dramatic progress has been made in the last two decades. This is largely due to the parallel developments of grazing incidence optics and positional sensitive detection planes. In particular, advances at soft X-ray wavelengths have been spectacular since the

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introduction of X-ray sensitive silicon CCD's coupled to Wolter 1 mirror systems. However, at hard X-ray energies (e.g., > 10 keV) progress has been much slower due to the increasing inefficiency of grazing incidence optics, coupled with the poor detection efficiencies of Si based detectors. While Ge devices could in principle provide good detection efficiency above 10 keV, such systems are so constrained by cryogenic and fabrication problems as to be of limited use in space borne applications. Unfortunately, the energy range above 10 keV also represents the important transition region from thermal to non-thermal emission processes. In astrophysics, non-thermal X-rays arise from the accretion of material onto compact objects such as a neutron stars or black holes, or from cyclotron and synchrotron emissions from relativistic electrons moving in strong magnetic fields. Also present can be line emissions, absorption features, Compton degraded features and reflection components. Such features provide a direct channel with which to study particle and field densities as well as the geometry and nature of the emission environment.

In planetary physics, fluorescent imaging of X-rays excited by the Sun from solar bodies can yield scientifically important composition maps. At present there is a paucity of such data. Chemical composition maps are crucial in the determination of not only the geochemical nature of planetary surfaces but also chronicle the history of volcanotectonic and impact activity on these bodies. Semiconductor materials have been used in

Parameter	Si	GaAs	Ge	Cd _{1-x} Zn _x Te	HgI ₂	TlBr
Density (g cm ⁻³)	2.33	5.32	5.33	~6	6.4	7.56
Average atomic number	14	32	32	41-50	62	58
Band gap (eV)	1.12	1.43	0.67	1.5-2.2	2.15	2.68
Pair creation energy (eV)	3.62	4.2	2.96	~5.5	4.2	6.5
Electron mobility (cm ² V ⁻¹ s)	1400	8000	3900	1350	100	7.7
Hole mobility (cm ² V ⁻¹ s)	1900	400	1900	120	4	?
Electron lifetime (s)	>10 ⁻³	10 ⁻⁸	>10 ⁻³	1 x 10 ⁻⁶	10 ⁻⁶	2.5 x 10 ⁻⁶
Hole lifetime (s)	10 ⁻³	10 ⁻⁷	2 x 10 ⁻³	5 x 10 ⁻⁸	10 ⁻⁵	?
Electron $\mu\tau$ product (cm ² V ⁻¹ s)	>1	8 x 10 ⁻⁵	>1	1 x 10 ⁻³	10 ⁻⁴	5 x 10 ⁻⁴
Hole $\mu\tau$ product (cm ² V ⁻¹ s)	~1	4 x 10 ⁻⁶	>1	1 x 10 ⁻⁶	4 x 10 ⁻⁵	2 x 10 ⁻⁶
Crystal structure	Cubic	Cubic (ZB)	Cubic	Cubic (ZB)	Tetragonal	Cubic (CsCl)
Knoop hardness (kg mm ⁻²)	1150	750	692	?	<10	12
Melting point (°C)	1412	1238	958	1092-1295	259	460
Dielectric constant	11.7	12.8	16	?	8.8	29.8
Resistivity (Ω -cm)	<10 ⁴	10 ⁷	50	3 x 10 ¹⁰	10 ¹³	10 ¹²
1/e abs depth (mm) @ 10 keV	0.118	0.049	0.050	0.011	0.011	0.118
@ 100 keV	23.19	3.46	3.51	1.01	0.46	0.32
Typical fwhm ΔE @ 60 keV (keV)	0.4	0.7	0.3	1.5	3.5	7.9
Typical thickness	0.3	0.2	20	2	10	1

Table I. Compilation of physical properties of materials considered in this study, grouped according to their density. For comparison, the properties of the elemental semiconductors, Si and Ge are also listed.

alpha, proton and X-ray spectroscopy, in both remote and direct sensing experiments. Direct sensing measurements are extremely difficult to carry out, since they require a lander and as such, remote sensing is technologically simpler. However, while remote sensing missions have studied the moon, they suffer from limited sensitivity and crude

spatial resolution. This is because, up till now, planetary science has lacked short focal length optics matched to a thin but highly absorbent detection plane, which for interplanetary missions must also be free of the power, weight and cryogenic constraints normally associated with traditional detection technologies. Recently, new developments in slumped micro channel plate technology and wide band-gap compound semiconductors offer the potential of circumventing these problems, providing spatially resolved, Fano limited spectroscopy across the energy range 1-200 keV.

