

Elimination of Te Inclusions in $\text{Cd}_{1-x}\text{Zn}_x\text{Te}$ Crystals by Short-term Thermal Annealing

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Abstract—The presence of Te inclusions degrades the quality of today's CdZnTe (CZT) crystals used for X- and gamma-ray detectors; both their sizes and concentrations densities must be reduced. Over the past years, many researchers proposed using long-term annealing (>24 h) under Cd vapor pressure to reduce or even eliminate the inclusions visible under IR microscopes. We annealed detector-grade CZT samples for periods of 15 to 60 min under Cd-, Zn-, or Te-overpressure or in vacuum at 1000–1200 K. We determined the optimal temperature, duration, and the vapor atmosphere for such high-temperature annealing, typically at ~ 1100 K for 0.5–1.0 h. The results were very promising in eliminating Te-rich inclusions, even on twins where the inclusions are more stable than in the unperturbed lattice; indeed, we saw almost no inclusions whatsoever by IR transmission microscopy after such annealing. We note that eliminating inclusions at lower temperatures takes much longer. However, annealing under a Cd vapor pressure at temperatures above ~ 1170 K generates a large quantity of irregular Cd inclusions. The samples' resistance after annealing was estimated by I-V curves.

Index Terms—Annealing, $\text{Cd}_{0.9}\text{Zn}_{0.1}\text{Te}$, component overpressure, crystals, inclusions.

I. INTRODUCTION

ALTHOUGH $\text{Cd}_{1-x}\text{Zn}_x\text{Te}$ (CZT) crystals are the leading choice for room-temperature gamma-ray detectors, their quality must be improved by reducing the size and density of Te inclusions before they can reach their full market potential [1]–[3]. Over the past years, researchers proposed long-term annealing (>24 h) under Cd vapor pressure to reduce or completely eliminate the inclusions visible with IR microscopy. As

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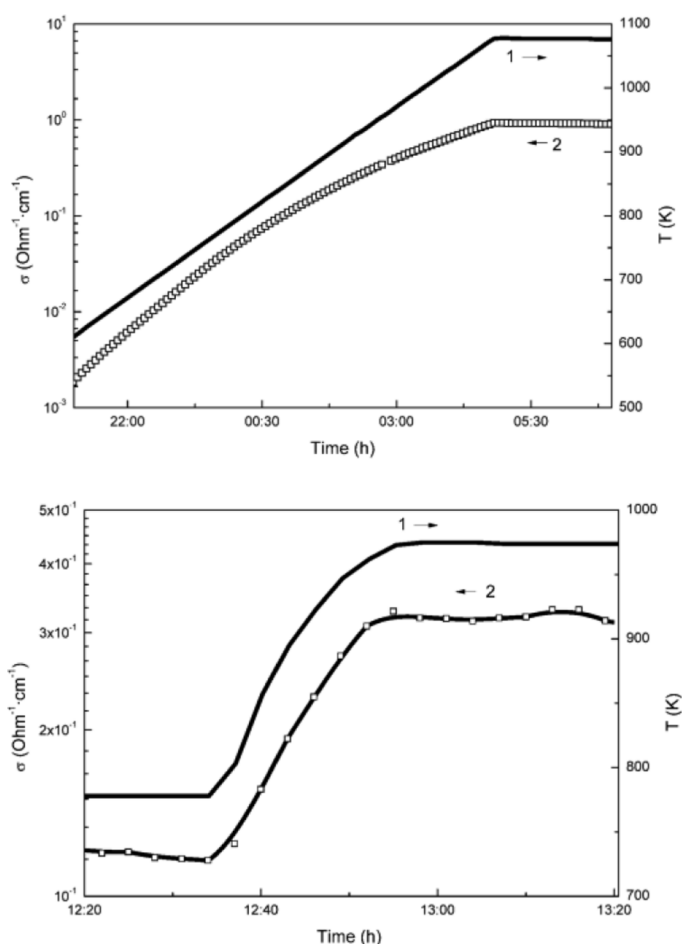


Fig. 1. Electrical conductivity (σ) changes in as-grown CZT samples during heating under Cd (upper graph) and Te (bottom graph) saturation. 1—CZT sample's temperature, 2—conductivity.

early as 1992, Vydyanath *et al.* subjected CZT substrates to a lengthy annealing (~ 100 h at 770 K) under Cd overpressure to reduce the numbers of Te inclusions and improve their quality [4], [5]. Sen *et al.* reported similar results from annealing material under Cd- and $\text{Cd}_{1-x}\text{Zn}_x$ ($x = 0.005$) alloy vapor pressure at 770–970 K for up to seven days [6]. The possibility to growth inclusion-free CdZnTe by proper setting of the growth parameters was studied in [7].

