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Temperature characterisation of spectroscopic InGaP X-ray 1 photodiodes 2 3 S. Butera^{1,*)}, G. Lioliou¹, A. B. Krysa², A. M. Barnett¹ 4 5 ¹Space Research Group, School of Engineering and Informatics, University of Sussex, 6 7 Brighton, BN1 9QT, UK. 8 ²EPSRC National Epitaxy Facility, University of Sheffield, Mappin Street, Sheffield, 9 S1 3JD, UK. 10 11 Email: S.Butera@sussex.ac.uk (S. Butera) 12 Tel.: +441273872568 13 14 Abstract. In this paper for the first time, an InGaP photodiode was used in a high 15 temperature tolerant X-ray spectrometer. The use of InGaP in X-ray spectrometers 16 shows a significant advance within this field allowing operation up to 100 °C. Such 17 results are particularly important since GaP and InP (the InGaP binary parent 18 compounds) are not spectroscopic even at room temperature. The best energy 19 resolution (smallest FWHM) at 5.9 keV for the InGaP spectrometer was 1.27 keV at 20 100 °C and 770 eV at 20 °C, when the detector was reverse biased at 5 V. The observed 21 FWHM were higher than the expected statistically limited energy resolutions indicating 22 that other sources of noise contributed to the FWHM broadening. The spectrometer's 23 Si preamplifier electronics was the limiting factor for the FWHM rather than the InGaP 24 photodiode itself. The InGaP electron-hole pair creation energy (ε_{InGaP}) was 25 experimentally measured across the temperature range 100 °C to 20 °C. EInGaP was 4.94 26 $eV \pm 0.06 eV$ at 20 °C. 27 28 Keywords: InGaP; X-ray spectroscopy; electron-hole pair creation energy; 29 semiconductor. 30 31 32 **1. INTRODUCTION** 33

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34 High-resolution X-ray astronomy and X-ray fluorescence spectroscopy [1] have been 35 made possible because of the use of photon counting X-ray spectrometers. The ability 36 to determine the energy of individual X-ray photons and the number of the detected X-37 ray photons at a particular energy can be essential in space missions. These attributes 38 are particularly useful to study planetary surfaces, magnetospheres, and solar physics, 39 as well as for terrestrial applications such as industrial monitoring and non-destructive 40 testing. The use of wide bandgap materials in such spectrometers is attractive because 41 such materials can have low thermally generated leakage currents; as such they can 42 operate at high temperatures without cooling systems thus resulting in more compact, 43 lower mass, and lower power instrumentation.

44

45 High energy resolution and temperature tolerant photon counting X-ray spectrometers 46 have been reported using various wide bandgap semiconductors detectors coupled to 47 low-noise preamplifiers electronics [2, 3, 4, 5]. Lioliou et al. [2] reported a GaAs diode 48 with energy resolution (Full Width at Half Maximum, FWHM) at 5.9 keV of 840 eV at 49 60 °C. Barnett et al. [3] demonstrated an Al_{0.8}Ga_{0.2}As detector with energy resolution 50 at 5.9 keV of 2.2 keV at 90 °C. In both cases the Si preamplifier electronics were also 51 operated uncooled at the same temperature as the compound semiconductor 52 photodetector. A SiC X-ray spectrometer with an energy resolution at 5.9 keV of 53 233 eV at 100 °C has also been developed by Bertuccio et al. [4]. Recently, another 54 wide bandgap semiconductor, $Al_{0.52}In_{0.48}P$, has shown exceptional promise as a newly 55 emerging material for photon counting X-ray spectroscopy. Butera et al. [5] reported 56 an Al_{0.52}In_{0.48}P detector spectrometer with an energy resolution at 5.9 keV of 1.57 keV 57 at 100 °C and 0.90 keV at 20 °C. The spectroscopic performance of CdTe and CdZnTe 58 detectors has also been investigated at high temperature. Squillante et al. [6] reported 59 a CdTe spectrometer with an energy resolution at 122 keV of 53 keV at 92 °C. 60 Egarievwe et al. [7] developed a CdZnTe spectrometer with an energy resolution at 32 61 keV of 9.4 keV at 70 °C. CdTe and CdZnTe detectors have been widely developed for 62 room temperature X-ray spectroscopy. For example, Zappettini et al. [8] demonstrated 63 CdZnTe detectors with an energy resolution at 59.5 keV of 2.5 keV using low-noise 64 application specific integrated circuit (ASIC) readout electronics. Abbene et al. [9] 65 reported a CdZnTe structure showing energy resolutions of 3.8% (2.26 keV) and 3.2% (3.91 keV) at 59.5 keV and 122.1 keV, respectively, at low count rate. Recently an 66 67 In_{0.5}Ga_{0.5}P X-ray photodiode was also demonstrated to be spectroscopic at room

temperature when coupled to a low noise charge sensitive preamplifier [10]. This was particularly surprising given that its parent materials InP and GaP had been previously found to be non-spectroscopic [11, 12, 13, 14]. The use of In_{0.5}Ga_{0.5}P is important because it has large X-ray and γ -ray attenuation coefficients leading to high quantum detection efficiencies per unit thickness [15, 16].

