X-ray Measurements with Compound Semiconductor Arrays

A. Owens¹, H. Andersson², M. Bavdaz¹, G. Brammertz¹, T. Gagliardi³, V. Gostilo⁴, I. Lisjutin⁵,
A. Loupilov⁵, I. Major³, Y. Mastrikov⁴, S. Nenonen², A. Peacock¹, A. Poelaert¹, H. Sipila², L.
Tröger⁵

¹*Astrophysics Division, Space Science Department, ESA/ESTEC, P.O.Box 299,
2200 AG Noordwijk, The Netherlands*

²*Metorex International Oy, P.O.Box 85, FIN-02201 Espoo, Finland*

³*Metorex Inc., 250 Philips Boulevard, Ewing, NK 08618 United States of America*

⁴*Baltic Scientific Instruments, 26 Ganību dambis, P.O.Box 33, Riga LV-1005, Latvia*

⁵*HASYLAB at DESY, Notkestrasse 85, D-22607 Hamburg, Germany*

ESLAB 2000/052/SA

To be published in the proceedings of the SPIE International Symposium
on Optical Science and Technology,
30 July - 4 August 2000, San Diego, United States of America

6622222

X-ray measurements with compound semiconductor arrays

Alan Owens^{a1}, H. Andersson^b, M. Bavdaz^a, G. Brammertz^a, T. Gagliardi^c,
V. Gostilo^d, I. Lisjutin^d, A. Loupilov^d, I. Major^e, Y. Mastrikov^d, S. Nenonen^b,
A. Peacock^a, A. Poelaert^a, H. Sipila^b, L. Tröger^e

^aAstrophysics Division, ESA/ESTEC, Postbus 299, 2200AG Noordwijk, Netherlands

^bMetorex International Oy, P.O. Box 85, FIN-02201 Espoo, Finland

^cMetorex Inc., 250 Philips Blvd, Ewing, NJ 08618, USA

^dBaltic Scientific Instruments, 26 Ganību dambis, P.O. Box 33, Riga LV-1005, Latvia

^eHASYLAB at DESY, Notkestrasse 85, D 22607 Hamburg, Germany

ABSTRACT

We present preliminary results of X-ray measurements on two small format compound semiconductor arrays. The devices, a 5×5 gallium arsenide array and a 3×3 cadmium zinc telluride array, were produced specifically to address the material, electronic and technological problems that need to be solved in order to develop mega-pixel, Fano limited spectroscopic arrays. The GaAs device was fabricated on $40 \mu\text{m}$ epitaxial material and has a pixel size of $200 \times 200 \mu\text{m}^2$ with pitch $250 \mu\text{m}$. The CdZnTe array was fabricated on a $5 \times 5 \times 1.6 \text{ mm}^3$ single crystal of spectroscopic quality. The pixel sizes were $350 \times 350 \mu\text{m}^2$ with a pixel pitch of $250 \mu\text{m}$. Measurements from 5.9 keV to 100 keV were carried out both in our laboratory and at the HASYLAB synchrotron research facility in Hamburg, Germany. The typical FWHM energy resolutions recorded at 5.9 keV by the GaAs and CdZnTe arrays were 394 eV and 900 eV, respectively.

Keywords: Compound semiconductors, CdZnTe, GaAs, arrays, X-rays

1. INTRODUCTION

In recent years, considerable effort has been expended in producing X-ray imaging detectors with high spectral and spatial resolution. In practical terms, this means producing pixelated detectors, comprised of $> 10^3$ pixels each of order 100 microns in size, with spectral resolving powers, $E/\Delta E > 20$ at 10 keV. While such devices already exist at soft X-ray wavelengths (largely due to the introduction of X-ray sensitive CCDs) progress at higher energies has been slow. This is because traditional high resolution detectors either suffer from poor detection efficiencies above 10 keV, as in the case of Si

¹ Correspondence: Email: aowens@astro.estec.esa.nl; Telephone: +31 71 565 5326; Fax +31 71 565 4690

based technology, or are very constrained by cryogenic and fabrication problems in the case of Ge based detectors. Recent developments in wide band-gap compound semiconductors have shown that such materials may bridge Ge and Si technologies. The band-gap in these materials is high enough so that they do not require cryogenic cooling but low enough that sub-keV spectral resolution is achievable at hard X-ray energies. In addition, materials drawn from groups II to VI of the periodic table, have high enough atomic numbers to ensure high stopping powers and therefore high detection efficiencies above 10 keV.

In this paper, we present preliminary results of X-ray characterizations of two small format compound semiconductor arrays – a 5×5 GaAs array and a 3×3 CdZnTe array. The measurements were carried out over the energy range 12 keV to 98 keV at the HASYLAB synchrotron research facility in Hamburg, Germany. The goal of this work was assess the feasibility of producing such arrays for future ESA astrophysics and planetary missions. Preliminary results from our material science and detector programs have been reported elsewhere [1-5].