The effects on CdTe and on $\text{Cd}_{0.96}\text{Zn}_{0.04}\text{Te}$ of annealing in cadmium- or tellurium-vapor were explored by Shen *et al.* using different characterization techniques [8]. They found that

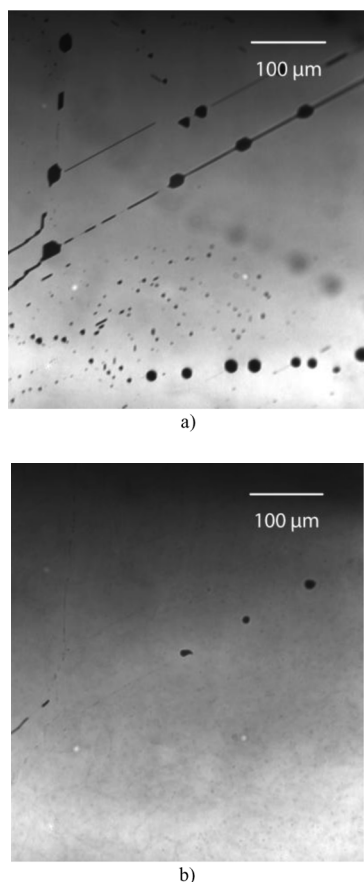


Fig. 2. (a) Infra-red transmission microscopy (IRM) images of CZT sample before annealing. (b) Infra-red transmission microscopy (IRM) image of CZT sample after annealing under a Cd overpressure ($T_{\text{CZT}} = 1100$ K, $T_{\text{Cd}} = 1060$ K, duration—60 min, and quenching).

0.5–5.0- μm tellurium-rich inclusions disappeared after first annealing at 970 K for ~ 20 h and subsequently cooling in tellurium vapor at a rate of 33 K/h until the temperature fell to 770 K, at which point they closed the vapor flow. However, inclusions of 5- and 15- μm size remained. After annealing in cadmium vapor, the concentration of the larger inclusions and precipitates declined.

Later, Li *et al.* demonstrated an improvement of the quality of CZT crystals after thermal treatment using a $\text{Cd}_{1-y}\text{Zn}_y$ alloy as the annealing source [9]. As guidance, they used their own calculations of the correlations of the partial pressures over the CZT crystal and $\text{Cd}_{1-y}\text{Zn}_y$ alloy to optimize the conditions for gas-solid equilibrium CZT annealing. They lowered the density of defects by more than one order-of-magnitude, while bettering the structural- and electrical- properties of the CZT crystals. Further, these authors proposed an annealing method with pure Cd and Zn as separate vapor sources [10], having established that the CZT crystal's homogeneity was highly improved and its structural-, optical-, and electrical-properties ameliorated by such annealing. They showed that 1073 K was the preferable annealing temperature for CZT crystals.

Belas *et al.* studied annealing conditions that would lower both Te and Cd inclusions [11], [12], finding that 24-h Cd-rich annealing above 930 K with subsequent cooling (1–2 K/min) effectively eliminated most Te inclusions. However, in some