The Space Science Department of ESA has worked for a number of years to produce monolithic compound semiconductor detectors and large area arrays for future space missions. Several materials are under study including; GaAs, CdZnTe, TlBr and HgI₂. These materials have band-gaps sufficiently high so they do not require cryogenic cooling but low enough that sub-keV spectral resolution may be achieved at hard X-ray energies. In addition, the materials are drawn from group III and V compounds and so have high enough atomic numbers to ensure high detection efficiencies above 10 keV. The general properties of these materials are listed in Table I, compared to Si and Ge. In this paper, we present results of the X-rays testing of several prototype detectors. Preliminary results from both the material science and detector program have been reported elsewhere [1-8].

Figure 1. The standard detector housing used for all monolithics.

2. EXPERIMENTAL

The detectors are packaged in standard housings shown in Fig. 1. They are mounted on ceramic holders which are coupled to two stage Peltier coolers capable of cooling the detectors to -35°C . The hot side of the Peltier can be water cooled allowing the detectors to be cooled an additional -10°C . The temperature of each detector was set to that which gave optimum performance. For example, GaAs detectors were operated at -45°C , HgI_2 at -20°C , CdZnTe at -40°C and TlBr at -30°C . From Table I, we note that, in general, the mobility-lifetime product ($\mu\tau$) of electrons is much greater than that for holes. Therefore, negative bias was applied to the upper contact to ensure the efficient collection of holes. The signals are extracted from the lower contact by the pre-amplifier input FET, which is also maintained at the detector operating temperature. The rest of the analogue chain consists of a resistive feedback pre-amplifier used in conjunction with an ORTEC[®] 671 spectroscopy amplifier. Both the signal amplitude and rise time are digitized by a pair of Canberra 8071 12-bit ADC's and processed and stored by a PC. X-ray measurements were carried out in our laboratories using a set of standard radioactive (^{55}Fe , ^{57}Co , ^{109}Cd , ^{241}Am) and fluorescent target (Cu, Rb, Mo, Ag, Ba and Tb) sources.

Figure 2. The temperature dependence of the FWHM energy resolution at 5.9 keV measured with a 7mm^2 , 0.5 mm thick HgI_2 detector under full beam illumination. The solid line is a best-fit cubic polynomial.

2.1 Mercuric Iodide

Mercuric Iodide (HgI_2) has been investigated as a room temperature X- and gamma ray detector since the early 1970's [9,10]. Its wide bandgap in conjunction with the high atomic number of its constituent atoms makes it an attractive material for room

temperature operable X- and gamma-ray spectrometers. In fact, since photoelectric absorption varies as z^5 , the specific sensitivity of HgI_2 is about 100 greater than germanium for energies < 200 keV. HgI_2 is a relatively soft material that forms a tetragonal lattice at temperatures below 130°C . The crystals appear red in colour (alpha phase) with a band-gap of energy of 2.13 eV.

The detector used in the present study has an area of 7 mm^2 and thickness 0.5 mm. It was grown by vapour phase deposition using the vertical ampoule method. After cutting, the detector was etched in methanol and cleaned with de-ionized water. Palladium contacts were then deposited by vacuum evaporation and the crystal coated with a thin (few microns) polymer coating for surface passivation and to prevent evaporative degradation. The leakage current was measured to be < 100 pA at the nominal operating bias of -800 V and the detector capacitance was 3 pF.

Figure 3. HgI_2 energy spectrum measured with an ^{241}Am source under full-illumination at a detector temperature of 245 K (-19.2°C). The pulser width is 343 eV FWHM.

Using an ^{55}Fe source, the detectors response to bias was investigated. The peak position of the $\text{K}\alpha$ X-ray was found to vary weakly with bias – the percentage change being 6×10^{-3} per volt bias below -400 V. For X-rays, no significant change in peak position or energy resolution was found for biases below -700 V. Similarly, varying the shaping time

showed that 6 μ s was optimum. A bias of -800 V and shaping time of 6 μ s was chosen for subsequent measurements. The iron line FWHM energy resolution, measured as a function of detector temperature is shown in Figure 2, from which we can see there is a minimum near -20°C . From the residuals of a best-fit line centre energies of the lines from six fluorescent target sources (Cu, Rb, Mo, Ag, Ba and Tb) and two radioactive sources, the detector non-linearity was determined to be no worse than 1% over the energy range 5.9 to 59.5 keV.