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74 In this paper, for the first time, an $In_{0.5}Ga_{0.5}P p^+-i-n^+$ mesa photodiode was coupled to 75 a custom-made low-noise charge-sensitive preamplifier and investigated for its 76 performance at high temperature (from 100 °C to 20 °C). The material's electron-hole 77 pair creation energy was also determined. The performance of the spectrometer was 78 analysed under the illumination of a 192 MBq ⁵⁵Fe radioisotope X-ray source over the 79 temperature range using different shaping times and applied biases. At 100 °C, the best 80 energy resolution at 5.9 keV was 1.27 keV, which improved to 770 eV at 20 °C. The 81 different noise contributors to these determined energy resolutions were computed and 82 are discussed in detail. The electron-hole pair creation energy, ε_{InGaP} , was measured 83 using a dedicated experiment. It was found that $\varepsilon_{InGaP} = 4.94 \text{ eV} \pm 0.06 \text{ eV}$ at 20 °C. 84 ε_{InGaP} is the average energy consumed in the generation of an electron-hole pair during 85 the creation of a charge cloud of electron-hole pairs upon absorption of an X-ray photon 86 within In_{0.5}Ga_{0.5}P.

87

88 2. EXPERIMENTAL

89 2.1 STRUCTURE DESIGN90

91 An $In_{0.5}Ga_{0.5}P p^+-i-n^+$ epilayer was grown on a heavily doped n^+ GaAs substrate by 92 low-pressure (150 Torr) metalorganic vapour phase epitaxy using trimethylgallium, 93 trimethylindium, arsine, and phosphine as precursors, and hydrogen as a carrier gas. 94 Disilane and dimethylzinc:triethylamine were used for n- and p-doping, respectively. 95 The epitaxial surface of the substrate had an orientation of (100) with a miscut angle of 96 10° towards the GaAs <111> plane terminating with Ga atoms. The unintentionally 97 doped i layer (thickness of 5 μ m) was between a top p⁺ layer (thickness of 0.2 μ m; doping concentration of 2×10^{18} cm⁻³) and a bottom n⁺ layer (thickness of 0.1 µm; 98 doping concentration of 2×10^{18} cm⁻³). It has to be noted that the thickness of the p⁺ 99 100 and n^+ layers were as thin as possible such to decrease the absorption in these layers. 101 The thicknesses for the p^+ layer (0.2 µm) and n^+ layer (0.1 µm) were chosen based on

102 our own experience of growth of high quality $In_{0.5}Ga_{0.5}P$. The thickness of the i layer, 103 instead, was thick to increase the absorption, and consequently the quantum efficiency, 104 in this layer. It has to be highlighted that the $In_{0.5}Ga_{0.5}P$ device is the thickest i layer 105 In_{0.5}Ga_{0.5}P photodiode so far reported; i layers thicker than 5 µm may be produced in 106 the future. On top of the $In_{0.5}Ga_{0.5}P p^+-i-n^+$ epilayer, a thin p^+ GaAs layer (thickness of 0.01 μ m; doping concentration of 1 \times 10¹⁹ cm⁻³) was grown to help achieve a good 107 top Ohmic contact. n type GaAs, n type In_{0.5}Ga_{0.5}P and unintentionally doped 108 109 $In_{0.5}Ga_{0.5}P$ were grown at a temperature of 700 °C, and the subsequent p-doped layers 110 were grown at 660 °C. At room temperature, the grown In_{0.5}Ga_{0.5}P had a 111 photoluminescence peak energy of 1.89 eV. This energy is in good agreement with the 112 bandgap of the material with a suppressed spontaneous long-range ordering in the group 113 III sublattice [17]. The Ohmic contact on top of the p⁺ GaAs layer was formed from Ti 114 (thickness of 20 nm) and Au (thickness of 200 nm). The Ohmic rear contact, deposited 115 onto the rear of the n⁺ GaAs substrate, was formed from InGe (thickness of 20 nm) and 116 Au (thickness of 200 nm). The In_{0.5}Ga_{0.5}P photodiode was not passivated. Chemical 117 wet etching techniques (1:1:1 K₂Cr₂O₇:HBr:CH₃COOH solution followed by a 10 s 118 finishing etch in 1:8:80 H₂SO₄:H₂O₂:H₂O solution) were used to fabricate the 200 µm 119 diameter In_{0.5}Ga_{0.5}P mesa device used in the study. The device layers, their relative 120 thicknesses and materials are summarised in TABLE 1.

121

Layer	Material	Thickness (μm)	Dopant	Dopant Type	Doping density (cm ⁻³)
1	Ti	0.02			
2	Au	0.2			
3	GaAs	0.01	Zn	p^+	1×10^{19}
4	$In_{0.5}Ga_{0.5}P$	0.2	Zn	p^+	2×10^{18}
5	$In_{0.5}Ga_{0.5}P$	5	undoped		$< 5 \times 10^{16}$
6	$In_{0.5}Ga_{0.5}P$	0.1	Si	n^+	2×10^{18}
7	GaAs buffer	0.3	Si	n^+	2×10^{18}
8	Substrate n ⁺ GaAs	350	Si	n^+	2×10^{18}
9	InGe	0.02			
10	Au	0.2			

122 TABLE 1. Layer details of the $In_{0.5}Ga_{0.5}P$ photodiode.

124 A 192 MBq ⁵⁵Fe radioisotope X-ray source (Mn K α = 5.9 keV, Mn K β = 6.49 keV) was

125 positioned 5 mm away from the top surface of the 200 μ m diameter In_{0.5}Ga_{0.5}P mesa

126 photodiode such as to study the detector performances under illumination.

