Compensation and trapping in CdZnTe radiation detectors studied by thermoelectric emission spectroscopy, thermally stimulated conductivity, and current-voltage measurements


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Abstract

In today’s commercially available counter-select-grade CdZnTe crystals for radiation detector applications, the thermal ionization energies of the traps and their types, whether electron or hole traps, were measured. The measurements were successfully done using thermoelectric emission spectroscopy (TEES) and thermally stimulated conductivity (TSC). For reliability, the electrical contacts to the sample were found to be very important and, instead of Au Schottky contacts, In Ohmic contacts had to be used. For the filling of the traps, photoexcitation was done at zero bias, at 20 K, and at wavelengths which gave the maximum bulk photoexcitation for the sample. Between the temperature range from 20 to 400 K, the TSC current was found to be on the order of ~10000 times or even larger than the TEES current, in agreement with theory, but only TEES could resolve the trap type and was sensitive to the deep traps. Large concentration of hole traps at 0.1 and 0.6 eV were observed and smaller contraction of electron traps at 0.4 eV was seen. These deep traps cause compensation in the material and also cause trapping that degrades the radiation detection measurement.

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I. Introduction

In recent years, room temperature semiconductor radiation detectors have found myriad of applications [1]. For the detection of gamma-rays, Cd$_{0.9}$Zn$_{0.1}$Te (CZT) has been an excellent candidate, because (1) it has a large energy band gap necessary for room temperature operation and (2) a large cross section for the photoelectric absorption of gamma-rays, which is necessary for efficient conversion of the gamma-ray energy to electrical energy. However, in general, for a material to be a suitable room temperature semiconductor radiation detector, it must have high resistivity, on the order of $10^9 \, \Omega \, \text{cm}$, to minimize the noise due to the leakage current, and it must also have large electron and hole drift lengths [2], for maximum charge collection efficiency and maximum signal.

The carrier drift length is defined as the product of the carrier mobility and lifetime, and, physically, it is approximately equal to the distance that an electron or a hole travels before becoming trapped. Since the carrier mobility at room temperature is determined primarily by phonon scattering and the electronic energy band structure, it is the lifetime of the electrons and holes that must be long to give large carrier drift lengths.

As grown by most methods, CZT has a problem that it has relatively large concentrations of impurities and native defects that cause low resistivity and short carrier drift lengths. A solution to this problem is to compensate for these unintentional impurities and defects by introducing additional impurities or native defects. A well known and understood example of compensation is semi-insulating GaAs, in which Cr and O create deep electron and hole traps that pin the Fermi level near middle of the energy band gap, at the expense of shortening the carrier lifetimes [3].
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For CZT, compensated semi-insulating materials are currently available, and most of these are made by the high-pressure Bridgeman growth [4]. However, the mechanism of compensation is not understood and the yield of usable CZT is low. Understanding and controlling the compensation mechanism is the key to increasing the yield of usable CZT for radiation detection applications.

Our approach to studying compensation and trapping in CZT has been to use thermoelectric emission spectroscopy (TEES) [Ref. 5], thermally stimulated conductivity (TSC) [Ref. 6], and current-voltage measurements (I-V). TEES is a recently invented spectroscopy primarily for measuring the trap types, i.e. whether a trap is an electron or a hole trap, and the trap energies of deep traps in semiconductor materials. In addition, for some semiconductor materials, it is possible to infer the trap cross sections and trap concentrations. In TEES, the traps are filled at low temperature and they are made to detract by increasing the sample temperature. A thermal gradient is maintained across the sample to force the detrapped electrons and holes to move toward the colder electrode, and an electrometer measures the current at the electrodes. This is shown schematically in Fig. 1. Due to opposite charges, the electron and hole currents give opposite signs, thereby enabling the distinction between the electron and hole traps. The temperature and the rate at which the traps empty give their energy with respect to the conduction band, for the electron traps, or the valence band, for the hole traps. The shape and the magnitude of the TEES currents can give the trap cross sections and the concentrations.

