Analysis of CZT Crystals and Detectors Grown in Russia and the Ukraine by High-Pressure Bridgman Methods

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ABSTRACT

Sandia National Laboratories (SNL) is leading an effort to evaluate vertical high pressure Bridgman (VHPB) $\text{Cd}_{1-x}\text{Zn}_x\text{Te}$ (CZT) crystals grown in the former Soviet Union (FSU) (Ukraine and Russia), in order to study the parameters limiting the crystal quality and the radiation detector performance. The stoichiometry of the CZT crystals, with $0.04 < x < 0.25$, has been determined by methods such as proton-induced X-ray emission (PIXE), X-ray diffraction (XRD), microprobe analysis and laser ablation ICP mass spectroscopy (LA-ICP/MS). Other methods such as triaxial double crystal x-ray diffraction (TADXRD), infrared transmission spectroscopy (IR), atomic force microscopy (AFM), thermoelectric emission spectroscopy (TEES) and laser induced transient charge technique (TCT) were also used to evaluate the material properties. We have measured the zinc distribution in a CZT ingot along the axial direction and also its homogeneity. The $(\text{Cd}+\text{Zn})/\text{Te}$ average ratio measured on the Ukraine crystals was 1.2, compare to the ratio of 0.9-1.06 on the Russian ingots. The IR transmission showed highly decorated grain boundaries with precipitates and hollow bubbles. Microprobe elemental analysis and LA-ICP/MS showed carbon precipitates in the CZT bulk and carbon deposits along grain boundaries. The higher concentration of impurities and the imperfect crystallinity lead to shorter electron and hole lifetimes in the range of 0.5-2 μs and 0.1 μs respectively, compared to 3-20 μs and 1-7 μs measured on US spectrometer grade CZT detectors. These results are consistent with the lower resistivity and worse crystalline perfection of these crystals, compared to US grown CZT. However, recently grown CZT from FSU exhibited better detector performance and good response to alpha particles.

1. INTRODUCTION

$\text{Cd}_{1-x}\text{Zn}_x\text{Te}$ crystals, with $0.05 < x < 0.4$, are leading materials for room-temperature operating radiation detector applications [1-5]. Due to the addition of Zn, CZT has a

larger energy gap than CdTe and therefore also has some distinct advantages over CdTe by having a higher value of bulk resistivity, lower dark current and reduced electronic noise. Crystal growth of CZT is performed by different methods, however for radiation detector applications, it is performed mainly by a vertical high-pressure Bridgman (VHPB) process. Sandia National Laboratories has been involved in many studies to characterize the various VHPB grown CZT crystals produced commercially in the US and in parallel has also evaluated CZT crystals grown in Russia [6-11] and in the Ukraine [6,10].

The present need for higher-grade CZT spectrometers, with better electrical properties, requires higher purity for the starting materials and higher crystalline perfection of the final product. Better understanding of the growth parameters is a must in order to improve the material quality. It is not clear yet how much the microstructural defects, such as precipitates and inclusions, affect the total performance of the CZT. Observation by SEM, AFM [12] and IR transmission shows many structural defects, carbon precipitates and highly decorated grain boundaries, which may deteriorate the crystal's electrical properties. These defects are much less common in spectrometer grade CZT detectors. It is suspected that microstructural defects, which can potentially deteriorate the electrical properties of the detector [8-11,13] are generated during the growth process. Alpha particle spectroscopy is one of the most common methods used to evaluate the electrical properties of wide band gap semiconductors, however, it appears that the highly imperfect regions of the detectors do not respond to alpha radiation and hole transport properties are difficult to quantify. In order to evaluate the electrical properties of those regions, a much more intense source of energy is required to produce detectable electron hole pairs. One possible source is a pulsed laser which produces many orders of magnitude more charge in each pulse. Some preliminary electrical results using the combination of focused pulsed laser and IR transmission micrographs will be described later in this paper. In parallel, we have focused on the evaluation of the elemental impurities in the bulk CZT as well as of the microstructures. The most common elemental analysis method used was the ICP/MS or ICP/OES, which involve a few steps prior to the analysis such as dissolution of the CZT sample in an acidic aqueous solution. Unfortunately, its sensitivity is limited by the dissolution rate and the residue of non-soluble substances. Additional impurities could be introduced from the solvent itself. To overcome this disadvantage, we used the laser ablation ICP/MS method which is more sensitive, especially for the minor impurities, and for the low Z elements such as carbon. Carbon was found to be one of the main impurities in Russian and Ukrainian CZT, which originates from the graphite-coated growth crucible. In an earlier publication [10], it was shown that carbon could be found as precipitates in the middle and also along grain boundaries. These impurities could, in turn, lower the resistivity and increase the leakage currents, thus deteriorating the electrical properties. Using microprobe elemental analysis, carbon was measured on numerous precipitates and grain boundaries (presented below) supporting this hypothesis.