¹²³

128 The $In_{0.5}Ga_{0.5}P$ X-ray quantum efficiencies (*QE*) through the device's optical window 129 (region not covered by contacts) were calculated using the Beer-Lambert law and 130 assuming complete charge collection in the p and i layers. Figure 1 shows the 131 $In_{0.5}Ga_{0.5}P$ X-ray quantum efficiencies as a function of photon energy up to 10 keV. 132



133

134Figure 1. Calculated $In_{0.5}Ga_{0.5}P$ X-ray quantum efficiencies as a function of photon135energy.

136

137 X-ray quantum efficiencies (*QE*) of 53% at 5.9 keV and 44% at 6.49 keV were 138 computed for the structure. TABLE 2 shows the attenuation coefficients at 5.9 keV 139 and 6.49 keV for $In_{0.5}Ga_{0.5}P$ as well as other different materials. The attenuation 140 coefficients for binary and ternary compounds were estimated from the attenuation 141 coefficients of their single elements, properly weighted [15, 16].

143 TABLE 2. Attenuation coefficients at 5.9 keV and 6.49 keV for different materials.

Material	Attenuation coefficient at 5.9 keV	Attenuation coefficient at 6.49 keV
	(cm ⁻¹)	(cm ⁻¹)
In _{0.5} Ga _{0.5} P	1464	1130
$Al_{0.52}In_{0.48}P$	1301	1004
GaAs	837	642
Al _{0.8} Ga _{0.2} As	788	604
Si	346	263

¹⁴⁴

145 *QE* at 5.9 keV greater than 90% may be obtained by increasing the $In_{0.5}Ga_{0.5}P$ i layer 146 thickness to 30 µm; this i layer thickness may be achieved in future $In_{0.5}Ga_{0.5}P$ 147 structures as consequence of advances in growth and fabrication technologies. Because 148 of the higher linear attenuation coefficients of $In_{0.5}Ga_{0.5}P$ with respect to SiC, the

quantum efficiency of the 5 μ m In_{0.5}Ga_{0.5}P device at high X-ray photon energies (> 48 keV) are expected to be higher than those of a 300 μ m SiC detector at the same energies.

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- 152

2 2.2 CHARACTERIZATION SETUP

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The In_{0.5}Ga_{0.5}P device was installed inside a TAS Micro MT climatic cabinet for temperature control. The temperature was initially set to 100 °C and decreased to 20 °C, in steps of 20 °C. Before taking any measurements at each temperature, the device was left for 30 minutes to ensure stabilisation.

158

159 The In_{0.5}Ga_{0.5}P leakage current as a function of reverse bias was measured using a 160 Keithley 6487 picoammeter/voltage source. The uncertainty associated with individual 161 current readings was 0.3% of their values plus 400 fA, while the uncertainty associated 162 with applied biases was 0.1% of their values plus 1 mV [18]. The In_{0.5}Ga_{0.5}P 163 capacitance as a function of reverse bias was measured using an HP 4275A Multi 164 Frequency LCR meter. The uncertainty associated with each capacitance reading was 165 0.12% [19], while the uncertainty associated with applied biases was 0.1% of their 166 values plus 1 mV [18]. The test signal was sinusoidal with a 50 mV rms magnitude 167 and 1 MHz frequency. In both leakage current and capacitance measurements, the 168 reverse bias increased from 0 V to 15 V (in 1 V increments).

169

170 X-ray spectra were obtained using the ⁵⁵Fe radioisotope X-ray source to illuminate the 171 200 µm diameter In_{0.5}Ga_{0.5}P device at temperatures from 100 °C to 20 °C. The 172 experimental setup utilised a custom-made charge-sensitive preamplifier of feedback 173 resistorless design, similar to that reported in ref. [20]. The preamplifier was operated 174 at the same temperature as the photodiode. The signal from the preamplifier was 175 shaped by an Ortec 572a shaping amplifier, and digitized by a multichannel analyser 176 (Ortec Easy-MCA-8K). Spectra were accumulated and analysed at shaping times of 177 $0.5 \mu s$, 1 μs , 2 μs , 3 μs , 6 μs , and 10 μs . The In_{0.5}Ga_{0.5}P device was reverse biased at 0 178 V, 5 V, 10 V, and 15 V, in each case. The live time for each spectrum was 200 s.

179

All the experiments were performed in dry nitrogen atmosphere (relative humidity
<5%) as a precautionary measure to eliminate any formation of water vapor at high
temperatures and water condensation at low temperatures inside the chamber.

183

184 **3. RESULTS**

185

187

186 **3.1 Current and capacitance measurements**

188 The measured leakage currents of the packaged device at 100 °C and 80 °C are shown 189 in Figure 2; leakage currents at temperatures below 80 °C are not reported because they 190 were below the picoammeter's noise floor. Measurements of the leakage current as a 191 function of the reverse bias of the system when the diode was not connected showed 192 that the system was contributing to the measured leakage current. At 100 °C and at 80 193 °C, the packaged device (defined as the semiconductor and system combined) had 194 leakage currents of 1.5 pA and 0.5 pA, respectively, at a reverse bias of 10 V. At the 195 same temperatures and reverse bias condition, the system (with no diode connected) 196 had leakage currents of 1.1 pA and 0.2 pA, respectively. When the reverse bias was 197 increased to 15 V in each case, the leakage currents measured for the packaged device 198 and the system became indistinguishable at both temperatures. Considering the 199 uncertainties associated with the leakage current measurements, the leakage current 200 from the diode itself can be considered negligible compared with the other leakage 201 currents.

202



203**Reverse Bias (V)**204Figure 2. Leakage current of the packaged $In_{0.5}Ga_{0.5}P$ detector (i.e. from both the205semiconductor junction and the package) as a function of applied reverse bias at 100206°C (filled circles) and 80 °C (empty squares).