TSC is a similar spectroscopy used primarily for measuring the trap energies, and, sometimes, the trap cross sections and concentrations. The experimental difference between TSC and TEES is that, instead of applying a temperature gradient across the
sample an electric field gradient is applied across the sample in TSC. As shown on Fig.1, because of the internal electric field, the detrapped electrons and holes move toward the anode and the cathode, respectively, giving currents that have the same sign.

The main advantages of TEES are that (1) it enables distinction between electron and hole traps (this is important in trying to understand the compensation mechanism) and (2) it is sensitive to deep traps that detraps only near room temperature or above. Lacking these two advantages of TEES, TSC has the advantage that its signal is much larger than TEES. This will be explained in the theory section. In this paper, in addition to discussing the compensation and trapping in CZT, we will discuss the experimental and the theoretical aspects of TEES in some detail, in order to establish the method as a reliable deep trap spectroscopy.

II. Experimental

The experimental measurements were taken using an integrated and automated data acquisition system developed recently at Sandia National Laboratories by the authors, and it is schematically shown in Fig. 2. The system consists in part of a closed-cycle helium cryostat that allowed the sample temperature to vary between 14 K and 475 K [Ref. 7]. The sample stage was homemade, to provide an adjustable spring-loaded electrical and thermal contact to a horizontally placed sample having a range of dimensions from 1 to 10 mm thick and up to 1 cm wide. The sample holder also had two mirrors to allow the external light from the view port of the cryostat to illuminate a side face of the sample. Two silicon diode sensors were placed on the sample stage to measure the temperature at the top and bottom electrodes. In thermal equilibrium, the
sensors read the same temperature within 0.1 K. The two signal wires were both made of Au to avoid thermoelectric voltage from the electrical junctions.

During the measurements, the cryostat was pumped to $10^{-7}$ Torr using a turbomolecular pump and a backing roughing pump, to avoid the condensation of moisture and atmospheric gases. The optical view port of the cryostat was covered by a metallic box, for shielding from stray light and electrostatic disturbances. The signal wires from the cryostat was connected by a triaxial cable to an electrometer that had fA sensitivity and also provided a voltage source. The sample temperature was controlled by a temperature controller, that monitored the two silicon diode sensors in the cryostat and also heated the sample stage. The heating rate varied from 0.1 to 0.5 K/sec. Both the electrometer and the temperature controller were interfaced to a personal computer for automated data acquisition. The I-V measurements were made in dark at $10^{-7}$ Torr and at stable temperatures.

For the photoexcitation at low temperature to fill the traps for TEES and for TSC, a tungsten halogen lamp was the light source. Two lenses were used to guide the light to a monochromator and to match its numerical aperture. The monochromator slit width was set to give 5 nm bandwidth, and the wavelength was varied between 200 nm and 1 µm. The light from the monochromator was coupled by a fiber optic bundle to the view port of the cryostat with stray-light rejection. The wavelength of the light was varied at 5 nm intervals at low temperature while the sample was held at 10 V, to find the optimum wavelength. For too large wavelengths, there was negligible photoexcitation of electrons across the energy band gap, and, for too small wavelengths, photoexcitation occurred only at the illuminated surface, where the materials properties differ significantly from
the bulk. At the optimum wavelength, the maximum photocurrent is obtained due to the
photoexcitation of the largest number of electrons from the valence band to the
conduction band. In this case, the photoexcitation occurs preferentially in the bulk of the
sample, rather than at the surface, thereby filling electron and hole traps away from the
surface. After establishing the optimum wavelength for the photoexcitation, which was
750-780 nm for the samples for this study, the photoexcitations prior to TEES and TSC
were done at zero bias, to avoid polarization effects. The sample was also initially cooled
at zero bias, for the same reason.

The three samples used for this study were supplied by eV Products, located in
Saxonburg, Pennsylvania. Samples A and B were purchased this year, were 1x1x0.2
cm\(^3\), and were from the same ingot. Sample C was purchased last year and it was
0.3x0.3x0.2 cm\(^3\). All three samples were chosen at random, and they were categorized as
count-select grade [8] by the supplier. As received, the samples had two Au contacts on
opposite faces, and they also had red coating between the Au contacts.

