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Characterization of CdZnTeSe Nuclear Detector Chemically Etched in Bromine Methanol

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Abstract

Semiconductor nuclear radiation detectors made from tertiary and quaternary compounds of cadmium telluride (CdTe) can operate at room temperature without cryogenic cooling. One of such materials that have become of great interest is cadmium zinc telluride selenide (CdZnTeSe). Compared to other CdTe-based materials, such as cadmium zinc telluride (CdZnTe), CdZnTeSe can be grown with much less Te inclusions and sub-grain boundary networks. Chemical etching is often used to smoothen wafer surfaces during detector fabrication. This paper presents the characterization of CdZnTeSe that is chemically etched using bromine methanol solution. Infrared imaging shows that the wafer has no sub-grain boundary networks that often limit detector performance. The current-voltage (I-V) characterization experiment gave a resistivity of 4.6×10^{10} Ω-cm for the sample. The I-V curve was linear in the ±10 to ±50 volts range. An energy resolution of 7.2% was recorded at 100 V for the 59.6-keV gamma line of ^{241}Am.

Keywords

CdZnTeSe, Chemical Etching, Energy Resolution, Gamma-Ray Spectroscopy, Infrared Imaging, Nuclear Detectors

1. Introduction

The growing scientific, technological, medical, and industrial applications of rad-

iation detectors have attracted researchers to work to achieve design and fabrication of nuclear detectors of high resolution and precision that is operable at room temperature. There have been consistent and pungent research efforts worldwide in the past forty years that have been channeled to identify semiconductors for room-temperature x-ray and γ-ray detection [1]. Early efforts in the development of room-temperature semiconductor nuclear detectors include mercuric iodide (HgI_2), cadmium telluride (CdTe), cadmium zinc telluride (CdZnTe or CZT), and thallium bromide (TlBr) [2] [3] [4] [5] [6]. Among these materials, CdZnTe has been widely accepted due to its ability for high-resolution nuclear detection at room temperature without cryogenic cooling. CdTe and CdZnTe have found success commercially. Major companies that produce CdZnTe detector devices include Kromek Group PLC [7], Redlen Technologies [8], FLIR Systems [9], Eurorad [10], and H3D [11].

Notwithstanding many years of intense research, innovative improvement, and dominance in the market for its physical and optoelectronics properties, CdZnTe is still hurt by three major damaging imperfections which include high concentration of sub-grain boundaries and their networks, Te inclusion/precipitates and compositional inconsistency that severely degrade the energy resolution of detectors [1] [12] [13] [14]. The indiscriminate distribution of sub-grain boundary networks in the CdZnTe matrix and associated defects are responsible for uneven spatial distribution of trapping centers, which causes spatial variability in the charge transport properties throughout the active volume of CdZnTe devices [1] [15]. Such spatial non-uniformity of the charge collection efficiency is responsible for variability in the pulse-height spectra, depending on the drift paths of the ionized electrons, resulting in broadened photopeak [15]. Thus, there have been recent efforts to develop CdTe-based materials that are less prone to Te inclusions and sub-grain boundary networks. Examples of these materials are cadmium manganese telluride (CdMnTe or CMT) [16] [17]-[23] and cadmium zinc telluride selenide (CdZnTeSe or CZTS) [1] [24] [25] [26] [27] [28].

It has been shown that CdZnTeSe can be grown with much less Te inclusions and sub-grain boundary networks [1] [24] [25] [27]. The performance of CdZnTeSe and other CdTe-based detector could be limited by surface defects which are often produced during device fabrication. Most surface defects come from the cutting of detector wafers from ingots and during mechanical polishing. The processes for reducing and eliminating the fabrication-induced defects and residues include surface polishing, chemical etching, and chemical passivation [28]-[33]. In this paper, we present the characterization of CdZnTeSe that is chemically etched using bromine methanol solution.