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2. CRYSTAL GROWTH

Russian p-type CZT [10] was prepared using pre-synthesized ZnTe and CdTe mixed in the desired proportion of 5N purity Zn, Cd ("ELMA", Russia) and Te ("Pure metal plant", Ukraine). An excess of 1% tellurium was added to the ZnTe and CdTe mixtures, and the mixtures were synthesized at 980 C and 1100 C, respectively. The Ukrainian CZT was similarly grown and it also has a p-type character. The Russian crystals were then grown using the VHPB method [8,9] under 8 MPa argon pressure and at a crucible pulling rate of 2 mm/h, whereas the Ukrainian material used a similar VHPB method but with a pulling rate of 0.5-0.8mm/h. The Russian and Ukrainian n-type CZT [10] crystals were prepared in a similar way to the USA n-type grown material, by directly mixing the elements Cd, Zn and Te, presynthesizing CZT, and then growing the crystals in a similar way as the p-type CZT. Some of the results obtained on both n-type and p-type Russian CZT are published elsewhere [8,9].

3. MICROSTRUCTURE MEASUREMENTS

The infrared microscopy setup includes a Research Devices infrared microscope and 570 lines Sony B/W CCD camera. In addition we have a macro IR transmission setup which includes Apogee CCD camera with resolution of 1500x1000 pixels and 12 bit gray levels. The optics includes wide ranges of 35mm Nikon lenses and filters. Infrared microscopy has been used to identify small precipitates, inclusions, grain boundaries, voids and pipes, as well as photographing of large CZT wafers. The resulted micrographs then were used with other evaluation methods such as AFM, TCT, SEM, microprobe elemental analysis, XRD and charge transport mapping in order to correlate spatially between the detailed microstructural defects and other electrical and chemical properties. The infrared transmission micrographs shown in Fig.1 display a grain boundary in CZT.

Fig. 1. Transmission IR images of highly decorated Russian CZT. Left- round shape voids and precipitates on the grain boundaries. Right- 50mm diameter center slice of sample #61974, the left arrow points to a black region, which could be a carbon or tellurium layer located on a grain boundary. The top dark region is solid tellurium.

4. TCT MEASUREMENTS

Electrical transport properties, such as mobility μ , and trapping lifetimes τ , for electrons and holes have been obtained using pulsed laser transient charge technique (TCT). This method has been applied prior to this study to measure the electrical transport properties of mercuric iodide [13]. The system is equipped with a 3ns-pulsed 337nm nitrogen laser and a laser dye, LN₂ cold temperature stage, vacuum test chamber with an optical window on top, fiber optic cable and an oil-free vacuum pump. The pulsed laser made by LSI model VSL-337 with attached dye module was tuned to a wavelength of 525nm. The pulse width is 3 nsec with energy of 120 mJ per pulse; theoretically this is equal to 1.5×10^{14} electron hole pairs produced for each pulse. The dimensions of the test chamber are 20x20x12 cm with additional vacuum and LN₂ lines attached. The laser light easily penetrates through the gold electrode, therefore one does not need to remove the gold electrode. Using the IR transmission and reflection images, one can select any region of interest to probe its charge transport with a spatial definition of >0.5 mm. The setup also enables us to obtain transport properties on a bare surface, using a spring-loaded conductive and hollow probe which permits laser photons at a spot sizes of 0.8×0.8 mm². The output from the detector is very strong, therefore eliminating the need for additional amplification stages. This enables reading of the transients at low bias conditions, which is required for measuring the trapping times. Measuring the detrapping times is also possible at low temperature conditions [13]. Fig. 2 shows the set up used for TCT measurements and a typical waveform resulting from the electron transport process.