It was necessary to remove the red coating for the photoexcitation of the sample.
The removal was done by an acetone etch followed by a methanol rinse. To remove the
Au contacts, the contacts were mechanically polished using a fine grain sand paper and
rinsed in methanol. To obtain an Ohmic contact, pure In was applied while momentarily
heating the sample on a hot plate at 200 °C.

III. Theory

In the steady state, the electrical current in a nondegenerate semiconductor can be
described by the following generalized drift-diffusion equation derived from the
Boltzmann transport equation, under the relaxation time approximation and diffusive approximation for nondegenerate semiconductors [9].

\[ J_e = (\lambda_e + 1)\mu_e n kT \nabla T + \mu_e kT \nabla n + e\mu_e n E, \]  
Eq. (1)

\[ J_h = -(\lambda_h + 1)\mu_h p kT \nabla T - \mu_h kT \nabla p + e\mu_h p E, \]  
Eq. (2)

where the subscripts distinguish the electron and hole parameters and \( \mu \)'s are mobilities given by

\[ \mu = \frac{e\tau_0}{m}(kT)^\lambda \frac{4}{3\sqrt{\pi}} \Gamma(\lambda + \frac{5}{2}), \]  
Eq. (3)

\[ \tau = \tau_0 e^\lambda, \]  
Eq. (4)

where \( \Gamma \) is the Gamma function, \( \tau \) is the relaxation time of the charge carriers as a function of their kinetic energy, and both \( \lambda \)'s and \( \tau_0 \)'s, are, in general, different for the electrons and holes. Near room temperature, phonon scattering dominates the carrier relaxation process [10] and \( \lambda = -\frac{1}{2} \). At low temperature, ionized impurity scattering is expected to dominate and \( \lambda = \frac{3}{2} \).

In the absence of a temperature gradient, one obtains the usual drift-diffusion equations from Eq. (1) and Eq. (2). In TSC, one applies a constant bias across the sample and there is negligible temperature gradient and negligible carrier concentration gradient. Hence in TSC, the first and second terms of Eq. (1) and Eq. (2) vanish and the TSC current is proportional to the electrical conductivity, \( \sigma = e\mu_e n + e\mu_h p \). For TEES, there is a constant temperature gradient across the sample, but there is negligible voltage gradient and negligible carrier concentration gradient. Therefore, for TEES, only the first term of Eq. (1) and Eq. (2) survives.
One should note that the ratio of the third term to the first term is \( \frac{eV}{(\lambda + 1)k\Delta T} \), where \( V \) is the voltage across the sample in TSC and \( \Delta T \) is the temperature difference across the sample in TEES. For typical experiments, \( V = 10 \) V and \( \Delta T = 10 \) K, and, therefore, the ratio is on the order of \( \sim 10000 \), implying that the TEES current should be smaller than the TSC current by this factor or even more; if electron and hole peaks in TEES overlap, they will result in partial cancellation of the currents, whereas, in TSC, the currents will add. We will examine the experimental ratio in a following section.

**IV. Result and discussion**

In this section, we will discuss results from 3 counter-select grade samples recently purchased from eV Products and refer to them as samples A, B, and C. Samples A and B were from the same growth ingot, and sample C was from another. The supplier tested the radiation detection performance of the material and provided the pulse height spectrum for each. Sample A was the best radiation detector, in terms of the energy resolution for 59 keV gamma-rays. In this section, we will discuss I-V measurements first, because we found that Ohmic contacts are crucial for TEES, TSC, and bulk resistivity measurements. Next we will describe our trap filling procedure using photoexcitation, since proper trap filling is prerequisite for successful TEES and TSC. And then, we will discuss our TEES and TSC results. These results will enable us to construct an energy level diagram of the observed traps, and also enable us to discuss the compensation and trapping in these materials.