2. Detector Fabrication

2.1. CdZnTeSe Material

The CdZnTeSe is a large-grain polycrystalline material with a grain area of approximately 2.5 × 1.75 cm^2 being the largest [1]. The material was grown by the

Traveling Heater Method (THM) using $Cd_{0.9}Zn_{0.1}Te$ and CdSe materials of 6N purity. The resulting polycrystalline material is $Cd_{0.9}Zn_{0.1}Te_{0.98}Se_{0.02}$. This is an improvement on CdZnTe since the addition of Se produces a better material with less defects associated with Te inclusions and sub-grain boundary networks. CdZnTe materials are prone to performance-limiting defects that results from Te inclusions and sub-grain boundary networks.

The random distribution of Te inclusions and sub-grain boundary networks in the CdZnTe matrix causes inhomogeneous distribution of charge-carrier trapping centers. The result is spatial variability of the transportation of charge careers in the detector matrix, thus causing degradation to energy resolution, especially in applications that require thick detector of about 1 cm or more [1]. The lesser concentration or absence of performance-limiting Te inclusions and sub-grain boundary networks and better crystalline homogeneity in CdZnTeSe implies that more detector-grade wafers can be harvested from the as-grown polycrystalline ingot, and thus reduce the cost of production compared to CdZnTe. The wafer used in this study was cut from the as-grown ingot of the $Cd_{0.9}Zn_{0.1}Te_{0.98}Se_{0.02}$ material.

2.2. Cutting of Detector Wafer

The fabrication of CdTe-based nuclear radiation (x-rays and γ-rays) detection devices starts with the cutting of wafers of desired sizes from the ingot. We used a cutting machine that is equipped with a diamond impregnated wire saw. The wafer used in this study was cut from as-grown CdZnTeSe ingot. The cutting machine is programmable and can be set to cut wafers of desired dimensions. It consists of a movable platform on which the ingot or large wafer is mounted. The platform is automatically moved between cutting cycles based on the dimensions set in the controller. There is a water pump attached to the system that runs water on the sample through a nuzzle. The water serves as a coolant and a lubricant. The cutting process often introduces surface damages and defects. These could increase surface leakage currents and result in the trapping of charge carriers generated by ionizing radiation, and thus increase electronic noise, which in turn degrade the energy resolution of the detector [34] [35] [36]. The three major steps used in removing fabrication-induced damages and defects are mechanical polishing, chemical etching, and surface passivation. Chemo-mechanical polishing is sometimes used as an alternative to chemical etching. The sample used in this study has the dimensions of $6.7 \times 5.7 \times 1.8$ mm^3 after mechanical polishing.

2.3. Surface Polishing

Mechanical polishing is used as the first stage of removing surface damages caused by cutting and dicing the wafers. The rough surfaces from the cutting process could sometimes create new defects that act as trapping centers for charge carriers transported between the detector's electrodes [31]. The surface

polishing process starts with the mechanical polishing of the surface of CdZnTeSe wafer with silicon-carbide abrasive papers to produce smooth mirror-like surfaces. This is followed by fine polishing with 3.0- to 0.1-μm alumina (Al_2O_3) powder on a MultiTex pad [31] [37] [38], resulting in a final sample area of 6.7 × 5.7 mm^2 and a thickness of 1.8 mm. Further smoothing of the surface is often achieved through chemical etching with chemicals that do not alter the stoichiometry of the surface [4].

2.4. Chemical Etching

Chemical etching is deployed at the second stage to remove fine residual mechanical damages using bromine-methanol solution of 1% - 2% concentration, a typical etching chemical [38] [39]. Kim *et al.*, [38] reported a surface roughness reduction of CdZnTe wafers from 9.25-nm root mean square (rms) for mechanical polishing to 2.50-nm rms after etching in 2% bromine-methanol (Br:MeOH) solution. However, bromine-methanol solutions superficially induce surface features that make the wafers more conductive which alter the material's surface and interfacial properties, so that it results in increased surface conductivity [32] [39] [40]. A CdZnTe wafer that was fine polished with 0.3-μm alumina powder followed by 1% Br:MeOH solution was reported to yield lower surface currents compared to similar samples that were polished and etched before depositing the gold contacts [40]. Chattapadhyay *et al.* [39], in an earlier study, reported that the effect of increasing the surface current by etching in Br:MeOH solution was lowered by passivating the sample in a potassium hydroxide (KOH) aqueous solution, which leaves a more stoichiometric and smoother surface.