At different temperatures, the capacitance of the packaged $In_{0.5}Ga_{0.5}P$ detector as a function of reverse bias was measured. The capacitance of an empty package of the same type was also measured at different temperatures and subtracted from the

measured capacitance of the packaged In_{0.5}Ga_{0.5}P photodiode. At each temperature, 212 213 the capacitances were measured multiple times; the mean and its relative root mean 214 squared (RMS) error were considered. The capacitances of the empty package were 215 measured to be 1.27 pF \pm 0.02 pF and 1.132 pF \pm 0.003 pF at 100 °C and 80 °C, 216 respectively. The uncertainties reflect not only the uncertainty in one measurements, 217 but also the variation in measured value upon repetition; greater variation was seen at 218 100 °C than at 80 °C. In the temperature range studied, the capacitance of the 219 In_{0.5}Ga_{0.5}P detector itself (C) was found to be temperature invariant. $1/C^2$ as a function of reverse bias at 100 °C and at 80 °C is shown in Figure 3, similar results were found 220 at temperatures ≤ 60 °C. A dependence between $1/C^2$ and the reverse bias was found 221 at reverse biases below 3 V; $1/C^2$ was constant at reverse biases higher than 3 V. 222

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224

Figure 3. $1/C^2$ as a function of applied reverse bias. The temperatures analysed were 100 °C (filled circles) and 80 °C (empty squares).

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230

229 **3.2 X-ray spectroscopy and noise analysis**

X-ray spectra were obtained using the ⁵⁵Fe radioisotope X-ray source. Although temperatures above 100 °C can be achieved by the TAS Micro MT climatic cabinet, temperatures higher than 100 °C were not studied because of limitations in the working temperature range of the spectrometer's electrical cables. At 100 °C, the diode was stable throughout the spectrum acquisition time. The diode did not degrade after being used at such temperatures. Moreover, polarization phenomena were not observed in the detector at any of the temperatures or biases studied.

239 An improvement in energy resolution (as quantified by the FWHM at 5.9 keV) was 240 observed when increasing the applied reverse bias from 0 V to 5 V. This result can be 241 explained considering the reduction in capacitance of the detector and possibly 242 improved charge collection. No further change in FWHM was observed when 243 operating the detector at reverse biases > 5 V. The latter behaviour can be explained 244 considering that the $In_{0.5}Ga_{0.5}P$ photodiode is fully depleted above 5 V.

245

246 The optimum shaping time (i.e. that which produced the smallest FWHM) varied with 247 temperature, as shown in Figure 4. The FWHM decreased at lower temperatures 248 because of the lower leakage currents of the In_{0.5}Ga_{0.5}P photodiode and Si JFET at such 249 temperatures. The spectra with the best energy resolution (smallest FWHM) at 100 °C and 20 °C with the photodiode reverse biased at 5 V are presented in Figure 5. The 250 251 observed ⁵⁵Fe photopeaks were the combination of the characteristic Mn K α (5.9 keV) and Mn K_β (6.49 keV) lines of the ⁵⁵Fe radioisotope X-ray source. To determine the 252 253 FWHM of the 5.9 keV peaks in Figures 4 and 5, Gaussian fitting was performed on the 254 photopeaks: the Mn K α and Mn K β peaks were deconvolved from detected combined 255 photopeak. The fitting took into account the relative X-ray emission rates of the ⁵⁵Fe radioisotope X-ray source at 5.9 keV and 6.49 keV in the appropriate ratio [21] as well 256 257 as the relative efficiency of the detector at these X-ray energies.

258



The smallest observed FWHM of the 5.9 keV peak as a function of Figure 4. 261 temperature at the optimum shaping time, when the In_{0.5}Ga_{0.5}P detector was reverse biased at 5 V. 262

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266 267

Figure 5. Best energy resolution ⁵⁵Fe X-ray spectra collected at 100 °C (a) and at 20 °C (b) with the In_{0.5}Ga_{0.5}P photodiode reversed bias at 5 V. Also shown in each spectrum are the deconvolved Mn K α (dashed line) and Mn K β (dashed-dot line) peaks.

272 The FWHM of the 5.9 keV peak as a function of shaping time at 100 °C and 20 °C,

with the photodiode reverse biased at 5 V, are presented in Figure 6.



274

Figure 6. FWHM of the 5.9 keV peak as a function of shaping time at 100 °C (filled circles) and 20 °C (empty circles), when the In_{0.5}Ga_{0.5}P detector was reverse biased at 5 V.

279 The energy resolution (FWHM) of a non-avalanche X-ray photodiode spectrometer is 280 degraded by the Fano noise, the charge trapping noise, and the electronic noise [22, 23]. 281 The Fano noise is due to the statistical nature of the ionisation process; it is calculated 282 and explained in section "C. Fano-limited energy resolution and electron-hole pair 283 creation energy". At each temperature studied, the observed FWHM was greater than 284 the expected Fano limited energy resolution, indicating that noise sources other than 285 the statistical charge creation process were significant. In a photodiode X-ray 286 spectrometer, the electronic noise is caused by 5 different components: parallel white

287 noise, series white noise, induced gate current noise, 1/f noise, and dielectric noise [22, 288 23]. The leakage currents of the detector and the Si input JFET of the preamplifier 289 (which was operated uncooled at each temperature) influenced the parallel white noise, 290 as shown in equation 1 [22, 23, 24]. The capacitances of the detector and input JFET of the preamplifier influence the series white noise and 1/f noise, as shown in equations 291 292 2 and 3 [22, 23, 24]. Parallel white noise and series white noise are, respectively, 293 directly and inversely proportional to the shaping time; whilst 1/f noise and dielectric 294 noise are independent of shaping time [22, 23].