**IV.A. Current voltage measurements and metal-semiconductor contacts**

To reliably perform the spectroscopies of deep traps and to measure the bulk resistivities, we found it critical to have Ohmic contacts on the samples. For example,
Fig. 3(a) shows a I-V plot of sample A at 293.7 K. For this measurement, the sample had the Au contacts made by the supplier [11], and the only processing we performed was the removal of the red coating from the samples. The symmetric “S” shape in the I-V curve was seen for all the samples from the supplier, and it shows that the Au contacts form back-to-back leaky Schottky barriers, giving the minimum differential resistance, \( \frac{dV}{dl} \), at zero bias. The inset shows that \( \frac{dV}{dl} \) at zero bias is 8.3x10^9 Ω. When the Au contacts were removed and In contacts were applied, the I-V curve changed, as shown in Fig. 3(b). The “S” shape was replaced by a curve indicative of Ohmic contacts and \( \frac{dV}{dl} \) at zero bias decreased by a factor of 2.2 to 3.8x10^9 Ω. In contrast to Fig. 3(a), \( \frac{dV}{dl} \) decreases with voltage in Fig. 3(b). Since sample A had dimensions of 10x10x2 mm^3, the corresponding bulk resistivity is 1.9x10^10 Ω cm, consistent with the range of values reported in literature for such CZT materials. It is clearly very important to have Ohmic contacts for bulk resistivity measurements.

We also found that it is important to have Ohmic contacts for TEES and TSC measurements, because, otherwise, the electrical resistance of the contacts can profoundly alter the magnitude and the shape of the spectra. For example, in TSC, it will be shown later that, for a peak in TSC spectra, the current can be as large as 10 µA with 10 V bias, even at 50 K. This implies a bulk sample resistance of less than 1 MΩ. If the sample contact resistance is larger than the sample bulk resistance, then most of the voltage drop across the sample occurs at the contacts. Since the current is proportional to the electric field inside the sample, the reduced field results in a much reduced TSC
current. We have seen the magnitude of the TSC current change by many orders of magnitude, due to the contacts. We have also seen the TEES and TSC spectra shapes change due to the contacts, but a detailed discussion of the effects of bad contacts are beyond the scope of this paper.

**IV.B. Filling traps by photoexcitation**

At low temperatures, the free carrier concentration of a nondegenerate semiconductor approaches zero. For our TEES and TSC experiments, to fill the traps at low temperature, we increased the free carrier concentrations by photoexcitation of the electrons across the energy band gap. Some of the resulting free carriers became trapped, and, to maintain local charge neutrality, the free carrier concentrations near the traps increased, resulting in increase of the photoconductivity with trap filling. By applying a bias, we saw the photocurrent increase with trapping, and this was a convenient means of monitoring the progress of trap filling. During this study, we found that, with our setup and the samples, the optimum wavelengths were between 750 and 780 nm, and the photoexcitation increased the photocurrent, at 10 V applied bias, from ~0 pA to on the order of 1 nA in 30 minutes. Furthermore, the rate of the increase of the photocurrent was nearly linear at the end of the 30 minutes, indicating that only partial filling of the traps occurred. Fig. 4 shows the photocurrent as a function of time for a typical sample.

For TEES and TSC experiments, we applied zero bias during photoexcitation, to eliminate the possibility of polarization of the internal electric field of our samples. It was found that the thermal velocities of the free carriers were large enough, even at 20 K, that biasing was unnecessary for the trap filling. For a given sample, the biasing was
done only once, in order to determine the optimum wavelength for the photoexcitation and to monitor the initial progress of the trap filling.

IV.C. TEES and TSC measurements

The TEES and TSC measurements were taken after the Au contacts were removed and In contacts were applied. The I-V measurements and the photoexcitations were done using the procedures described in the previous sections. The TEES and TSC spectra for sample A are shown in Fig.5(a). The TSC spectrum shows only one large peak near 40 K. The TEES spectrum, taken under the identical photoexcitation conditions and the heating rates, (1) identifies the peak as a hole trap peak, (2) shows additional peaks in the temperature range between 150 K and 300 K, and (3) identifies the room temperature electrical conductivity of the sample as n-type. In Fig. 5(b) is shown an expanded plot of the TEES spectrum between 100 and 300 K. Two additional electron traps and two additional hole traps can be seen.