The CdZnTeSe sample used in the present study has dimensions of 6.7 × 5.7 × 1.8 mm^3. It was chemically etched using Br:MeOH solution. The etching process involve dipping the wafer into a 2% solution of bromine methanol for 2 minutes, and then dried with compressed air.

2.5. Surface Passivation

Surface passivation can be regarded as the third stage of removing impactions and surface damages on the CdZnTeSe wafer to enhance the performance of an x-ray and γ-ray nuclear detector at room-temperature. In surface passivation, the chemical treatment is used to minimize surface oxidation, and increase the detector's shelf and operational life [3]. Kim *et al.* [38] used ammonium sulfide to passivate CdZnTe after etching in a 2% bromine-methanol solution and found that the apparent mobility-lifetime product of the 2-mm-thick CdZnTe detector improved by 21%, with a noticeable decline in the leakage current, surface-state densities, and improved performance of the detector. It was also reported that, by removing the space-charge accumulated on the detectors' side surfaces, the ammonium sulfide passivation of the CdZnTe 9-mm thick detectors, supports establishing a "focusing" internal electric field within the detector's cavity that coxed electrons away from the surface and towards the anode [38]. Further re-

search efforts to improve the etching process, as well as replacing the chemical etching step in fabricating devices with chemo-mechanical polishing have been deployed in previous work. Improvement efforts include using either a low concentration bromine-based etchant with a surface-passivation reagent and a non-bromine-based etchant or a chemo-mechanical polishing in place of full chemical etching [3] [32] [41].

2.6. Deposition of Electrical Contacts

Gold electrical contacts were deposited on the two opposite 6.7×5.7 mm^2 surfaces of the CdZnTeSe wafer. This involved pipetting gold chloride solution on each surface. After the gold chloride solution reacts with the surface, the excess solution is removed using a felt paper.

In the boundary layer between metals (contacts) and semiconductors there exist thermal stress at the contact-wafer interface caused by the mismatch of the thermal expansion coefficient between the electrode's material and the CdZnTeSe crystal. Thus, it plays an important role in the determination of the electrical and transport characteristics of the contacts [42]. The method of electroless contact deposition is generally used because of its simplicity and it creates a stronger chemical bond between the metal and the semiconductor compared to sputtering and thermal evaporation methods of which both can create defects in the interface region, leading to trapping effects [3] [43]. It has been reported in literatures that gold (Au) is found to be the contact of choice, because it is sufficiently stable chemically and mechanically to allow bonding and prolonged operation, thus reducing current leakage [44]. Zhang *et al.* [45], reported that the electroless Au deposition method creates more inter-diffusion between the contact and CdZnTe material, so resulting in a better gamma response compared to other metal contacts and techniques. Thus, we adopted and used the electroless Au contact deposition technique in this study.

3. Detector Characterization Experiments and Results

3.1. Infrared Transmission Imaging

An infrared transmission imaging was performed after chemical etching, prior to the deposition of electrical contacts. A Nikon Eclipse LV100 microscope was used. The microscope is equipped with an infrared light source, a motorized xyz-translation stage, and an infrared camera that is connected to a computer. It comes with a software for image capturing and analysis. The matrix of the CdZnTeSe wafer is transparent to the infrared light while Te inclusions are opaque. Hence, any Te inclusion present will appear as a dark spot as the infrared light transmits through the bulk of the wafer. The infrared image is shown in Figure 1. The image shows that the CdZnTeSe wafer is almost free with Te inclusions and there are no sub-grain boundary networks. Since Te inclusions trap charge carriers, this image indicates a very good wafer for charge collection uniformity, and hence good radiation detection efficiency [4].