2. EXPERIMENTAL

X-ray experiments were carried out, both in our laboratory and at the X-1 beamline at the HASYLAB synchrotron research facility. This beamline utilizes a channel-cut double Si crystal monochromator to produces a highly monochromatic X-ray beam across the energy range 12 keV to 98 keV [6]. To achieve such an extreme energy range, a [511] reflection is used (because of the more favourable lattice spacing), yielding an intrinsic energy resolution of ~ 1 eV at 10 keV rising to 20 eV at 100 keV. The beam spot size used in the measurements was $20 \times 20 \mu\text{m}^2$ and is set by a pair of slits located in front of the detector. For completeness, additional full-area illumination measurements were also carried out using ^{55}Fe and ^{241}Am radioactive sources. Detectors are mounted on an X-Y table capable of positioning the sensitive elements to a precision of $<1 \mu\text{m}$ in each axis.

2.1 Gallium arsenide array

In recent years, 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 compound semiconductors with a band-gap 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.

A small GaAs array was produced by growing an ultra pure $40 \mu\text{m}$ epitaxial layer onto an n+ semi-insulating substrate using chemical vapor phase deposition (CVPD) techniques. To reduce leakage currents, a $10 \mu\text{m}$ thick p+ layer was then deposited

Figure 1. Photograph of the 5×5 GaAs detector assembly used in the present study. The device is bonded to a ceramic substrate, which in turn is mounted on a two stage Peltier cooler. A Nomarski photomicrograph of the array is shown in the upper left-hand corner.

directly onto the epi-layer, effectively forming a p-i-n structure. This layer was then patterned by etching to form a 5×5 pixel structure surrounded by a guard ring (see figure 1). These were metallized with alternate depositions (Au/Pt/Ti), forming Schottky contacts. A full ohmic contact (Ni/Au/Ge/Au) was applied to the n+ substrate. The pixel sizes are $200 \times 200 \mu\text{m}^2$ and the inter-pixel gap, $50 \mu\text{m}$. The inter-pixel resistivity was measured to be $7 \times 10^{12} \Omega$. The guard is $200 \mu\text{m}$ wide and is operated at ground potential. The typical leakage current at a bias of 100 V was determined to be 0.004 nA at room temperature.

Contacting to the chip is achieved by wire bonding. Two pixels were actually instrumented – these will be referred to as the center and right pixels (see the inset in fig. 1). The chip is bonded to a ceramic substrate. To ensure low noise, the preamplifier input FETs are also mounted on the substrate, close to the array. The entire assembly is mechanically coupled to a two stage Peltier cooler (capable of cooling the array to -40°C) and enclosed within a vacuum vessel. X-rays are incident on the array through a thin ($25\mu\text{m}$) Be window.

Figure 2. The response of the center pixel to an ^{55}Fe source under full-area illumination at -40°C . The measured energy resolution is 394 eV at 5.9 keV with an electronic noise component of 378 eV FWHM. The corresponding energy-loss distribution for the other pixel is the same within statistics.

The initial characterization of the device was made using ^{55}Fe and ^{241}Am radioactive sources. The optimum performance in terms of energy resolution was found using pixel biases of +100 V and electronic shaping times of 2 μs . The leakage currents were low enough to permit room temperature operation, with measured energy resolutions at 5.9 keV of 660 eV FWHM for both pixels (pulsar line width = 470 eV). Cooling the detector to -40°C resulted in a 70% reduction in ΔE to 394 eV FWHM. In this case, the electronic system noise was 378 eV FWHM. The measured ^{55}Fe energy-loss spectrum of the center pixel is shown in Figure 2 under full area illumination.

In figure 3, we show a composite of energy-loss spectra recorded at HASYLAB by the center pixel over the energy range 12 keV to 98 keV. Plots for the right pixel data are statistically identical, both spectrally and in terms of resolution. The response of both pixels is remarkably linear with regression coefficients for a best-fit linear function to the peak channel number versus incident X-ray energy, in excess of 99.99% over the entire energy range. The smaller peaks in the spectrum, which appear at energies of 10.4 keV and 11.9 keV below the photopeaks, are due to the Ga and As escape peaks, while the peaks near 10 keV are due to X-ray fluorescence from the inert p+ layer above the

depletion depth. The noise threshold (defined to be the lowest energy for which the low energy noise attains an amplitude of 0.5 of the photopeak) is 1.3 keV for both pixels.

Figure 3. Composite pulse height spectra of monoenergetic lines measured by the center pixel at HASYLAB. The beam size was $20 \times 20 \mu\text{m}^2$, incident at the center of the pixel. The enhancement at low energies is due to Compton scattering at the higher energies.