Trapping times have been measured on dark and clear regions of the Russian boule 61974. The material also exhibited high leakage currents, which in most cases prevented the production of spectrometers. This CZT has a resistivity of 10^9 to 10^{10} ohm-cm, which should be enough for some spectroscopic response; however, it poorly resolved the photopeaks associated with the gamma ray radiation. It also had large black regions as shown in Fig.1 and high concentration of precipitates and bubbles. Observing its grain boundaries, one can notice that the boundaries are also followed by cracks especially in the last to freeze region. This could indicate high concentration of impurities along the grain boundaries, which promote cracks during the cooling stage. The size of the black regions are in the range of $10 \times 10 \times 20$ mm, however the black regions are not confined to the boundary alone as has been shown in an earlier report [9,10], but are present throughout much of the whole volume. TCT measurements showed that the electrical transport properties of the boule are poor. However the black regions exhibited much poorer results as is shown in Table 1. The lifetimes measured on the transparent region of boule 61974 are in the same range as those measured in other crystals obtained from the FSU. These

results are consistent with the lower quality of these crystals, compared to US grown CZT. The higher leakage current measured could originate from the high level of impurities, especially carbon, which could create conductive pathways.

Fig. 2. Laser induced TCT setup (left) and typical TCT waveform obtained from high-grade spectrometer.

We have measured trapping lifetimes τ , on an identified grain boundary on a commercial 5x5x2 mm US grown CZT detector, however, the trapping time measured was approximately equal to the bulk value. This evidence could indicate that the deterioration of τ produced by some grain boundaries may be insignificant to the total transport properties. However, one should use a highly collimated single particle radiation source rather than a laser, in order to have more conclusive results. Other results show low $\mu\tau$ values along grain boundaries.

Table 1. Typical electrical properties measured on various CZT crystals.

Sample	electron-lifetimes τ_e	hole-lifetimes τ_h	Resistivity (ohm)
Russian 61974 black regions	<0.2 μs	not available	10 ⁸
Russian 61974 transparent regions	0.5-2 μs	~0.1 μs	10 ⁹ -10 ¹⁰
5x5x2 mm US grown CZT detector	15-20 μs	3 μs	10 ¹¹
Grain boundary on US grown CZT detector	6-7 μs	3 μs	10 ¹¹
Lifetimes τ , Range for detector grade CZT	3-20 μs	1-7 μs	10 ¹¹
Lifetimes τ , measured on detector prepared from the last to freeze part of the ingot	1-6 μs	0.5-3 μs	10 ¹⁰ -10 ¹¹

5. IMPURITY ANALYSIS

The preferred method to identify carbon precipitates is by microprobe elemental analysis. Fig. 3 shows carbon precipitates with their concentrations, however one should note that

these numbers depend on the coverage and the focusing of the electron beam and are apparently affected by the sampling area.

Fig.3. SEM micrographs and microprobe carbon analysis of sample 61974 (left) and KO-20 (right) show carbon rich precipitates.

The relatively new technique Laser ablation, LA-ICP/MS, was used for more comprehensive elemental analysis. The method uses a pulsed laser, which provides enough energy to directly ablate the CZT material from the solid sample to the mass spectrometer. Argon or helium gas is used to sweep the ablated particles into the ICP torch. By directly analyzing the solid, one can increase the sensitivity by a few orders of magnitude over the common solution analyzed ICP/MS method, thereby enhancing the statistics of the results. However, the drawback is that the results are very dependent on the nature of the ablated particles, which may react with other substances existing in the ablated media, the ionization efficiency, and the volatility rates, each of which is poorly known. The calibration and optimization of the process is still under study. Currently, spots which are ~30 micron apart and 5-30 micron in diameter are ablated, creating three sets of ten laser hits each, as is shown in Fig. 4. The penetration depth after each set of hits is 10, 20 and 30 microns respectively with about 60 seconds accumulation time between each set of hits. The LA-ICP/MS measurements were complimented by microprobe analysis of carbon impurities observed on SEM or IR micrographs.