3.2. Current-Voltage Measurements

Current-voltage (I-V) measurements were carried out to determine the I-V characteristics and resistivity of the wafer. High resistivity of the order of 10^8 Ω-cm and above is required for CdTe-based detectors to have low leakage current in the nanoamp range [23]. The resistivity for the CdZnTeSe wafer is 4.6×10^{10} Ω-cm. A customized aluminum box was used for the I-V measurements. The box is fitted with a Keithley model 6487 Picometer and Voltage source. The I-V measurement results are shown in **Figure 2**.

3.3. Detector Response Characterization

The detector repose measurement system consists of a standard eV Product sample holder connected to a high voltage power supply, a pre-amplifier, a shaping amplifier, and a multichannel analyzer (MCA) that is connected to a computer with a software for spectral analysis. See schematic diagram in **Figure 3**. The sample holder in **Figure 3** is made of brass and has a beryllium window against
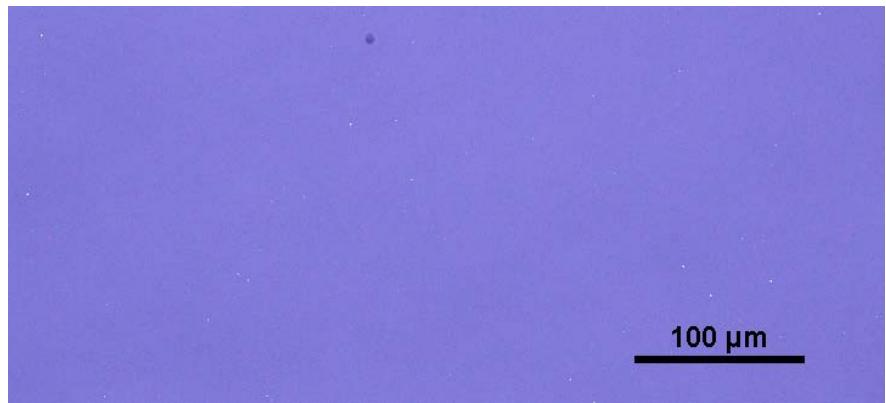
Figure 1. Infrared transmission image of part of the CdZnTeSe wafer. It shows that the wafer is almost free with Te inclusions, and there are no sub-grain boundary networks.
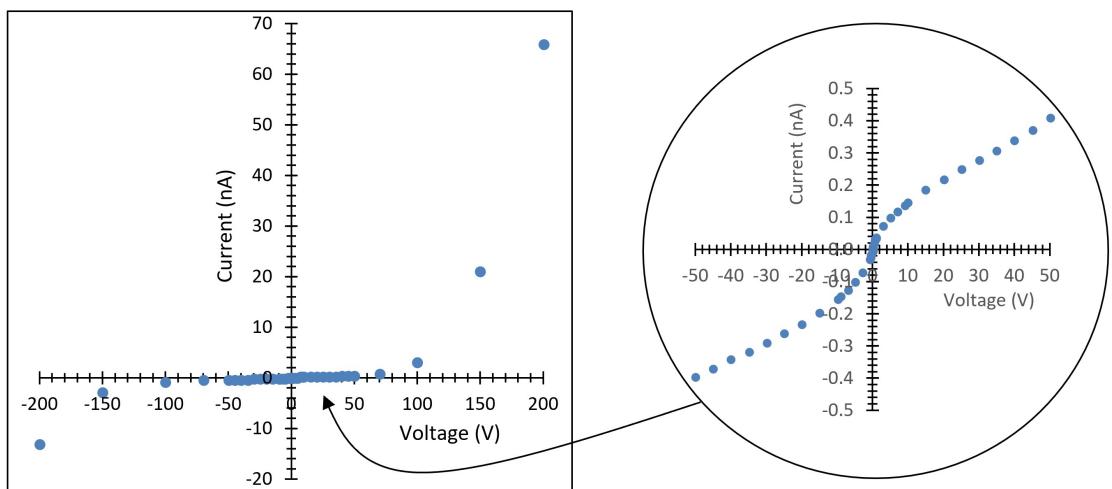

