Investigating the suitability of GaAs:Cr material for high flux X-ray imaging

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ABSTRACT: Semi-insulating wafers of GaAs material with a thickness of 500 µm have been compensated with chromium by Tomsk State University. Initial measurements have shown the material to have high resistivity (3×10^9 Ω cm) and tests with pixel detectors on a 250 µm pitch produced uniform spectroscopic performance across an 80×80 pixel array. At present, there is a lack of detectors that are capable of operating at high X-ray fluxes (>10^8 photons s⁻¹ mm⁻²) in the energy range 5–50 keV. Under these conditions, the poor stopping power of silicon, as well as issues with radiation hardness, severely degrade the performance of traditional detectors. While high-Z materials such as CdTe and CdZnTe may have much greater stopping power, the formation of space charge within these detectors degrades detector performance. Initial measurements made with GaAs:Cr detectors suggest that many of its material properties make it suitable for these challenging conditions. In this paper the radiation hardness of the GaAs:Cr material has been measured on the B16 beam line at the Diamond Light Source synchrotron. Small pixel detectors were bonded to the STFC Hexitec ASIC and were irradiated with 3×10^8 photons s⁻¹ mm⁻² monochromatic 12 keV X-rays up to a maximum dose of 0.6 MGy. Measurements of the spectroscopic performance before and after irradiation have been used to assess the extent of the radiation damage.

KEYWORDS: X-ray detectors; Gamma detectors (scintillators, CZT, HPG, HgI etc); Radiation-hard detectors; Instrumentation for FEL

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1 Introduction

The last decade has seen the development of powerful new X-ray free electron laser (FEL) light sources, such as the Linac Coherent Light Source (LCLS) at the Stanford Linear Accelerator Center (SLAC) and the European X-Ray Free Electron Laser (XFEL) which is currently under construction at Deutsches Elektronen-Synchrotron (DESY) [1]. These facilities will allow exciting new science to be completed from mapping the detailed atomic structure of viruses to studying chemical reactions as they occur [2]. At FEL sources X-rays are delivered in ultra-short pulses with lengths of $\sim 100$ fs with intensities per pulse as high as $10^{12}$ photons. The peak brilliance of these sources can reach as high as $5 \times 10^{33}$ photons s$^{-1}$ mrad$^{-2}$ mm$^{-2}$ 0.1% BW almost ten orders of magnitude higher than that of current 4th generation synchrotrons [1].

The development of these next-generation facilities is placing extreme demands on existing detectors and is driving the development of new technologies. The large X-ray fluxes present in FELs mean that detector systems will be potentially exposed to radiation doses as high as 1 Giga Gray (GGy) over their lifetime [3].

1.1 Hybrid pixel array detectors

Historically silicon and germanium based detectors have been the instruments of choice for spectroscopy, imaging and scattering experiments at synchrotrons. Detectors currently under development for the European XFEL [4] are based on Hamamatsu $p^+n$ silicon sensors which include the Large Pixel Detector (LPD) [5] and the Adaptive Gain Integrating Pixel Detector (AGIPD) [6]. Both these systems are hybrid pixel array detectors in which the silicon pixels are directly bonded to a readout Application Specific Integrated Circuit (ASIC). In this arrangement the direct detection of X-rays and the large number of channels produces detectors with good spatial resolution that are capable of measuring very high fluxes of X-rays.
Recent X-ray (12 keV) radiation hardness studies with these sensors have demonstrated that for doses $> 100 \text{kGy}$ changes in the pixel leakage currents and breakdown voltages are observed in the silicon sensors [7]. Damage to the Si/SiO$_2$ interface causes high field regions to appear, reducing the breakdown voltage, increasing leakage currents and leading to charge loss. The poor stopping power of silicon at an X-ray energy of 12 keV means that, even with the use of a 500 $\mu$m thick sensor, 35% of X-rays pass straight through the sensor without being absorbed. These transmitted X-rays are then able to deposit energy in the ASIC which can lead to significant radiation damage and loss of functionality. For these reasons, there is significant interest in materials with higher X-ray stopping power and radiation tolerance.

1.2 Chromium compensated gallium arsenide

X-ray detectors based on gallium arsenide have been under development for many decades due to the high charge carrier mobilities, good stopping power at hard X-ray energies and room temperature operation. However, the presence of deep defects within the material that limit the charge carrier lifetimes and produce electric field instabilities in bulk grown crystals mean that, until very recently, X-ray detectors have been limited to thin layers of epitaxially grown material [8, 9]. The development of chromium compensated gallium arsenide (GaAs:Cr) by Tomsk State University has allowed crystals of thicknesses of up to 1 mm to be produced that have good charge transport properties and stable performance with time [10].