296
$$ENC_{wp} = \frac{1}{q} \sqrt{\frac{A_3}{2} 2q(I_D + I_{JFET})\tau}$$
 (1)

297
$$ENC_{ws} = \frac{B}{q} \sqrt{\frac{A_1}{2} 4kT \frac{\gamma}{g_m} (C_D + C_{JFET})^2 \frac{1}{\tau}}$$
 (2)

298
$$ENC_{1/f} = \frac{1}{q} \sqrt{A_2 \pi \gamma 4kT \frac{f_c}{g_m} (C_D + C_{JFET})^2}$$
 (3)
299

where A_1 , A_2 and A_3 are 1.85, 1.8, and 1.85, respectively [24]; I_D the experimentally 300 301 measured packaged device leakage current at different temperatures, I_{JFET} the JFET 302 leakage current at different temperatures (at 20 °C the leakage current of the JFET was 303 1 pA); C_D the experimentally measured packaged device capacitance at different 304 temperatures, C_{JFET} the JFET capacitance (assumed to be 2 pF at all the temperatures 305 studied), g_m the JFET transconductance (assumed to be 6 mS at the operating condition 306 of the JFET), γ the product of the noise resistance and the transconductance of the JFET 307 (0.85), B the induced gate current correction (0.8)[23], f_c the corner frequency of the 308 JFET (assumed to be 1000 Hz at the operating condition of the JFET) [25].

309

The obtained parallel white noise, series white noise (adjusted for induced gate current noise [22, 23, 24], and 1/f noise, as well as the measured equivalent noise charge, at shaping times of (a) 0.5 μ s, (b) 1 μ s, and (c) 10 μ s, with the In_{0.5}Ga_{0.5}P photodiode reverse biased at 5 V, are shown in Figure 7. In Figure 7 the measured equivalent noise charge was calculated using the value of the In_{0.5}Ga_{0.5}P electron-hole pair creation energy as determined at each temperature in section "3.3. Fano-limited energy resolution and electron-hole pair creation energy".



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Figure 7. Equivalent noise charge as a function of temperature at shaping time of (a) 0.5 μ s, (b) 1 μ s, and (c) 10 μ s, when the In_{0.5}Ga_{0.5}P photodiode was reverse biased at 5 V. The graphs show the contributions of the parallel white noise (empty circles), the series white noise (empty squares) and the 1/*f* noise (empty triangles), as well as the measured equivalent noise charge (crosses).

The high parallel white noise observed at increased temperatures and at increased shaping times is not due to the high leakage current of the $In_{0.5}Ga_{0.5}P$ detector, but instead due to the higher current of the uncooled Si input JFET of the preamplifier [22].

- The FWHM of the 5.9 keV peak as a function of shaping time, reported in Figure 6, shows that at 100 °C the noise was leakage current limited, as expected when combining in quadrature the parallel white noise and the series white noise of Figure 7. Therefore, the shortest shaping time ($0.5 \ \mu s$) gave the best energy resolution. The noise at 20 °C was not leakage current limited, as suggested by the FWHM of the 5.9 keV peak as a function of shaping time (Figure 6). Thus, a long shaping time, 6 μs , resulted in the best energy resolution.
- 336

The temperature dependence of the residual noise is shown in Figure 8. At each shaping
time, the residual noise was estimated by subtracting in quadrature the known noise
components from the measured ENC. In Figure 8a, the residual noise dependence on

the temperature at all the six studied shaping times was reported. In Figure 8b, the mean of the residual noises among the six shaping times (at each point the root mean squared error was associated) as a function of temperature is shown. The measured FWHM was converted into ENC using the values of the electron-hole pair creation energies at each temperature as determined in section "3.3. Fano-limited energy resolution and electron-hole pair creation energy".





Figure 8. (a) Equivalent noise charge of the residual noise at 5.9 keV at each shaping time studied as a function of temperature, when the $In_{0.5}Ga_{0.5}P$ photodiode was reverse biased at 5 V. (b) Mean of the equivalent noise charge of the residual noise at 5.9 keV among the six shaping times as a function of temperature (at each point the root mean squared error was associated), when the $In_{0.5}Ga_{0.5}P$ photodiode was reverse biased at 5 V.

354 In the temperature range 100 °C to 20 °C, the residual noise contribution at 5.9 keV

linearly decreased with decreasing temperature: at 100 °C a value of 94 e⁻ rms \pm 15 e⁻

rms was calculated; whilst at 20 °C a value of 63 e⁻ rms \pm 2 e⁻ rms was determined.

357

The $In_{0.5}Ga_{0.5}P$ spectrometer allowed high temperature operation (up to the maximum investigated, 100 °C). It presented better FWHM than was achieved using $Al_{0.52}In_{0.48}P$ [5] and $Al_{0.8}Ga_{0.2}As$ [3] spectrometers, but not as good as has been demonstrated using SiC detectors with lower noise readout electronics [4], at the same temperatures. It should also be noted that the use of ultra-low-noise readout electronics, such as those reported in Ref. [26] would likely improve the energy resolution achieved.