Figure 2. I-V plot for the CdZnTeSe detector. The curve is symetrical and linear in the ±10 to ±50 volts range. Resistivity is 4.6×10^{10} Ω-cm.
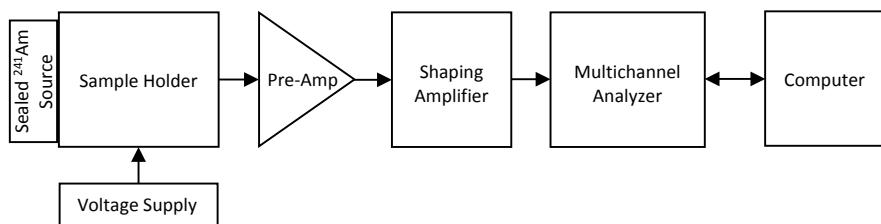
Figure 3. Schematic diagram of the detector repose measurement system (not drawn to scale).
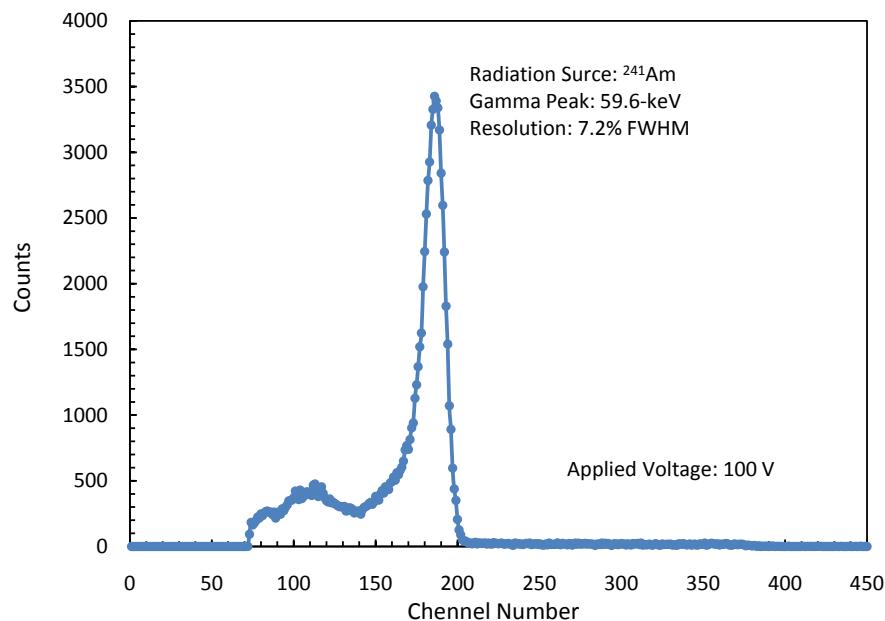

Figure 4. Response of the CdZnTeSe detector to a sealed [241]Am gamma-ray source at 100 V applied voltage.

which a sealed [241]Am radiation source is placed. The applied voltage was 100 V. The recorded spectrum is shown in **Figure 4**. The detector resolution measured as full-width-at-half-maximum (FWHM) for the 49.6-keV gamma line of [241]Am is 7.2%.

4. Conclusion

We have characterized a CZTS nuclear detector that is chemically etched using bromine methanol solution. The infrared transmission imaging result showed that the CZTS crystal has no grain boundary network, and it is mostly free of Te inclusions. This implies that the CZTS matrix does not have defects that could adversely affect the transport of charge carriers. The current-voltage curve is linear in the ±10 to ±50 volts range and gave a resistivity of 4.6×10^{10} Ω-cm. The recorded energy resolution is 7.2% at 100 V bias for the 59.6-keV gamma line of [241]Am. This CZTS has shown to be an effective material for the detection of γ-rays at room temperature. In future studies, we plan to focus on surface passivation, and on the variations in surface current with time after chemical etching.

Acknowledgements

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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