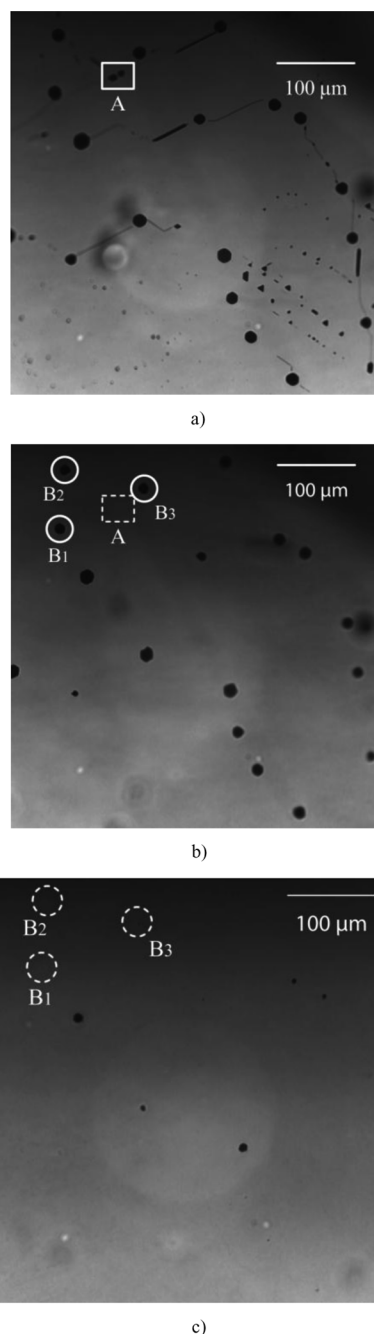


Fig. 3. IRM images of a CZT sample before (a) and after 1st (b) and 2nd annealing and (c) under a Cd vapor overpressure during 15 min at 1100 K ($T_{\text{Cd}} = 1060$ K and quenching in air).

cases, only the diameter of inclusions was reduced; small residual spots survived at the same position in the matrix, even after long-term Cd-rich annealing at 1070 K, or after 24-h Cd-rich annealing at 1270 K. The investigators interpreted these spots either as being a reduced part of the inclusions, located in their original positions in the matrix, or as voids whose walls probably were decorated by the Cd-rich CdTe.

Zhang recorded the effect of the annealing on two kinds of CZT wafers grown by the modified vertical Bridgman method, one under a Te-rich condition and the other under a stoichiometric condition [13]. The micron-sized Te inclusions were eliminated in CZT wafers annealed at 1120 K for 200 h in

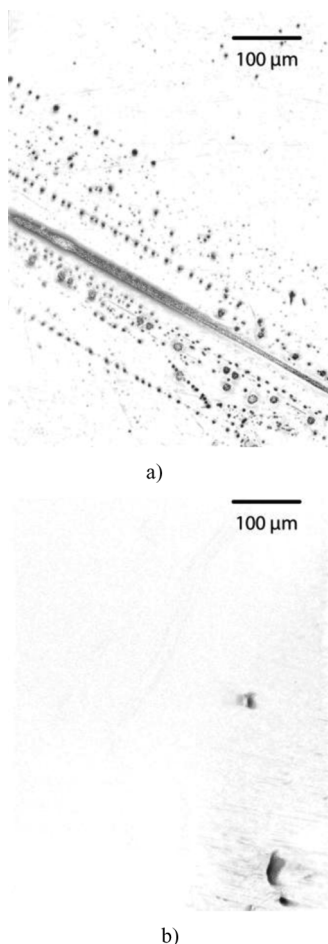


Fig. 4. Volumetric superposition of 20 IRM images of the CZT sample from the front to rear surface before (a) and after (b) sample annealing under a Cd vapor overpressure for 30 min at 1100 K ($T_{Cd} = 1080$ K and quenching in air).

saturated Cd vapor; further, the density of large inclusions was reduced by more than tenfold. However, annealing did not significantly influence the full-width at half-maximum (FWHM) value of the rocking curve, nor the etched pit density (EPD) of CZT wafers. In contrast, CZT annealed at 1020 K for 240 h still possessed a high density of inclusions, equivalent to the level as before annealing; nevertheless, there was a reduction in the size of the inclusions.

During our present measurement of the high-temperature Hall effect, we established that after annealing the CZT samples reached electrical equilibrium in less than ~ 20 –30 min at 970–1070 K. We surmised (as was shown for annealing at ~ 770 K), that a brief thermal treatment at 700–1070 K influences, nearly simultaneously, the crystal's electrical parameters and the dissolution/elimination of inclusions. The second reason for choosing a short annealing time was to observe, systematically, the process of reducing the numbers of inclusions, so helping our understanding of the mechanism of their dissolution, and our identifying the main technological parameters determining the improvement of the crystals' structure. The use of these parameters in the crystal's growth process promises to increase the yield of inclusion-free detector-grade material.