Fig. 3 shows two micrographs of Russian and Ukrainian grown CZT. The black precipitates shown have been analyzed by microprobe elemental analysis for carbon impurity levels. The results exhibit high carbon content of up to 74% on the black precipitates. However, analysis of smaller precipitates showed lower amounts of carbon. This could be a result of the dimension of the microprobe analysis spot, which is very close to the dimension of the precipitate and hence may also read the CZT present in the matrix from outside of the precipitate boundaries. The precipitates shown are much different in their origin. For example in the Ukrainian sample (KO-20) the precipitates are placed on the grain boundary, while the Russian sample (#61974) has carbon precipitates dispersed in the bulk. Fig. 5 summarizes the laser ablation elemental analysis from the surface and one level deeper, penetrating 30µm below the crystal surface (see Fig. 4.). Fig.

5 shows the most common impurities present, divided into surface analysis and immediately below. The results exhibit little or no variation. The major impurities according to these results are Se, Cu, Al, S, Cl, Ni, Si and Fe.

Fig. 4. Micrograph of sample KO2-70 that shows the laser ablated grain boundary. The numbers matched the data given above.

Fig. 5. Typical surface (first 10 μm depth) and bulk (10-30 μm depth) elemental analysis (ppm) measured on KO2-70 by LA-ICP/MS.

6. TEES ANALYSIS

The thermoelectric emission spectroscopy (TEES) measurements have been performed inside a closed-cycle He cryostat manufactured by Janis Research Company, Inc., in

which the sample temperature can be varied between 14 K and 475 K. The cryostat is continuously pumped by a turbopump system made by Varian. The sample stage consists of a spring loaded sample holder and mirrors to direct the light from the cryostat window down to the sample.

The spring loading is necessary to keep the sample stationary and also to provide good thermal and electrical contacts to the sample through out the temperature range from 14 K to 475 K. The samples are placed flat on the sample holder using a spring loaded Teflon piece. The samples have In ohmic contacts. For photoexcitation at low temperature, the sample is illuminated from the side. External to the cryostat, a 250 W tungsten-halogen lamp is used as a light source. The light is focused to a McPherson monochromator that ranges from 200 to 1000 nm. The output of the monochromator is collected by a fiber optic that guides the light, which has a bandwidth of ~ 5 nm, into the cryostat. The measuring apparatus consists of Keithley 6517A electrometer to measure currents between ~ 1 mA and ~ 1 fA and a Lakeshore temperature controller, DRC-93CA, with a precision of ~ 0.1 K to control the cryostat temperature. All were interfaced by GPIB to a PC.

During cooling of the sample, it is biased at 0 V to avoid polarization effects. At ~ 20 K, the external tungsten-halogen lamp is turned on, and the photoconductivity of the sample is monitored. For the US CZT samples, the excitation wavelengths for maximum photoconductivity currents are found to be between 750 to 780 nm, and for the Russian 61974 CZT sample, the excitation wavelength is found to be ~ 710 nm. Under identical illumination intensity, the photocurrent for the Russian sample is orders of magnitude smaller than the US sample, indicative either of much greater ionized impurity scattering, different barriers at the In contacts, or very short recombination life times. Detailed report of this phenomenon will be given elsewhere. After 30 minutes of photoexcitation, the tungsten-halogen light is turned off. The sample, which is kept in complete darkness, is gradually heated to room temperature.

In TSC, the sample is gradually heated and the voltage bias is left on. Any carriers emitted from the traps in the sample are driven by the applied electric field and measured as the TSC current. However, in TEES, a temperature gradient of a few degrees is applied across the sample. In the absence of an applied electric field, this temperature gradient causes the carriers emitted from the traps to travel from the hot region to the cold region of the sample. Since electrons and holes have opposite charge polarities, their motion in the same direction gives opposite signs of the currents. This means that, unlike TSC, TEES can be used to distinguish between electron and hole traps by noting the sign of the TEES current at a given temperature.

In the TEES spectrum shown in Fig. 6, one can see that the Russian 61974 CZT sample has two electron trap peaks at 188 K and 240 K and two hole trap peaks at 45 K and 361 K. Their corresponding energy trapping levels are given in Table 2. The positive TEES current is due to emission of electrons from electron traps in the sample and a negative TEES current is due to emission of holes from hole traps in the sample.

Fig. 6. TEES spectrum of Russian grown CZT detector.

Table 2. Analysis of the TEES Spectrum and the Transient Charge Technique (TCT) of the Russian CZT Sample #61974.