364

365 The ability to work at such high (100 $^{\circ}$ C) temperatures together with their greater X-

366 ray attenuation coefficients makes In_{0.5}Ga_{0.5}P spectrometers preferred over recently

- 367 reported GaAs spectrometers which have a maximum operating temperature of 60 °C
- 368 [2]. However, at more modest temperatures (e.g. 60 °C) the previously reported GaAs

369 spectrometer had a better FWHM at 5.9 keV (840 eV) than the $In_{0.5}Ga_{0.5}P$ spectrometer 370 (1.02 keV). The presently reported $In_{0.5}Ga_{0.5}P$ X-ray spectrometer also performed 371 better at 100 °C than the previously reported $Al_{0.52}In_{0.48}P$ X-ray spectrometer. The 372 FWHM at 5.9 keV for the $In_{0.5}Ga_{0.5}P$ device was 1.27 keV at 100 °C c.f. 1.57 keV for 373 the $Al_{0.52}In_{0.48}P$ device using similar device readout electronics. $In_{0.5}Ga_{0.5}P$ also has

- $\label{eq:2.1} 374 \qquad \text{larger linear attenuation coefficients than } Al_{0.52} In_{0.48} P.$
- 375

376 Since the readout electronics used to characterise these materials have been broadly 377 comparable, the difference in obtained FWHM for these materials (GaAs, AlInP, 378 InGaP) can be explained considering their different electron-hole pair creation energies 379 and the noise contributions of the readout electronics at high temperature (see section 380 "3.3 Fano-limited energy resolution and electron-hole pair creation energy"). A total 381 noise at the input of the preamplifier of 86 e⁻ rms, for example, corresponds to 840 eV 382 in GaAs, to 1.00 keV in In_{0.5}Ga_{0.5}P and to 1.08 keV in Al_{0.52}In_{0.48}P. The observed 383 FWHM of 1.02 keV at 5.9 keV at 60 °C for the In_{0.5}Ga_{0.5}P spectrometer was very close 384 to the expected value. Therefore, the total noise in e⁻ rms was similar in the GaAs and 385 In_{0.5}Ga_{0.5}P spectrometers, since the preamplifier was limited by noises other than the 386 detector leakage current at these temperatures.

387

388 However, the energy resolution achieved with the very best SiC X-ray detectors 389 coupled to much lower noise readout electronics [4] is superior to that obtained with 390 In_{0.5}Ga_{0.5}P and our preamplifier electronics. A SiC detector with FWHM of 233 eV at 391 5.9 keV has been reported at 100 °C [4]. It would be interesting to characterise the 392 In_{0.5}Ga_{0.5}P detectors with the same ultra-low noise electronics used for the SiC 393 detectors to establish a better comparison between the materials. It should also be noted 394 that the X-ray attenuation coefficients of In_{0.5}Ga_{0.5}P are much greater than those for 395 SiC. Thus, even if the ultimately achievable energy resolution with In_{0.5}Ga_{0.5}P is more 396 modest than SiC, In_{0.5}Ga_{0.5}P may still be preferred for low-flux, high-energy 397 applications.

398

400

399 3.3 Fano-limited energy resolution and electron-hole pair creation energy

401 The Fano-limited energy resolution is related to the charge creation process at the 402 absorption of an X-ray photon, and is the statistically limited energy resolution of a 403 non-avalanche X-ray photodiode spectrometer [27]. The Fano-limited energy
404 resolution (FWHM in eV) can be calculated using equation 4:

405

$$406 \quad FWHM = 2.35\varepsilon \sqrt{\frac{FE}{\varepsilon}} \tag{4}$$

407

408 where ε is the semiconductor electron-hole pair creation energy, *F* is the Fano factor, 409 and *E* is the X-ray photon's energy. Different semiconductors have different Fano 410 limited energy resolutions at the same X-ray photon's energy. This is because the Fano 411 limited energy resolution at each energy is dependent on physical material properties 412 (average electron-hole pair creation energy and Fano factor).

413

For the room temperature (20 °C) measurements of the electron-hole pair creation energy, a method similar to that reported by other researchers [28, 29, 30, 31] was used. The charge created by the absorption of X-rays from the ⁵⁵Fe radioisotope X-ray source in the In_{0.5}Ga_{0.5}P photodiode was measured relative to that created in a reference 200 μ m GaAs mesa photodiode. The structure of the GaAs device is summarised in TABLE 3. The In_{0.5}Ga_{0.5}P and GaAs detectors were connected in parallel to the custom-made low-noise charge-sensitive preamplifier.

422 TABLE 3. Layer details of the GaAs photodiode.

Layer	Material	Thickness	Dopant	Dopant	Doping density
		(µm)		Туре	(cm ⁻³)
1	Ti	0.02			
2	Au	0.2			
3	GaAs	0.5	Be	\mathbf{p}^+	2×10^{18}
4	GaAs	10	undoped		$< 10^{15}$
5	GaAs	1	Si	\mathbf{n}^+	2×10^{18}
6	Substrate n ⁺ GaAs				
7	InGe	0.02			
8	Au	0.2			

⁴²³

The In_{0.5}Ga_{0.5}P and the GaAs photodetectors were both independently reverse biased at 10 V. Spectra were accumulated with the ⁵⁵Fe radioisotope X-ray source illuminating the In_{0.5}Ga_{0.5}P device and the GaAs device separately, in turn. Gaussians were fitted to the detected Mn K α (5.9 keV) and Mn K β (6.49 keV) peaks of the accumulated

428 spectra; the ⁵⁵Fe X-ray spectra accumulated and the fitted 5.9 keV peaks for the 429 $In_{0.5}Ga_{0.5}P$ detector and the GaAs reference photodetector are shown in Figure 9.