From fig. 3, we can also see that, apart from Compton features below 30 keV, the level of continuum is extremely low, being $\sim 1\%$ of the photopeak amplitude. In fact, the peak to total ratios are 71% at 12 keV, 56% at 30 keV, 35% at 60 keV and 21% at 98 keV.

The energy resolution function of the center pixel was determined from the pulse height data and is shown in fig. 4. The FWHM resolutions range from ~ 400 eV at 5.9 keV to 700 eV at 98 keV. Those measured for the right pixel were statistically identical. The electronic noise of the system, as measured by a precision pulser was 380 eV FWHM for both pixels. The energy 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 ($\sigma_F^2 = FE\epsilon$), 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 resolution was also measured under full-area illumination using an ^{241}Am source. The resolutions for the 59.5 keV nuclear line and 60 keV HASYLAB pencil beam radiation were found to be the same within statistics, thus indicating uniform crystallinity and stoichiometry.

Figure 4. The energy dependent fwhm energy resolution of the center pixel measured at HASYLAB under pencil beam illumination. The upper solid line shows the best-fit resolution function to the combined data set. The lower dashed line shows the pulser subtracted energy resolution which can be directly compared to the expected resolution due to Fano noise alone (dotted curve), assuming a Fano factor of 0.18 (Eberhardt et al. [7]).

In figure 5, we have again plotted the pulser subtracted energy resolution to which we have fit the expected noise function due to carrier statistics, with the Fano factor treated as a free parameter. A best-fit χ^2 of 11.4 for 13 degrees of freedom was obtained for a Fano factor of 0.144 ± 0.005 - the error being purely statistical. The lower panel of the figure shows the fit residuals (*i.e.*, (measured-fit) / fit $\times 100\%$), from which we can see, that apart from a slight excess at 20 keV, there is no systematic trend with energy. This illustrates the quality of the fit and that, to first order, all major noise components have been correctly accounted for. Note, no account has been made for trapping noise and indeed the residuals would suggest that its contribution is very low - or alternately, the charge collection efficiency (CCE) is very high. In fact, a best-fit resolution function described by equation. 1 (which includes Fano, trapping and electronic noise components), indicates that the CCE must be in excess of 99.6%.

Figure 5. A plot of the best-fit Fano noise function to the pulser subtracted energy resolution. The lower panel shows the residuals, which are consistent with zero, indicating that to first order, all major noise contributions have been correctly accounted for.

Figure 6. The variation of the count rate (a) and the gain (b) across the center and right pixels measured at HASYLAB. The incident energy is 12 keV and the spatial sampling in X and Y, 10 μm . The beam spot size was $20 \times 20 \mu\text{m}^2$. Note the attenuation in the bond wires can be seen in (a).

Lastly, the spatial response of the detector was evaluated using a 12 keV pencil beam of size $20 \times 20 \mu\text{m}^2$, normally incident on the pixels. The beam was raster scanned across this area with $10 \mu\text{m}$ spatial resolution in both dimensions. Spectra were then accumulated at each position for a fixed time interval and the total count rate above a 3 keV threshold, the peak centroid position and the FWHM energy resolution determined by best-fitting. It was found that the count rate, peak centroid and energy resolution of individual distributions, were very uniform and are consistent with a flat response – *i.e.*, the variations seen in each distribution being consistent with the expected statistical variations, *i.e.*, 2% for the count rate distribution, 0.14% for the peak channel distribution and 5% for the energy resolution distribution. A spatial map showing the variation of the measured count rate and the centroid determination (*i.e.*, the gain map) is shown in fig. 6.

Figure 7. Close-up photograph of the CdZnTe detector assembly with the front covers and Be window removed. The array lies in the center of the photograph. A Nomarski photomicrograph is shown in the upper right hand corner. Note, in operation the array is actually illuminated from the other side. The pixels are numbered 1 to 9 from left to right, top to bottom.

2.2 Cadmium zinc telluride array

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.

A CdZnTe array was fabricated on a $5 \times 5 \times 1.6 \text{ mm}^3$ spectroscopic grade crystal, grown by the high pressure Bridgeman (HPB) method. This crystal was selected from the bulk material by I-V and IR microscopy screening and, after cutting and polishing in a 5% bromide/methanol solution, a 3×3 gold contact structure was deposited following photolithography. The pixel sizes were $350 \times 350 \mu\text{m}^2$ with an inter-pixel gap of $50 \mu\text{m}$. After surface treatment, the leakage currents were typically less than 2 nA at -200 V bias and the inter-pixel resistivities $\sim 60 \text{ G}\Omega$. All 9 pixels were instrumented - contacting being achieved by bond wires. The array is shown in detail in the inset of figure 7 and is mounted (upside down) on a thermally conductive daughter board. This in turn is mounted on a motherboard, mechanically coupled to a thermoelectric cooler, which is capable of cooling the device to -30°C . The entire assembly is contained within a vacuum vessel, which is partly shown in figure 7. The preamplifiers are an integral part of the detector assembly, although located outside the vacuum vessel.