Temp. for Hole trap.	Hole Trapping level	Hole Trapping time τ_h from TCT	Temp. for Electron trap.	Electron Trapping level	Electron Trapping time τ_e from TCT
45 K	0.09 ± 0.03 eV*	$< 0.1 \mu s$	188 K	0.44 ± 0.11 eV***	$2 \mu s$
361 K	0.96 ± 0.21 eV**		240 K	0.58 ± 0.13 eV	

Remarks

*Same as a shallow level observed in US material.

**This trap is probably the major cause of the short hole life time.

***Same as a deep level observed in US material.

Compared to previous results obtained from detector grade CZT [12], the observed hole trap signal is much stronger and is deeper than a similar peak found in the US material. It should be noted that the deep hole trap is near mid gap and hence it is likely to function as a strong recombination center. Also the 188 K and 240 K electron traps are stronger than the electron peaks observed in the US grown CZT. One can assume that electrical properties of CZT would be much worse due to the additional hole traps, in agreement with experiment.

7. ATOMIC FORCE MICROSCOPY

In AFM, the probe tip is attached to a cantilever with a small spring constant. There are three main interaction methods of the AFM operation: contact mode, tapping mode and non-contact mode. Contact mode is the most common method of operation of the AFM. The tip and sample remain in close contact as the scanning proceeds. By "contact" we mean in the repulsive regime of the inter-molecular force curve. The repulsive region of the curve lies above the horizontal plane. One of the drawbacks of remaining in contact with the sample is that there exist large lateral forces on the sample as the tip is "dragged" over the specimen. Tapping mode is the next most common mode used in AFM. When operated in air or other gases, the cantilever is oscillated at its resonant frequency (often hundreds of kilohertz) and positioned above the surface so that it only taps the surface for a very small fraction of its oscillation period. There is still contact with the sample, but the very short time over which this contact occurs means that lateral forces are dramatically reduced as the tip scans over the surface. When imaging soft samples, tapping mode may be a far better choice than contact mode. Using this method one can also obtain better image contrast. In constant force mode, the feedback loop adjusts so that the amplitude of the cantilever oscillation remains (nearly) constant. An image can be formed from this amplitude signal, as there will be small variations in this oscillation amplitude due to the control electronics not responding instantaneously to changes on the specimen surface.

CZT images were obtained on a Digital Instruments Dimension 3100 Scanning Probe Microscope using Tapping mode. The preparation of the CZT sample for AFM includes lapping and fine polishing of the CZT surface up to tens of nm flatness, followed by chemical etching. For this purpose we used 5% bromine in methanol etching solution. The sample was dipped in the 5% bromine etchant for 20 sec followed by rinsing with methanol. This procedure was repeated for three times. The two samples tested are US grown CZT and Russian grown CZT (see Fig. 7). One should note that both samples were subjected to the same polishing and etching procedures, however the microstructural defects (mainly the large and fragile grain boundaries) observed in the Russian CZT made the required high precision polishing difficult as shown in Fig. 7. One can also see that the selectivity of the etchant solution depends on the grain orientation, as we can see from the AFM micrograph shown in Fig. 7.

Fig. 7. AFM of polished and etched U.S. grown (left) and Russian grown (right) CZT, exhibiting a twin boundary and a deep grain boundary, respectively.

7. CONCLUSIONS

The analysis presented here shows that crystals originating from the former Soviet Union have more microstructural defects, namely carbon rich precipitates, grains that are opaque to IR, inclusions, bubbles and crystalline defects. These defects greatly deteriorate the electrical transport properties of the material. However, even in relatively clear regions,

the electrical properties were only moderately better, which may be due to the high level of carbon and other impurities dispersed in the CZT. We assume that the carbon is one of the major contributors to the measured high leakage current [6-10]. Laser ablation ICP/MS is a valuable analytical technique, however, more work needs to be done to improve its quantification capabilities. Preliminary results confirm the existence of carbon in FSU grown CZT crystals and the existence of other impurities such as Ca, Si, Fe, Cu, S, Cl, Ba, Ni, Al, and Se. It is noteworthy that the laser ablation analysis also supports earlier results that the segregation distribution of most impurities is less than 1, causing segregation to the last-to-freeze portion of the ingot. The existence of tellurium precipitates on the last-to-freeze portion was also observed for the Te rich growth runs. Except for carbon, which is much larger in the FSU crystals, the other impurities seem to be on the same order of magnitude as the US grown material. TEES and TCT measurements showed more deep electron and hole traps in the FSU crystals.

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