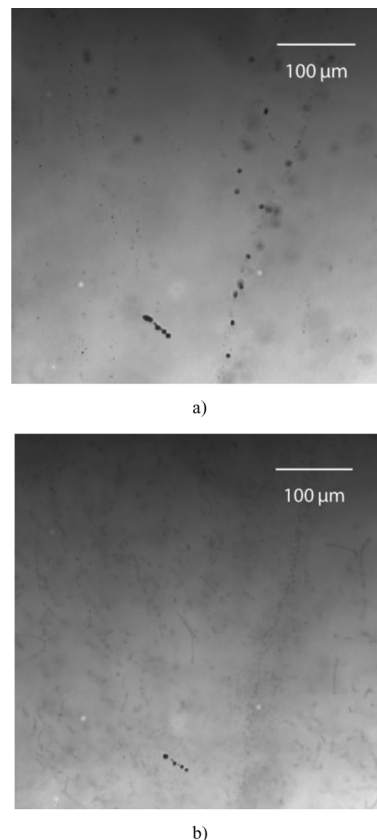


Fig. 5. IRM images of CZT sample before (a) and after (b) annealing under a Cd vapor overpressure for 20 min at 1100 K ($T_{Cd} = 1077$ K and quenching in air).

The goal of this work was to determine the effects of the precise timing and temperature, the stoichiometric deviation, and the cooling rate, both on the size and density of inclusions of CZT crystals after short-time annealing. The changes in samples' resistivity were also studied for all our experiments, and additional details regarding the resistivity changes will be reported elsewhere.

II. EXPERIMENTAL

Detector-grade CZT commercial crystals were grown by Endicott Interconnects using the Bridgman method. Approximately 60 samples ($\sim 7 \times 6 \times 3$ mm³) were polished and etched in a 2% Br-methanol solution before annealing. We obtained IR images and I-V curves before and after the high-temperature treatment. The samples were sealed in quartz ampoules and annealed under Cd-, Zn-, Cd-Zn-alloy or Te vapor overpressure, and in vacuum at 770–1200 K. The cooling rates were different, ranging from quenching in air to rates of 1 K/min. Annealing lasted 5–60 min at higher temperatures, and it was longer at lower temperatures. To compare the same areas before and after annealing, we marked the samples by scratching their surfaces. In addition, for some samples the translation station determined the precise position of the scratched cross-sections relative to one of the sample's corners. We evaluated the sizes and distributions of inclusions inside the samples before and after annealing.

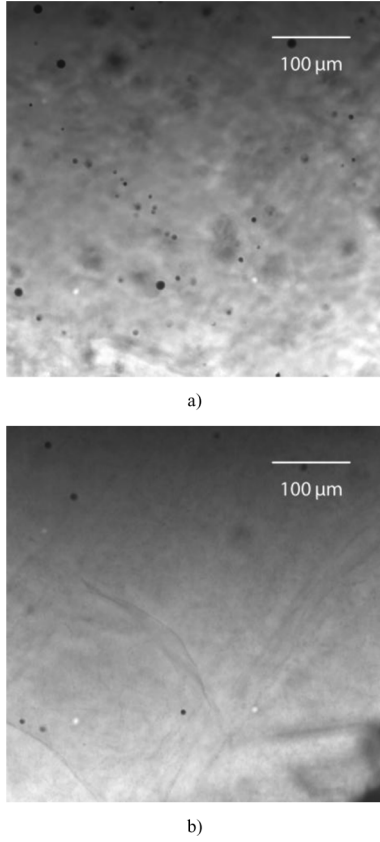


Fig. 6. IRM images of CZT sample before (a) and after (b) annealing under a Te vapor overpressure during 20 min at 1100 K ($T_{\text{Te}} = 1000$ K and quenching in air).

III. RESULTS AND DISCUSSION

Our high-temperature Hall-effect measurements of CZT samples under Cd- or Te-saturation showed that point defects reached equilibrium at 970–1070 K within ~ 30 min (Fig. 1), after the sample's electrical properties (mobility and charge carrier density) had long stabilized. The structural changes (inclusion transformation) occurred somewhat slower. Therefore, for our first experiments we selected a 60-min-treatment duration and a temperature of 1100 K. The samples contained many inclusions of different sizes and shapes. More specifically, the size was up to ~ 30 microns, and the measured density was $\sim 1.5 \times 10^5 \text{ cm}^{-3}$.

All the small inclusions (up to 10 microns) and almost all the large ones (10–30 microns) disappeared after annealing (Fig. 2) demonstrating the effectiveness of such treatment in improving of CZT crystal structure.