Fig. 5(c) shows an expanded plot of the room temperature region where the thermal excitation of the electrons across the energy band gap begins to have an appreciable effect. It can be seen that the TSC current is larger than the TEES current by a factor of ~ 4000, consistent with our theoretical expectations.

In Fig. 6 are shown the TEES spectra for samples B and C. In addition to the traps observed in sample A, more traps can be resolved. The TSC spectra for the samples are now shown, because they do not contain any additional information.

IV.D. Energy level diagram

The temperature and the heating rate determine the trap thermal ionization energy. The following formula was originally derived for TSC [Ref. 12], but it is now commonly
used for both TSC and TEES, to determine the trap energies of the electron traps. It assumes negligible retrapping and that the trap cross section and lifetime are relatively slowly varying function of the temperature.

\[ E_t = kT_m \ln \left( \frac{cm^+ \sigma E_{T_m}}{\beta} \right), \quad \text{where } c = \frac{4 \sqrt{6 \pi^3 k^3}}{h^3}. \quad \text{Eq. (5)} \]

In the above equation, \( E_t \) is the trap energy, \( k \) is the Boltzmann constant, \( T_m \) is the temperature of the TEES peak, \( m^+ \) is the effective mass of the electrons, \( \sigma \) is the trap cross section, \( \beta \) is the heating rate, and \( h \) is the Planck’s constant. For \( m^+ \), we use 0.14 \( m_e \), where \( m_e \) is the free electron mass. This is the known effective mass for CdTe and we use this, because the Zn concentrations in the CZT samples are \( \sim 0.1 \) [Ref. 4].

For hole traps, Eq. (5) applies as well, with the replacement of the electron effective mass by the hole effective mass.

In Eq. (5), the trap cross section is unknown. Realistically, it is expected to vary between \( 10^{-12} \) to \( 10^{-18} \) cm\(^2\), \( 10^{-15} \) cm\(^2\) being typical of a neutral trap. A negatively charged electron trap or a positively charged hole trap would have a smaller cross section, and a positively charged electron trap or a negatively charged hole trap would have a larger cross section. Due to the logarithmic dependence of the trap energies on the trap cross section, the 6 orders of magnitude variation in the trap cross section propagates as only \( \sim 30 \% \) variation in the trap energies.

Based on the trap energies, we can form an energy level diagram for CZT. The electron and the hole traps observed in samples A, B, and C are plotted in Fig. 7. It can be seen that similar traps exist in all three samples. At the present time, it is unknown what the levels are. However, it has been reported recently that, in CdTe, V\(_{Cd}\) causes
hole traps at \(-0.2\) eV and \(-0.8\) eV, and that TeCd causes an electron trap at \(-0.4\) eV [Ref. 13]. We speculate that some of the observed traps are these. Elemental analysis of these materials have shown the presence of other impurities such as Ni and Al [14].

It is important to note that shallow traps less than \(-100\) meV deep cannot be measured using our current setup, because our lowest attainable temperature is 20 K, at which the shallow traps are already empty. It is very likely that shallow levels do exist, since some of the observed impurities are known to be shallow n-type dopants.

**IV.E. Compensation and trapping**

The energy level diagram shown in Fig. 7 allows us to consider compensation and trapping. The common deep traps, E1, E2, H4, and H5 can compensate the shallow traps to pin the Fermi level near the middle of the energy band gap, depending on their concentrations and charge states. We have previously shown how this can happen, using our earlier TEES data [15]. However, the quantitative determination of trap concentrations requires knowing the carrier mobilities and lifetimes as a function of the temperature, and we are currently trying to measure these variables. In addition, it may be necessary to saturate the traps by either providing longer duration for photoexcitation or by using a more intense light source.