430



431

432 Figure 9. ⁵⁵Fe X-ray spectra accumulated at 10 V reverse bias using the $In_{0.5}Ga_{0.5}P$ 433 device (empty circles) and the GaAs reference photodetector (filled circles) under the 434 illumination of ⁵⁵Fe radioisotope X-ray source. Also shown are the fitted 5.9 keV lines 435 for the $In_{0.5}Ga_{0.5}P$ device (dashed-dot line) and the GaAs reference photodetector 436 (dashed line). For clarity, the fitted 6.49 keV Mn Kβ peaks are not shown but were 437 included appropriately in the fitting.

438

The quantity of charge corresponding to each MCA channel was calculated using the position of the zero noise energy peak of the preamplifier and the 5.9 keV peak detected by the GaAs reference photodiode. In this calculation, the GaAs electron-hole pair creation energy, 4.184 eV \pm 0.025 eV, [28] was also used. The In_{0.5}Ga_{0.5}P electronhole pair creation energy (ε_{InGaP}) was then determined using equation 5:

444

445
$$\varepsilon_{InGaP} = \varepsilon_{GaAs} \left(\frac{N_{GaAs}}{N_{InGaP}} \right)$$
 (5)

446

447 where ε_{GaAs} is the electron-hole pair creation energy in GaAs, N_{GaAs} and N_{InGaP} are the 448 number of charges created in the GaAs reference detector and $In_{0.5}Ga_{0.5}P$ detector, 449 respectively. An experimental value of 4.94 eV \pm 0.06 eV was measured for ε_{InGaP} at 450 room temperature (20 °C). To examine the effect of operating the In_{0.5}Ga_{0.5}P detector 451 at higher reverse biases, the reverse bias was increased to 15 V, and the experiment 452 repeated. An electron-hole pair creation energy of 4.90 eV \pm 0.04 eV was measured in 453 this instance. The similarity of the values further confirms that charge trapping was 454 negligible. If charge trapping was significant, a substantial reduction in apparent 455 electron-hole pair creation energy would have been observed at higher reverse bias as

456 a consequence of the improved charge transport at higher electric field.

457

458 The dependence of the $In_{0.5}Ga_{0.5}P$ electron-hole pair creation energy upon temperature 459 was studied across the temperature range 100 °C to 20 °C. For this set of measurements, 460 the In_{0.5}Ga_{0.5}P detector was individually connected to the custom-made low-noise 461 charge-sensitive preamplifier (i.e. without the GaAs reference detector) and illuminated by the ⁵⁵Fe radioisotope X-ray source. The change in conversion factor of the 462 463 preamplifier itself with temperature was measured across the temperature range by 464 connecting a stabilized pulse generator (Berkeley Nucleonics Corporation model BH-465 1) to the test signal input of the preamplifier. The change in position of the centroid of 466 the pulse generator peak allowed the change in performance of the preamplifier with 467 temperature to be untangled from the change in electron-hole pair creation energy of 468 the photodiode. The change in position of the centroid of the pulse generator peak was 469 appropriately corrected for the change in the test capacitance with temperature [32]. 470 Spectra were collected and Gaussians were fitted to the photopeak and the peak from 471 the pulse generator in order to determine the positions of their centroids with respect to 472 the zero noise peak. The charge created in the $In_{0.5}Ga_{0.5}P$ photodiode by the X-ray 473 photons was related to the relative change in position of the photopeak on the MCA's 474 charge scale. The latter was corrected for the preamplifier's change in conversion 475 factor with temperature (determined from the pulser peak) [31, 33]. The different 476 quantities of charge created at different temperatures was caused by the change in the 477 In_{0.5}Ga_{0.5}P electron-hole pair creation energy (ε_{InGaP}). At each temperature, the 478 absolute value of ε_{InGaP} was then computed using the previously determined room 479 temperature ε_{InGaP} . The apparent In_{0.5}Ga_{0.5}P electron-hole pair creation energy as a 480 function of temperature is reported in Figure 10. The uncertainties associated with the 481 electron-hole pair creation energy values were obtained by propagating the uncertainty 482 in the electron-hole pair creation energy at room temperature and the uncertainty in the relative change in the conversion factors. The uncertainty in the electron-hole pair 483 484 creation energy at room temperature ($\pm 0.06 \text{ eV}$) was an order of magnitude greater 485 than the uncertainties in the relative change in the conversion factors ($\pm 0.005 \text{ eV}$); 486 therefore, the former mainly affected the uncertainties in the electron-hole pair creation 487 energy at different temperatures. A similar experimental setup was used by other researchers to measure the electron-hole pair creation energy in other materials [31,

489 33].

490



491

Figure 10. Temperature dependence of the energy consumed to produce an electronhole pair in $In_{0.5}Ga_{0.5}P$.

494

495 An apparent slight trend suggesting that the $In_{0.5}Ga_{0.5}P$ electron-hole pair creation 496 energy increased with increasing temperature was found: at 100 °C, $\varepsilon_{InGaP} = 5.02 \text{ eV} \pm$ 497 0.07 eV, whereas at 20 °C ε_{InGaP} = 4.94 eV ± 0.06 eV. However, the data points were 498 all within the uncertainties of each other for the temperature range investigated. If the 499 trend (greater average electron-hole pair creation energy at higher temperatures) was 500 real, the results would be surprising. It is conventionally considered that the average 501 electron-hole pair creation energy decreases linearly as the temperature increases [28, 502 31, 33, 34, 35]. Such a decrease can be understood considering the dependence of the 503 electron-hole pair creation energy on the material bandgap energy. According to Klein 504 [36], the empirical relationship between the electron-hole pair creation energy and the 505 bandgap energy in a semiconductor is linear. Since the bandgap decreases at increased 506 temperatures, a similar behaviour is expected for the electron-hole pair creation energy, 507 due at least in part to the change in bandgap. Theoretical Monte Carlo calculations 508 conducted by Fraser et al. [34] for silicon predicted the decrease of the Si electron-hole 509 pair creation energy as a function of temperature.

