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Responsivity of Diamond X-ray Photodiodes Calibrated at NSLS

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Abstract. Single crystal, high purity synthetic diamond is used as photoabsorption and carrier transport medium in x-ray photodiodes. While the thermal / mechanical robustness and high x-ray transmission of diamond make such devices attractive for synchrotron instrumentation, state-of-the-art quality material and electrical interfaces further make such detectors feasible. The present work develops methodology for attaining calculable responsivity (photocurrent yield) over a wide range of photon energies (0.2 to 28 keV) to within 5% accuracy. These methods achieve linear response for up to 0.2 W absorbed x-ray power and response time as low as 1 ns. Details of contact formation / robustness and bias configuration are explored.

Keywords: Diamond, Single Crystal, Detector, Synchrotron, Photocurrent.
PACS: 29.40.-n; 29.40.Wk; 85.60.Dw; 07.85.Fv; 07.85.Qe; 72.40.+w

MOTIVATION

Diamond has been used as a robust x-ray sensor material for several decades [1-4]. Its high strength and thermal conductivity make it ideal for high radiation environments. However, only recently has material become commercially available with low enough impurity and defect density to realize its potential in radiometric applications such as synchrotron diagnostics and scientific instrumentation [5]. The present report describes the observation of calculable diode responsivity under controlled conditions, as measured at several NSLS synchrotron beamlines, spanning a wide range of photon energies and flux levels.

MATERIALS AND METHODS

Detector-grade synthetic diamond material available from Diamond Detectors Limited is characterized by single crystal grain orientation and ultra-low (sub ppm) nitrogen content. The nitrogen concentration is of particular importance as it directly relates to hole trapping in diamond; it is verified by infrared absorption spectroscopy. The crystal quality is confirmed by x-ray topography. Typical samples are polished square plates, measuring 4 mm by 4 mm by 0.5 mm, with the large surface normal parallel to <100> orientation of the crystal lattice. Each of the large sides are sputter-coated with a 3 mm diameter disk of 10-30 nm thickness of metal (we have so far used platinum, molybdenum, niobium, titanium, and copper) in order to provide electrical contacts. The metallized diamond sample is held in firm electrical and mechanical contact with a spring-loaded, vacuum-compatible mount.

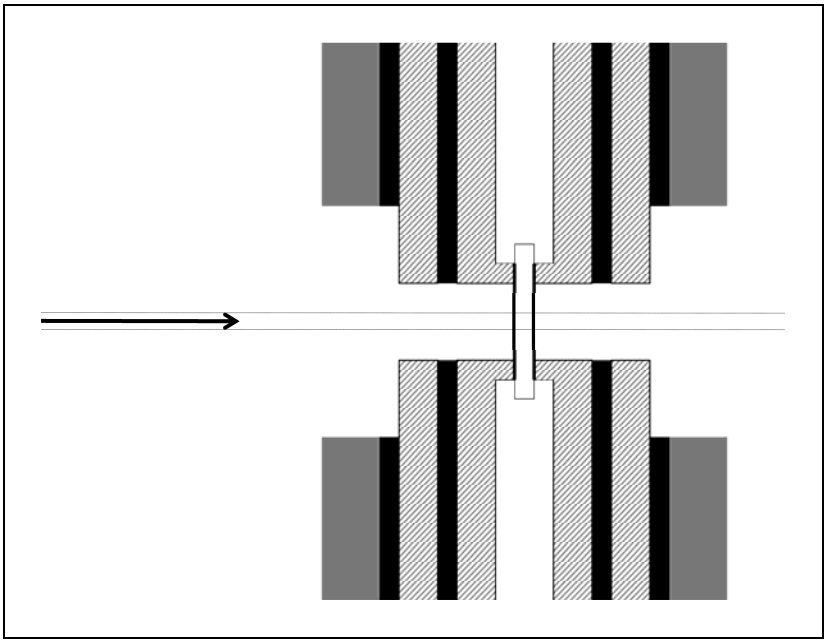

FIGURE 1. Cross-section of the metal-coated diamond sample in its mount, which provides both electrical contact and mechanical support. Black regions are ceramic insulator and light-hatched regions are addressable copper electrodes. An incident x-ray beam of 0.4 mm diameter is shown striking the sample from the left.

Responsivity testing of the diamond diode structures was performed at NSLS beamlines U3c, X6b, X8a, X15a, and X28c. Monochromatic x-rays are provided by beamline U3c over the range of 0.2 to 1 keV, by beamline X8a over 1-6.5 keV, and by beamline X15a over 6.5 to 30 keV; monochromatic intensity across these beamlines ranged from 1 nW to 10 uW per mm^2. X6b provides a monochromatic, focused x-ray micro-beam with energy between 8 and 19 keV for response uniformity tests and mapping. Beamline X28c provides intense (up to 0.1 W per mm^2) white or pink broadband x-ray photons from the NSLS x-ray bending magnet source with peak intensity near 10 keV. Incident flux levels were measured using calibrated silicon photodiodes [6], air-filled or nitrogen-filled ionization chamber, or copper calorimetric sensor depending on energy and intensity. Experiments were conducted in vacuum at U3c and X8a, in air at X15a and X6b, and in a nitrogen environment at X28c. X-rays were impinged upon the metallized diamond sample normal to the large surface, as shown in Fig. 1. The illuminated area was in most cases 0.1 mm^2 but was increased in some cases to as much as 2 mm^2. One contact is used to measure signal and the other is used to apply bias. Photocurrent responsivity measurements are made using Keithley electrometers, with a 10 nF capacitor to ground added for improved performance stability of this instrument. Bias is provided by 100x amplification of a low-voltage programmed signal using a linear amplifier from FLC electronics. Applied bias took the form of either DC level or square wave with adjustable frequency, magnitude, and duty cycle. At high energies, x-ray flux modulation was accomplished by means of aluminum transmission attenuators.