The global response of the detector can be seen in Figure 3 in which we show a spectrum resulting from exposure to an ^{241}Am radioactive source. The low energy threshold is set by the leakage current at ~ 800 eV. The energy resolution is such that the L-edge sequence of Np lines can be clearly resolved. The additional lines near the Np-Ly complex and the Am-26.3 keV line are due to Ag $K\alpha$ and $K\beta$ fluorescence. Ag is used in the packaging of the source. The FWHM energy resolution and detection efficiency for the principal line at 59.5 keV is 1.5 keV and 85% respectively. The pulser FWHM is 343 eV; thus the detector contributes most of the noise at the higher energies. For comparison, the measured room temperature energy resolution at 59.5 keV is 2.5 keV FWHM.

Figure 4. The measured energy resolution ΔE under full illumination at room temperature. The upper solid line shows the best-fit resolution function to the combined data set. The individual components of the FWHM are also shown, viz. ΔF , Δe and Δc (see text). The lower solid line is the resolution function determined at a detector temperature of -19.2°C . The individual data points are not shown for clarity.

The measured energy resolution of the system is shown in Figure 4. The resolution was found to be well fit by a function of the form;

$$\Delta E = 2.355\sqrt{\sigma_F^2 + \sigma_e^2 + \sigma_c^2} \quad (1)$$

where, σ_F^2 is the variance of the noise due to carrier generation or Fano noise ($\sigma_F^2 = FE\varepsilon$), σ_e^2 is the variance of the noise due to the leakage current and amplifier shot noise and σ_c^2 is the variance of the noise due to incomplete charge collection due to carrier trapping. The upper solid line shows the best-fit resolution function to the combined data set taken at room temperature. For comparison, the individual components of the FWHM are also shown (*i.e.*, ΔF , Δe and Δc), assuming a Fano factor of 0.12. From the figure, we see that electronic noise dominates the resolution function below ~ 10 keV whereas trapping noise dominates above this energy. The lower solid line shows the measured resolution at the temperature minimum in Figure 2 (*i.e.*, -19.2°C).

By accumulating a series of ^{241}Am spectra, 1 hour, 2 hours, 12 hours and days after the application of bias, the effects of polarization were investigated. Polarisation is usually defined as any time dependent change, which occurs in the performance of a detector and is generally a bias related effect. In HgI_2 , polarisation effects are believed to stem from trapping in the bulk of the crystal and from non-optimal detector fabrication [11,12]. It was found that polarization effects manifest themselves primarily as an increase in the level of the low energy continuum, which gradually decays with time after the application of bias. This can be more than a factor of 3 at 10 keV over 12 hours. In fact, it was found that reproducible results could be obtained by applying bias at least 12 hours before measurement.

2.2 Thallium Bromide

TlBr is the densest material under study ($\rho=7.6 \text{ g cm}^{-3}$). It has a CsCl-type cubic lattice structure with physical properties amenable to easy and rapid purification using standard growth techniques. It melts congruently at 480°C with a single phase transition, allowing good quality crystals to be grown directly from melt. The detectors studied here were cut from a thermally grown monocrystal. The boule was sawn into several 1 and 2 mm thick slices and a number of detectors diced from the wafers. The typical crystal dimensions were $2.8 \times 2.8 \times 0.5 \text{ mm}^3$. The samples were lapped followed by mechanical and chemical polishing. From the I-V characteristics, resistivities were found to be in the range $(6 - 10) \times 10^{10} \Omega \text{ cm}$. At nominal biases of a few hundred volts, the average recorded leakage currents were $\sim 10 \text{ nA}$ at room temperature and 0.1 nA at -2°C . After initial stability and noise tests of a large number of devices, several were packaged into detectors. Initial tests showed that biasing the detectors to preferentially collect holes gave significantly better spectral resolution than collecting electrons. Furthermore, no optimum operating temperature was found when collecting electrons, whereas for holes, an optimum temperature of -30°C was found.