To follow the process of transformation of the inclusions with time, we shortened the duration of annealing to 15 min and acquired images of the material. As Fig. 3(a) shows, many of the small inclusions and lines were eliminated within 15 min. A second 15-min annealing removed almost all other inclusions [Fig. 3(b)]. We found that the 2 biggest inclusions in Fig. 3(a), with the diameter ~ 14 and $12 \mu\text{m}$, marked as A by a square, disappeared [Fig. 3(b)] within 15 min of annealing at 1100 K at a Cd overpressure. In Fig. 3(b), the

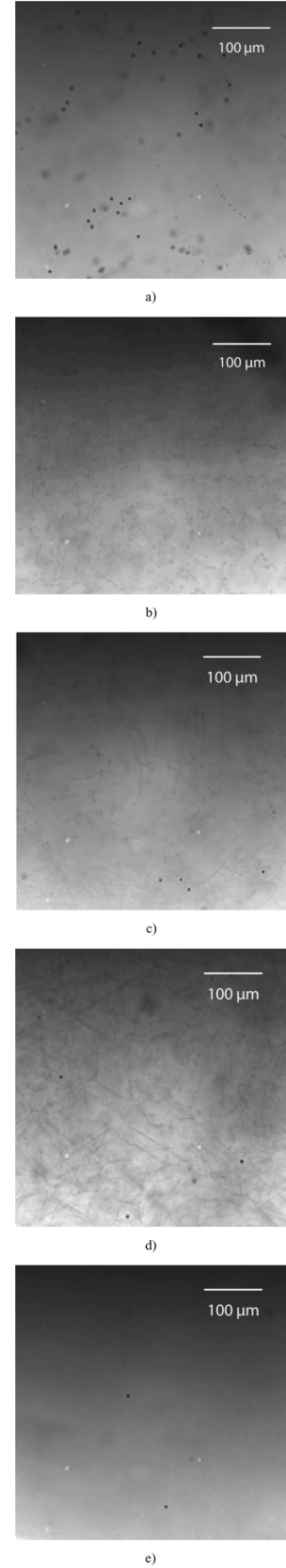


Fig. 7. a–e. IRM images of CZT sample before annealing (a) and annealed under a Zn- (b), Cd- (c), and Te- (d) vapor pressure and in vacuum (e) for 60 min at 1100 K (quenching in air). (a) before annealing. (b) Zn overpressure. (c) Cd overpressure. (d) Te overpressure. (e) vacuum.

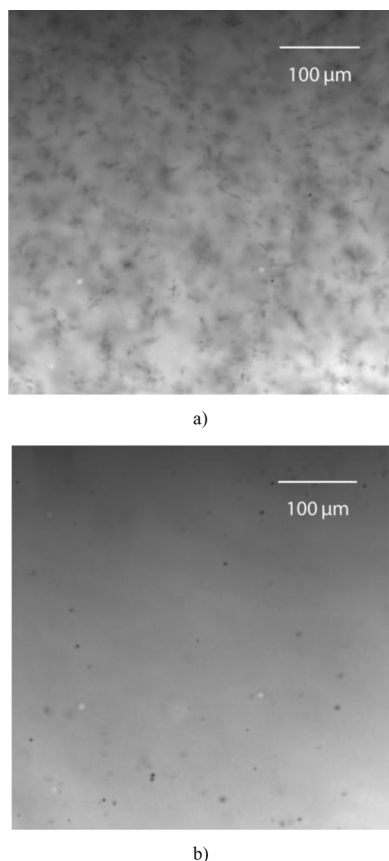


Fig. 8. IRM images of CZT sample annealed under a Cd vapor overpressure for 30 min at ~ 1200 K ($T_{Cd} = 1170$ K) with quenching (a) and slow cooling rate of the sample in the furnace (b).

three encircled inclusions with a diameter $\sim 18 \mu\text{m}$, viz., B1, B2, and B3, disappeared after 15 min of second annealing [Fig. 3(c)]. IRM scanning from the front to the rear surface of the sample (20 IR images) confirmed that the inclusions had been removed throughout the entire volume under inspection (Fig. 4). Some imperfections on the right image in Fig. 4 are scratches and pits, not inclusions.