RESULTS

Current-mode responsivity data for a typical diode-behaving diamond, tested under monochromatic illumination is shown in Fig. 2. While bias for electron transmission showed photoconductive gain (apparent electron trapping), hole transmission was found to be linear with flux for duty cycles as high as 99%. Voltage of 100 V ($2x10^5$ V/m field) was found to be sufficient for extraction of 95% of the available photocurrent. Responsivity was found to be largely insensitive to bias pulse frequency in the range of 10 Hz to 100 kHz. The data are fit with a simple form with accounts for finite thickness of the active sensor material and effective thickness of window materials responsible for attenuation and/or recombination losses. Among a variety of samples, mean electron-hole pair production energy (corresponding to peak responsivity) was found to be a constant 13.3 eV (0.075 A/W) within 4%.

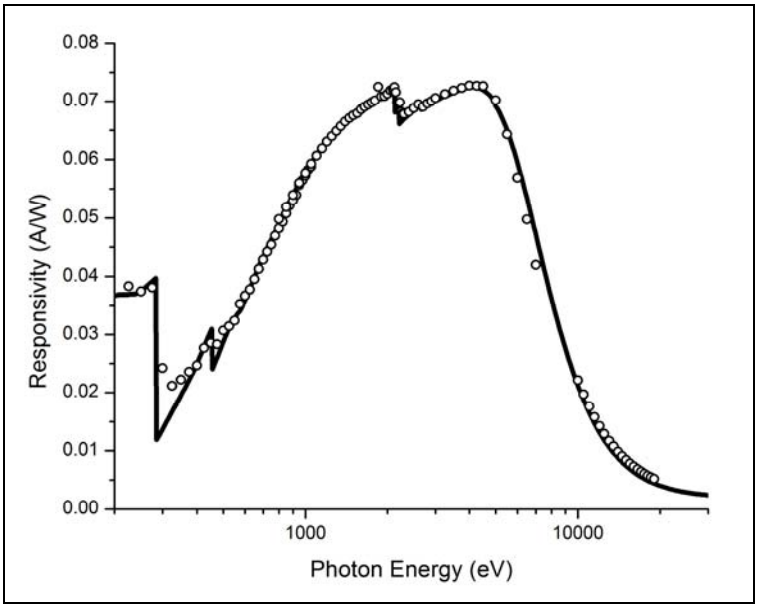

FIGURE 2. Responsivity (symbols=measured, curve=calculated) of a 0.5 mm thick single crystal diamond with Ti/Pt contacts (25 and 15 nm thicknesses, respectively). Peak responsivity corresponds to a native mean electron-hole pair production energy of 13.3 eV (0.075 A/W). Edge structure at the low energies indicates attenuation and recombination losses at the entrance window.

For low energy x-rays (below roughly 4 keV for these samples), selection of bias polarity implies a specific transmitted carrier type, since all photogenerated charge carriers are produced near the incident electrode; at higher energies, this distinction is lost. For example, at low energies, when a positive bias is applied at the incident electrode, the transmission of holes limits the observed photocurrent. When the photocurrent is thus limited by hole transmission, the photocurrent observed under DC bias condition is lower than that seen under pulsed-biasing for duty cycles at or below 99%, regardless of the contact type. On the other hand, when the photocurrent is limited by electron transmission, the steady-state value of the photocurrent under DC biasing is higher than the AC value by the ratio of the hole transit time to the electron trap lifetime (photoconductive gain) under ohmic contact conditions, but is similarly reduced for blocking contacts. That is, we observe charge trapping primarily of electrons. This issue is avoided in the high-flux and high-energy conditions of the X28c beam, where x-rays fully flood and permeate the material uniformly and DC bias can be used without complication. Otherwise, defect-free regions of the devices must be found which similarly can produce loss-free and gain-free photocurrent response under DC bias.

In an effort to explore the high-current and speed performance capabilities of such diamond sensors, the X28c beamline was also used. Diamond photocurrent was found to be linear with absorbed x-ray beam power in the range of 100 nA to 20 mA under illumination intensity spanning roughly 1 μW to 200 mW, with the same 13.3 eV mean ionization energy as seen for monochromatic tests, within 6%. Furthermore, the diamond was fitted into a high-speed mount (with semi-rigid coaxial cable and compensating capacitor), and under 1 kV applied bias the NSLS synchrotron bunches were readily observed with roughly 1 ns rise time.

Contacts and defects were found to be critical to the overall performance of these devices under current mode operation. Unannealed, oxygen-passivated contacts were found to preserve blocking performance, preventing photoconduction due to any charge trapping which may occur in the diamond. On the other hand, in an effort to produce a more robust device, annealing was tried, yet found to reveal or possibly even produce charge-trapping defects. Several tests performed under different treatment conditions found that blocking contacts can be reformed by either acid or ozone treatment, and that otherwise contaminated surfaces can easily produce leaky (high dark current) devices. “Dark” current was also found to be sensitive to humidity and UV spectral content of overhead lighting. Some annealed (ohmic) metal contacts did appear to reveal localized defects in the diamond as photoconductive gain “spikes” in response maps; this behavior is presently undergoing further investigation.

CONCLUSION

State-of-the art synthetic diamond has been found to provide a calculable x-ray photocurrent under controlled contact and biasing conditions. While improvements are underway, already we have seen that the potential for diamond-based synchrotron detectors is real. The possibilities for high-flux and high-speed applications have been demonstrated. The mean electron-hole pair formation energy for diamond of 13.3 eV provides a useful benchmark for identifying relative gain and loss due to charge recombination and photoconduction.

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