Figure 5 shows one of the detectors response to an ^{241}Am source. This detector was patterned with circular Au contacts (1.7 mm^2) and operated at a bias of -400V . The

measured energy resolution at -24°C is 2.6 keV FWHM for the principal gamma-ray line at 59.5 keV (pulser width 680 eV). The same detector yielded a FWHM energy resolution for the 5.9 keV line from ^{55}Fe of 816 eV (pulser width 690 eV).

Figure 5. The response of a 1.7 mm², 0.5 mm thick TlBr detector to ^{241}Am . The detector temperature was -24°C and the pulser noise width 680 eV FWHM.

The mobility-lifetime product for electrons $(\mu\tau)_e$ was determined using the Np $L\alpha$, $L\beta$ and $L\gamma$ X-ray peaks of Am^{241} at 13.9, 17.5 and 21.0 keV and the nuclear line at 26.32 keV. The photopeak pulse heights were measured as a function of detector bias and fit to a single carrier Hecht equation [13] given by,

$$H / H_o = ((\mu\tau)_e V / d^2) \{1 - \exp(-d^2 / (\mu\tau)_e V)\} \quad (1)$$

where H is the measured pulse height, H_o is the pulse height that would be obtained if the detector was 100% efficient, V is the bias potential applied to the detector and d is the thickness. Assuming that, $d^2/(\mu\tau)_e V \ll 1$, then $H/H_o \propto 1/V$ and $(\mu\tau)_e$ is related to the slope of H/H_o versus $1/V$. The derived $(\mu\tau)_e$ product is $(2.3 \pm 0.3) \times 10^{-4} \text{ cm}^2 \text{ V}^{-1}$ at -30°C which is about an order of magnitude higher than previously reported values [14].

2.3 Cadmium Zinc Telluride

CdZnTe is a ternary compound that has been extensively studied over the last decade at X- and γ -ray wavelengths. It has a cubic, zincblende type lattice with atomic numbers close to that of CdTe and a density ~ 3 times that of Si. CdTe was originally the focus of experimental study in the 1960's, until it was discovered that the addition of a few percent of zinc to the melt results in an increased band-gap as well as the energy of defect formation. This in turn increases bulk resistivities and reduces the dislocation density, resulting in lower leakage currents and higher temperature operation. Additionally, CdZnTe tends not to suffer from the polarization effects normally associated with CdTe.

Figure 6. The measured response of a 3.1 mm², 2.5 mm thick CdZnTe detector to ²⁴¹Am. The detector temperature was -37°C

Two detectors were studied in the present study. They were grown by the high pressure Bridgman (HPB) method. Spectroscopic grade crystals were then selected from the bulk material by I-V and IR microscopy screening. After cutting and polishing in a 5% bromide/methanol solution, platinum contacts were deposited by electrode-less deposition - platinum being chosen for its superior durability and adhesion properties. A circular anode (2 mm ϕ) and concentric guard ring structure were used for the irradiated contact to minimise surface leakage current. After surface treatment, the leakage currents were typically less than 1 nA at -100 V bias.

To achieve electronic noise levels less than 20 electrons rms, the detector utilises an experimental resistor-less feedback preamplifier design. Figure 6 shows the detectors response to ^{241}Am measured at a detector temperature of -37°C . Both the shaping time (10 μs) and the detector bias (-600V) were chosen so as to optimize the FWHM energy resolution at this temperature. The measured energy resolution at 59.5 keV was 824 eV FWHM and the corresponding pulser resolution 280 eV FWHM. The Fano noise at this energy is expected to be around 500 eV FWHM.

Figure 7. The response of a 0.8mm^2 , 40 micron thick GaAs detector to ^{241}Am . The detector energy resolution is 830 eV at 59.5 keV. The electronic pulser width is 438 eV FWHM.