510

511 The expected Fano limited energy resolution (FWHM) at 5.9 keV of X-ray detectors 512 made from $In_{0.5}Ga_{0.5}P$ was estimated using equation 4 and the determined values for 513 the electron-hole pair creation energy. The Fano factor for $In_{0.5}Ga_{0.5}P$ has not yet been 514 measured, but assuming a Fano factor of 0.12 (as for GaAs [37]), the Fano limited 515 energy resolution would be expected to be 139 eV at 5.9 keV at 20 °C. If the Fano 516 factor was 0.099 (as for CdZnTe [38]) a Fano limited energy resolution of 127 eV at 517 5.9 keV would be expected at 20 °C. Negligible changes over the 20 °C to 100 °C 518 temperature range were observed.

519

The electron-hole pair creation energy at 27 °C (300 K), which was interpolated from the experimental measurements at 20 °C (293 K) and 40 °C (313 K), equalled 4.95 eV ± 0.07 eV. This is in agreement with the value predicted for In_{0.5}Ga_{0.5}P (4.83 eV \pm 0.21 eV) by the empirical Bertuccio-Maiocchi-Barnett (BMB) relationship [31]. Figure 11 shows the average electron-hole pair creation energy for Ge, Si, GaAs, Al_{0.2}Ga_{0.8}As, Al_{0.8}Ga_{0.2}As, Al_{0.52}In_{0.48}P [39], and In_{0.5}Ga_{0.5}P, and as a function of their respective

526 bandgap energies, at a temperature of 300 K.



527

528 Figure 11. Electron-hole pair creation energy for Ge, Si, GaAs, $Al_{0.2}Ga_{0.8}As$, 529 $Al_{0.8}Ga_{0.2}As$, and $Al_{0.52}In_{0.48}P$ (filled circles), and $In_{0.5}Ga_{0.5}P$ (filled square), as a 530 function of their bandgap energy at 300 K. The equation shown for the relationship has 531 been refined using the new data for $In_{0.5}Ga_{0.5}P$. 532

A linear least squares fit of the data showed that the previously reported BMB dependence between electron-hole pair creation energy and bandgap energy can be refined using the new data for In_{0.5}Ga_{0.5}P. The new relation is $\varepsilon = AE_g + B$ with A = (1.54 ± 0.08) and B = 1.89 eV ± 0.14 eV. Using this relationship, the ε_{InGaP} would be expected to be 4.82 eV ± 0.3 eV.

538

As is the case for $Al_{0.8}Ga_{0.2}As$ [30, 33] and $Al_{0.2}Ga_{0.8}As$ [31], the electron-hole pair creation energy value reported here at 300 K for $In_{0.5}Ga_{0.5}P$ does not lie either on the main or secondary Klein function branches [1, 36]. If $In_{0.5}Ga_{0.5}P$ was on the main Klein function branch, the expected ε_{InGaP} would be 6.17 eV, which is substantially higher than that found here (4.95 eV \pm 0.03 eV). If it was on the secondary Klein function branch, the expected ε_{InGaP} would be 4.07 eV, which is substantially lower than the obtained value. This lends further weight to the view that the Klein relationship is incomplete [31].

547

549

548 4. CONCLUSIONS

For the first time an X-ray spectrometer with an InGaP detector was demonstrated 550 551 across the temperature range 100 °C to 20 °C. The spectrometer was characterised at 552 different shaping times and detector reverse biases. The best energy resolution 553 (smallest FWHM) at 5.9 keV was 1.27 keV at 100 °C using a shaping time of 0.5 µs, 554 this improved to 770 eV at 20 °C (using a shaping time of 6 μ s), when the In_{0.5}Ga_{0.5}P 555 detector was reverse biased at 5 V. An improvement in energy resolution (as quantified 556 by the FWHM at 5.9 keV) was observed when increasing the applied reverse bias from 557 0 V to 5 V. The better results obtained at 5 V can be explained considering the 558 improved charge collection in the greater electric field strength. Similar FWHM to that 559 measured at 5 V were observed at 10 V and 15 V, suggesting that charge trapping noise 560 at 5 V and above was negligible. System noise analysis showed that the observed 561 FWHM were higher than the likely statistically limited energy resolution (i.e. the Fano-562 limited energy resolution). The parallel white noise, series white noise, 1/f noise, and 563 residual noise were calculated. The higher parallel white noise observed at increased 564 temperatures was caused by the Si input JFET of the preamplifier rather than the 565 photodetector. At 100 °C and at 0.5 µs, for example, parallel white noises of 30.8 e⁻ 566 rms for the Si JFET and 2.8 e⁻ rms for the In_{0.5}Ga_{0.5}P device were found when the diode 567 was reversed bias at 15 V. A dedicated experiment was conducted to measure the 568 In_{0.5}Ga_{0.5}P average electron-hole pair creation energy (ε_{InGaP}) in the temperature range 569 100 °C to 20 °C. ε_{InGaP} was found to be 4.94 eV \pm 0.06 eV at 20 °C and 5.02 eV \pm 0.07 570 eV at 100 °C.

571

573

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