Figure 8. ^{55}Fe spectrum measured by pixel 9 at a temperature of -25°C .

As with the GaAs array, the initial characterization of the array was carried out using ^{55}Fe and ^{241}Am radioactive sources. A pixel bias of -250V was chosen with a shaping time of $10 \mu\text{s}$. Although, the leakage currents were low enough to permit room temperature operation, the device was operated at -24°C for subsequent measurements. Fig. 8 shows the response of pixel 9 to the ^{55}Fe source. The distributions for the other pixels were

Figure 9. Composite pulse height spectra of monoenergetic lines measured by pixel 6 at HASYLAB. The beam size was $20 \times 20 \mu\text{m}^2$, incident at the center of the pixel.

Figure 10. The energy dependent fwhm energy resolution of pixels 6 and 8 measured at HASYLAB under pencil beam illumination. These two pixels gave the best and worst resolution figures. Thus, the resolutions of the other pixels fall within these lines.

Figure 11. The spatial response of the CdZnTe array measured at HASYLAB. The incident energy is 12 keV and the spatial sampling in X and Y, $10\text{ }\mu\text{m}$. The beam spot size was $20 \times 20\text{ }\mu\text{m}^2$. (a) shows the variation in count rate profile above a 3 keV threshold, (b) the fitted centroid of the photopeak (*i.e.*, gain map), and (c) the energy resolution expressed in terms of resolving power. Note, for display purposes the pixels have been separated by an offset of $\sim 200\text{ }\mu\text{m}$ – otherwise the individual distributions merge.

similar. The measured FWHM energy resolution at 5.9 keV was 863 eV with an electronic noise contribution of 806 eV FWHM. By comparing spectral distributions measured using the 59.54 keV ^{241}Am nuclear line with HASYLAB 60 keV monoenergetic radiation, it was found that the resolutions for full-area illumination were approximately 15% broader than for pencil beam illumination.

In figure 9, we show a composite of energy-loss spectra recorded at HASYLAB by pixel 6 over the energy range 12 keV to 98 keV. Although this pixel has the best resolution in the array, plots for the other pixels are similar. The smaller peaks located just below the main photopeak are due to the Cd and Te escape peaks. From the figure, we note the effects of hole trapping - as evidenced by the increasing tailing at higher energies. In figure 10, we show the FWHM energy resolution of the array. The two lines represent best-fit exponential functions to the resolution data of the best and the worst pixels. Thus, these lines delineate the bounds within which the other pixels lie. On average, the resolution varies from ~ 800 eV at 10 keV to ~ 4 keV at 100 keV and is found to be dominated by electronic noise (the measured pulser resolution being ~ 800 eV FWHM).

Finally, in fig. 11, we show the spatial response across the pixels measured at an incident energy of 12 keV with a $20 \times 20 \mu\text{m}^2$ beam. The spatial sampling in X and Y is $10 \mu\text{m}$. Figure 11(a) shows the variation in count rate profile above a 3 keV threshold, (b) the fitted centroid of the photopeak (*i.e.*, gain map), and (c) the energy resolution expressed in terms of resolving power. From the figure, we see that the overall response is non-uniform with variations exceeding that expected from statistics alone. Specifically, the variations in count rate about the mean are at the $\pm 6\%$ level, in gain at the $\pm 11\%$ level and resolution at the $\pm 14\%$ level. The expected variations are 2%, 0.2% and 5%, respectively.

3. DISCUSSION AND CONCLUSIONS

While the performances of compound semi-conductors are not yet close to the Fano limit, they are sufficiently close to satisfy the requirements of many of the anticipated future space missions. The fact that the performance of both systems 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.

REFERENCES

1. M. Bavdaz *et al.*, Proc. SPIE, **3114**, 101, 1997.
2. M-A. Gagliardi, *et al.*, Mat. Res. Soc. Symp. Proc. vol., **487**, 77, 1998.
3. M-A. Gagliardi, *et al.*, *ibid*, 165.
4. M. Bavdaz *et al.*, *ibid*, 565.
5. A. Owens *et al.*, J. App. Phys, **86**, 4341, 1999.
6. http://www-hasyllab.desy.de/facility/experimental_stations/stations/X1.1_ROEMO_II.htm
7. J.E. Eberhardt, R.D. Ryan, A.J. Tavendale, Nucl. Instr. And Meth., **94**, 463, 1971.