2.4 Gallium Arsenide

In the last decade, gallium arsenide (GaAs) has attracted considerable interest as a viable alternative to Si or Ge for the detection of X-rays above a few keV. It has a simple cubic lattice structure and is one of the high group compound semiconductors with a bandgap sufficiently wide (1.42 eV) to permit room temperature operation but small enough so that its Fano limited spectroscopic resolution is close to that of Si. GaAs detectors have been fabricated using chemical vapor phase deposition (CVPD) techniques. A variety of detector configurations were patterned onto both 40 micron and 400 micron material epitaxial grown onto a 400 μm thick n+ GaAs substrate. A pin structure is formed by depositing a 5 μm thick p+ layer directly onto the epitaxial layer and then depositing a full ohmic contact (Ni/Au/Ge/Au) on the n+ side. The p+ side is patterned by etching to define detector (mesa) and guard ring structures. These are then metalised with alternate

depositions of Au/Pt/Ti, forming Schottky contacts. From the I/V characteristics, the typical current densities at -100V were $< 0.04 \text{ nA/mm}^2$ at room temperature. This is about a factor of 3-100 lower than normally obtained with CVPD detectors and a factor of 10^3 - 10^4 lower than bulk detectors.

Figure 7 shows the response of a 0.8 mm^2 , $40 \text{ }\mu\text{m}$ thick detector to ^{214}Am . The detector bias was -160 V and the temperature -40°C . A shaping time of $3 \text{ }\mu\text{s}$ was used in these measurements. The measured energy resolution at 59.5 keV was 533 eV FWHM with a pulser noise width of 470 eV FWHM . Lastly, Fig. 8 shows an ^{55}Fe spectrum taken with a pixel detector ($250 \times 250 \times 40 \text{ }\mu\text{m}^3$) from our latest production run. The detector utilised a resistor-less feedback preamp design which gives a significant improvement in system noise. For example, using the conventional preamplifier used by the previous detector resulted in a resolution of 394 eV FWHM at 5.9 keV and a detector temperature of -40°C (pulser width= 378 eV). With the new preamplifier, the FWHM energy resolution at -31°C is 219 eV at 5.9 keV with a pulser resolution of 163 eV . The corresponding resolutions at room temperature were 266 eV and 242 eV , respectively. The derived Fano factor at -31°C is thus $F=0.15$, assuming zero trapping noise.

Figure 8. ^{55}Fe spectrum measured with a $250 \times 250 \times 40 \text{ }\mu\text{m}^3$ GaAs detector at a temperature of -31°C .

3. DISCUSSION AND CONCLUSIONS

While the performances of most compound semi-conductors are not yet close to the Fano limit, theoretically they are close to satisfying the requirements of many of the anticipated future space missions. In Table II, we list some of the best resolution obtained with our detectors. Our results show that HgI_2 and TlBr detectors of reasonable detection area are useful as room temperature X-ray spectrometers down to 1 keV . Except for GaAs, the shape of photopeak in all other detector materials becomes increasingly tailed for

energies above 30 keV. This is primarily due to the geometrical effect of hole trapping coupled with the relatively low values of the hole mobility-lifetime product. Below ~30 keV the radiation is absorbed within a sufficiently short distance within the active region for the majority of holes to be collected, even with the transport properties of present material. Hole collection problems therefore limit the current useful thickness (and ultimately high energy performance) of these detectors to about 0.5-1.0 mm. For low noise systems, the thickness of the detector must also be traded-off with detector capacitance, which directly affects the electronic noise of the system. The fact that detector performance can be significantly improved with only modest cooling has important consequences for planetary spacecraft where power, mass and volume are at a premium. Such cooling can be readily achieved with Peltier systems or passive radiator panels alleviating the need for complex and expensive cryogenic systems - as in the case of Ge or Si detectors. Lastly, for HgI₂, the effects of polarization can be largely negated by applying bias at least 12 hours before measurements begin.

Material	Detector size Area × thickness	ΔE @ 5.9 keV (eV)	ΔE @ 59.5keV (eV)
TlBr	1.7 mm ² , 0.5 mm	650 @ -30°C	2300
HgI ₂	7 mm ² , 0.5 mm	438 @ -19°C	1540
CdZnTe	3.1 mm ² , 2.5 mm	311 @ -37°C	824
GaAs 1	0.8 mm ² , 40 μm	533 @ -40°C	830
GaAs 2	250 × 250 × 40 μm ³	266 @ RT (+23°C)	479

Table II. Some of the best energy resolutions achieved with prototype detectors. The measurements were carried out under uniform illumination with ⁵⁵Fe and ²⁴¹Am sources. For the iron line measurements, the equivalent FWHM pulser resolutions were, 574 eV (TlBr), 343 eV (HgI₂), 290 eV (CdZnTe), 470 eV (GaAs 1), 242 eV (GaAs 2). The detector temperatures at 59.5 keV are the same as those listed for 5.9 keV.

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