The same deep traps that compensate the shallow traps also cause carrier trapping. It can be shown from the principle of detailed balance that the electron trap decay time, \(\tau_d\), is related the trap cross section and energy by the following expression.

\[
\tau_d = \frac{1}{N_e \sigma v \exp(-\frac{E_d}{kT})}, \quad \text{Eq. (6)}
\]
where \( N_c \) is the effective density of states for the conduction band and \( v_{th} \) is the thermal velocity of the electrons. Physically, the electron decay time is the approximate duration that an electron, once it falls into a trap, spends in that trap, before detrapping. Naturally, it is a strong function of the temperature. In Fig. 8, the trap decay times for the electrons and holes are shown as a function of the trap energy for CZT at room temperature, assuming an average trap cross section of \( 10^{-15} \) cm\(^2\). It can be seen that the shallow traps detrap very fast, in less than 1 nsec, whereas the deeper ones take hours or even longer to detrap. Because the typical nuclear electronics operates in the time scale on the order of 1 \( \mu \)sec, the traps shallower than \(~ 200\) meV detrap so fast, on the order of 0.1 \( \mu \)sec or less, that they do not significantly degrade the radiation detection measurements. The traps deeper than \(~ 200\) meV, the same ones that are also very important for the compensation, are the ones that degrade the radiation detection measurement. The best radiation detector material would have the minimum concentration of deep traps required for compensation.

V. Conclusion and future

In conclusion, we have performed TEES, TSC, and I-V measurements on commercially available counter-select grade CZT crystals for radiation detector applications purchased recently from a US supplier. The I-V measurements showed that the detector have leaky Schottky contacts made by the supplier, and In Ohmic contacts were necessary for making accurate bulk resistivity measurements and also for doing TEES and TSC. When the traps were properly filled, we found the photocurrent to monotonically increase with time, and we found it necessary to have Ohmic contacts and to chose the optimum wavelengths. When TEES and TSC spectra were compared, it was
seen that the TSC currents were at least on the order of ~ 10000 times larger than the TEES currents, in accordance with theoretical expectations. The TEES spectra allowed us to identify the trap types and to determine the trap thermal ionization energies. The deep traps observed cause compensation and also cause trapping in the material. Large concentration of holes traps ~ 0.1 and ~ 0.6 eV deep were observed, and electron traps ~0.4 eV deep were seen. The large concentration of the hole traps is consistent with the known shorter lifetime of the holes in these materials.

In the future, we will correlate the analysis of the materials composition with the trap spectroscopy measurements, in order to identify the traps and to ultimately control the compensation and trapping in CZT for radiation detector applications.

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List of References


2. For some radiation detector designs called unipolar designs, the transport properties of only one type of carriers are important. For CdZnTe, the preferred carrier is the electron, because of its larger mobility and longer lifetime.


7. The actual temperature range used was 20 K and 410 K. The maximum possible temperature of 475 K was not used, since In parts in the sample holder could melt at 440 K.

8. Counter-select grade crystal is defined by the supplier to be a radiation detector that, when detecting gamma-ray under optimized setting at 25 °C, gives, in a pulse height spectrum, (1) peak-to-valley ratio greater than 3 and FWHM less than 15 keV at 59.5 keV and (1) peak-to-valley ratio greater than 1.6 at 122 keV.


11. Presumably, the Au contacts were made using gold chloride solutions, but we are not able to confirm this with the supplier, because this is a proprietary information.


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Fig. 1. Conceptual view of TEES and TSC.

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Fig. 4. Photocurrent vs time under 10 V bias, at 20 K, and at the optimum wavelength of 760 nm for the sample.

Fig. 5. (a) TEES and TSC of sample A, (b) expanded TEES plot showing additional electron and hole traps, (c) expanded TEES and TSC plot near room temperature, showing a large ratio of the two currents, on the order of ~ 4000.

Fig. 6. (a) TEES of sample B, (b) TEES of sample C.

Fig. 7. Energy level diagram of traps in count-select-grade CZT.

Fig. 8. Electron and hole trap decay time vs trap thermal ionization energy at room temperature.
Fig. 1
variable temperature cryostat (14 K to 475 K)

He gas cylinder

Fig. 2.
Fig. 3.
Fig. 4.
Fig. 5.
Fig. 5
Fig. 6.
deep traps that affect radiation detection

traps that do not affect radiation detection

trap thermal ionization energy (meV)