The 20-min annealing under Cd saturation (and also under Te overpressure) almost eliminated inclusions, too (Figs. 5 and 6). They disappeared even from twins, which sometimes was not achievable [11]. The treatment in vacuum under the same conditions (30 min at 1100 K) gave similar results: almost all inclusions were gone. This demonstrates that Cd diffusion inside the samples cannot be the only reason for the disappearance of inclusions. Therefore, we carried out the next annealing under four different stoichiometry conditions: Cd-, Zn-, Te-vapor overpressure or under vacuum [Fig. 7(b)–(d)]. Fig. 7(a) illustrates the typical initial distribution of inclusions in these samples.

In almost all images of the quenched samples, there is a network of short- and long-lines and irregular shaped stars. Only after the treatment under vacuum [Fig. 7(d)] was the image free of them. We surmised that the solubility of Zn, Cd, and Te in CdTe at 1100 K is high, and during quenching, excess atoms precipitate on the dislocations. During annealing under vacuum equilibrium, the concentration of Cd- and Te-atoms is

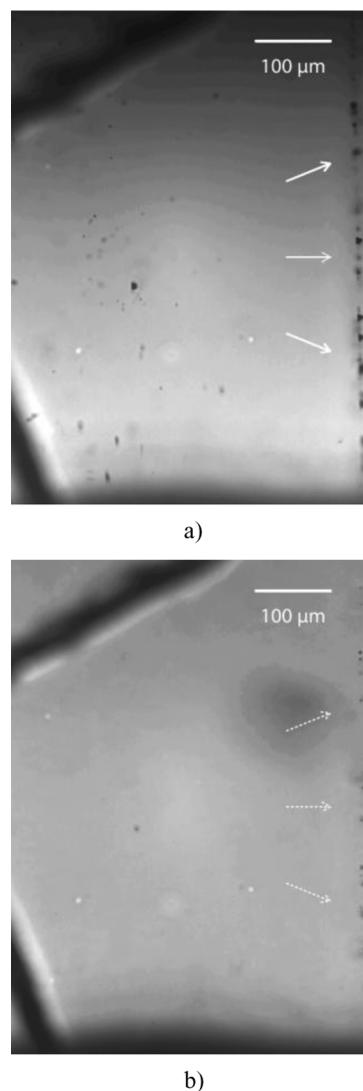
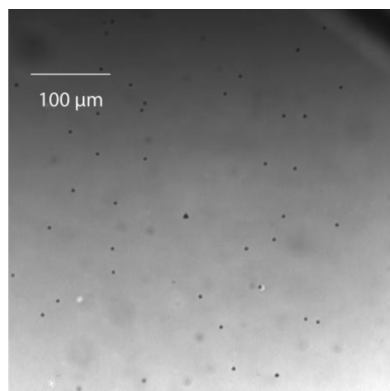


Fig. 9. IR images of CZT sample before (a) and after (b) annealing under a Cd vapor overpressure for 60 h at 900 K. The three arrows show inclusions on the twin.

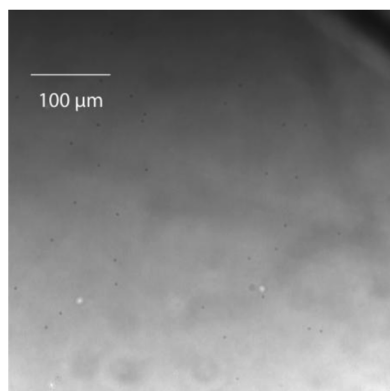
low, and no significant precipitation occurs. To reduce precipitation during saturation annealing, the rate of cooling must be slow.

To check this assumption we annealed two CZT samples in similar conditions (1200 K) under Cd saturation for 30 min. Then, one sample was quenched and another slowly cooled. The quenched sample exhibited features typical for all such ones, with different parts containing lines and unshaped figures [Fig. 8(a)]. In the slowly cooled sample, the IR image did not contain these figures, signifying that our supposition of excess atoms precipitating on the dislocations is reasonable [Fig. 8(b)].

Also many experiments were performed at lower temperatures to define the time necessary for inclusion removal. The main trend was obvious: if the lower temperature was used the longer annealing was needed. However in some experiments the obtained data demonstrated more complicated nature of CZT sample defect structure.