To produce these crystals, chromium is evaporated on to wafers of highly n-type doped Liquid Encapsulated Czochralski (LEC) grown gallium arsenide. The wafers are then annealed at high temperature to diffuse the chromium throughout the crystal lattice producing a material with a complex compensation scheme. The resulting wafers have high resistivity ($\rho > 1 \times 10^9 \Omega \text{cm}$) and good charge carrier transport properties ($\mu_n \sim 4,000 \text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ and $\mu_p \sim 300 \text{cm}^2 \text{V}^{-1} \text{s}^{-1}$) suitable for the production of X-ray imaging detectors [11, 12].

While results from imaging experiments with GaAs:Cr material have been published [13] very few studies have focused on the radiation tolerance of the material, especially for hard X-ray energies. In this paper GaAs:Cr imaging detectors hybridized with the Hexitec spectroscopic readout ASIC have been used to study the effects of doses of up to 0.6 MGy of monochromatic 12 keV X-rays on the detector performance.

2 Experimental method

2.1 The Hexitec detector system

The Hexitec ASIC was originally designed for applications requiring excellent energy resolution in fields such as medical imaging, homeland security and materials characterisation [14] and has also proved to be an excellent tool for studying the processes of charge induction and trapping in compound semiconductor materials [15]. The ASIC consists of an $80 \times 80$ pixel array on a $250 \mu$m pitch ($50 \mu$m spacing) with each channel containing a high-sensitivity charge pre-amplifier, 2 $\mu$s CR-RC shaping amplifier and peak-track-and-hold circuit. Each channel of the ASIC is fully spectroscopic recording not only the position of interaction but the precise energy of each photon interacting in the detector volume.
GaAs:Cr detectors were fabricated with the Hexitec pixel array by Tomsk State University. Electrodes were deposited on wafers of material with the structure SiO$_2$/Au/Ni/Cu/V/GaAs/Ni (Anode/GaAs/Cathode) and diced into individual detectors each with dimensions of 20.35 mm $\times$ 20.45 mm $\times$ 0.50 mm. Detectors were bonded to the Hexitec ASIC using a low temperature curing (45$^\circ$C) silver-loaded epoxy and a Suss Microtec FC150 flip-chip-bonder and were read-out with the Hexitec Data Acquisition (DAQ) system.

The GaAs:Cr detector was operated at a bias voltage of $-300$ V and was kept at a constant temperature of 5$^\circ$C using a peltier cooled copper cold finger. The current in the detector was measured at all times using a Keithley 2410 source meter.

2.2 High flux X-ray irradiations

Prior to high flux irradiation, calibration data was collected using a 180 MBq $^{241}$Am $\gamma$-ray sealed source. The lines in the $\gamma$-ray spectrum were used to perform a linear energy calibration to each of the 6,400 channels of the detector. Once calibrated, the detector was irradiated at the B16 beam line at the Diamond Light Source Synchrotron to test the radiation tolerance of the material.

The detector was exposed to a beam of 12 keV mono X-rays with a flux of $\sim 10^9$ photons s$^{-1}$ mm$^{-2}$. Eight different areas of the detector with sizes 3–36 mm$^2$ were irradiated to different doses at a rate of $\sim 10$ Gy s$^{-1}$ with exposure lasting $10^1$–$6.6 \times 10^4$ s. After each of the 8 irradiations, a 100 $\mu$m thick sheet of copper was inserted between the direct beam and the sensor to reduce the count rate. This allowed spectroscopic data to be collected to determine if any radiation damage to the sensor had occurred.

3 Results

3.1 $^{241}$Am sealed source measurements

Figure 1 (left) shows a typical $^{241}$Am spectrum taken by a single pixel of the GaAs:Cr Hexitec detector. The lines in the spectrum were used to calibrate each pixel individually, the calibration plot for the same pixel is shown in figure 1 (right). In each case a linear regression produced a good fit with $\chi^2$ values of $> 0.99$. The FWHM of the $^{241}$Am $\gamma$-ray peaks at 26.0 keV and 59.5 keV were measured to be 1.1 keV and 1.8 keV respectively.

The large low energy tail observed at the higher energy is due to a larger interaction depth in the detector. The mean free paths of the 26.0 and 59.5 keV $\gamma$-rays in GaAs:Cr are 90.5 $\mu$m and 901.5 $\mu$m respectively. As the mean free path at the higher energy is much larger this results in additional charge loss due to scattering processes and an increased hole contribution to the signal for interactions occurring close to the anode pixels. Despite this tailing the energy resolution of the detector is one of the best reported for bulk GaAs:Cr [12].

3.2 Dose rate calculation

While under direct irradiation with the synchrotron X-ray beam the current in the GaAs:Cr detector was monitored. Figure 2 (left) shows how the measured value varied with time for each of the 8 irradiations. The radiation current was determined for each of the irradiations and, assuming a W-Value for GaAs:Cr of 4.30 eV ehp$^{-1}$, the beam flux was calculated to be $3.4 \times 10^8$ photons s$^{-1}$ mm$^{-2}$; see figure 2 (right).
Figure 1. (Left) A typical $^{241}$Am spectrum produced by a 500 mm thick GaAs:Cr detector bonded to the Hexitec readout ASIC. (Right) An example of a calibration of a single pixel.

Figure 2. (Left) The variation of leakage current in the detector as a function of time. Labelled on the plot are the 8 different exposures. (Right) The calculated beam flux calculated from the variation in radiation current as a function of irradiated area.

The calculated flux compared well to an independent measurement made with a calibrated silicon photodiode which gave a value of $5 \times 10^8$ photons s$^{-1}$ mm$^{-2}$. It was also observed that the radiation current in the detector was stable over $6.6 \times 10^4$ s showing a standard deviation ($\sigma$) of $< 0.5\%$. This suggests that the electric field in the detector is stable at these fluxes.

3.3 Radiation hardness measurements

As the X-ray flux incident on the detector is known, it is possible to estimate the dose deposited in the GaAs:Cr crystal. The Dose, in Gray, is defined as the energy deposited per unit mass and in this instance is be given by equation (3.1):

$$D(Gy) = \frac{\Phi A E t}{M}.$$  \hspace{1cm} (3.1)

Where $\Phi$ is the beam flux, $A$ is the irradiated area, $E$ is the energy of a single photon, $t$ is the irradiation time and $M$ is the mass of the GaAs:Cr crystal. To calculate the absorbing mass it was assumed that all the photons incident on the detector were absorbed in a volume defined by the mean free path of the X-rays in the material. For 12 keV photons in GaAs:Cr the mean free path was calculated to be 11 $\mu$m. Table 1 lists the values used for the dose calculation.
Table 1. The values used to calculate the absorbed dose in the GaAs:Cr detector. Some of the units have been converted for ease of calculation.

| Quantity                        | Value            |
|---------------------------------|------------------|
| Radiation Flux (photons s⁻¹ cm⁻²) | 3.4 × 10¹⁰      |
| Irradiated Area (cm²)           | 0.03–0.36        |
| Photon Energy (J)               | 1.9 × 10⁻¹⁵      |
| Time (s)                        | 10¹–6.6 × 10⁴    |
| Density (Kg cm⁻³)               | 5.32 × 10⁻³      |
| Mean Free Path (cm)             | 1.1 × 10⁻³       |
| Volume (cm³)                    | 3.3 × 10⁻³–3.9 × 10⁻⁴ |

Figure 3. (Left) The spectrum of the 12 keV measured before and after a 1 MGy exposure. (Right) The variation in the FWHM of the 12 keV peak as a function of dose. Error bars are calculated from the standard error in the mean FWHM value calculated from pixels in the irradiated area.

Between each of the 8 irradiations a 100 μm thick sheet of copper was introduced in to the direct beam reducing the beam flux to ~ 2000 photons s⁻¹ mm⁻². At this flux it was possible to collect spectroscopic data with the detector. Figure 3 (left) compares the spectroscopic performance of the detector before and after a 0.6 MGy irradiation. The performance was quantified using the FWHM of the 12 keV photo-peak per pixel in the irradiated area.

Before the irradiation an initial FWHM of 1.46 +/− 0.02 keV was measured. After each subsequent irradiation the FWHM showed little change as shown in figure 3 (right). The standard deviation in the measured FWHM across all 8 different irradiations was 0.02 keV (1.2%). These measurements suggest that, for the dose range and X-ray energy studied, the GaAs:Cr material is radiation hard.

4 Conclusions

A 500 μm thick chromium compensated gallium arsenide detector was fabricated by Tomsk State University with the Hexitec small pixel geometry. The assembled detector demonstrated excellent performance when tested with an ²⁴¹Am sealed source with FWHM of 1.1 and 1.8 keV measured at 26 and 59.5 keV respectively. Measurements of the radiation tolerance of GaAs:Cr were made on the B16 beam line at the Diamond Light Source synchrotron. The variation of the radiation current...
in the detector as a function of irradiated area gave a beam flux of $3.4 \times 10^8$ photons s$^{-1}$ mm$^{-2}$. Eight areas of the detector were irradiated for varying amounts of time to deposit different radiation doses. Measurements of the spectroscopic performance of the detector before and after each irradiation showed that there was little to change in the FWHM of the 12 keV photo-peak ($< 1\%$). This result suggests that, for the doses and X-ray energies studied, the GaAs:Cr is radiation hard and would be a suitable material for the production of next generation imaging detectors at high flux X-ray sources.

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