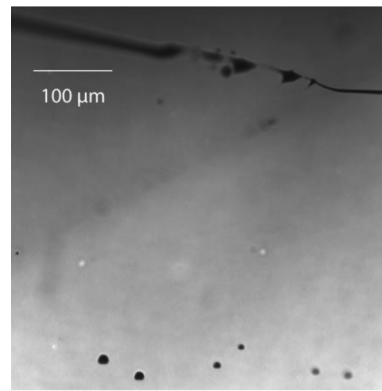


a)

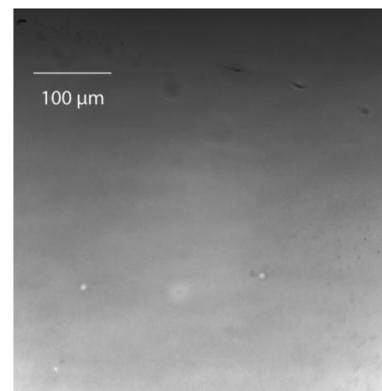


b)

Fig. 10. IRM images of CZT sample before (a) and after (b) annealing under a Cd vapor overpressure for 60 h at 965 K.



a)



b)

Fig. 11. IRM images of CZT sample before (a) and after (b) annealing under a Cd vapor overpressure for 3 h at 1010 K.

The annealing at lower temperature (900 K) over 60 h resulted in the elimination of almost all inclusions (Fig. 9). Only some of them, located in twins had not disappeared, although they had decreased in size.

However the annealing during the same time (60 h) at 965 K ($T_{\text{Cd}} = 896$ K) did not result in full inclusion elimination; they only substantially decreased in size (Fig. 10). Similar situation was observed at the annealing at 1010 K. In many cases the inclusions disappeared, even if they formed lengthy inclusions (Fig. 11). In one case only small inclusions were eliminated during the same annealing, and most of larger ones decreased their size a little, but did not disappear (Fig. 12).

Even at the annealing at 1100 K during 1 h, some large size and line inclusions disappeared but the very similar ones left without or with small changes (Figs. 13 and 14).

As illustrated in Fig. 15, annealing at 1100 K under Zn vapor overpressure for 60 min produced a network of lines on the sample's surface that were removable by polishing. It is evident that during the annealing, Zn saturated the sample's surface resulting in the formation of a CZT solid solution with high Zn content (rather very close to the ZnTe composition) at the surface. The lattice parameters of $\text{Cd}_{0.9}\text{Zn}_{0.1}\text{Te}$ and ZnTe crystals are significantly different, causing strains that deform the surface. The similar situation was also observed by Jones in [17]. After such annealing, one can observe a thin crystalline layer of

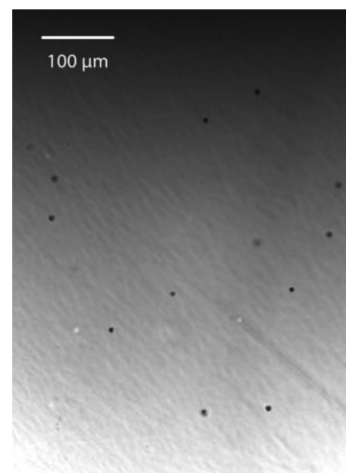
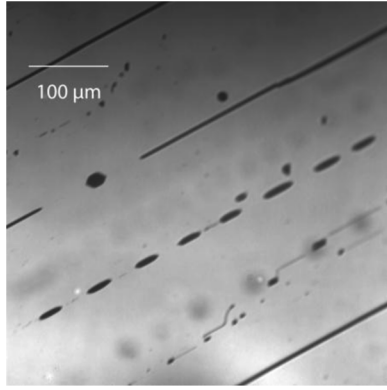


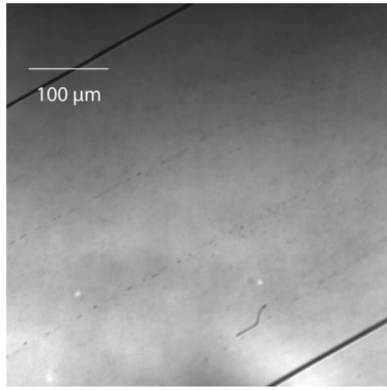
Fig. 12. IRM image of CZT sample after annealing under a Cd vapor overpressure for 3 h at 1010 K. Before the annealing the inclusion distribution was uniform.

rose color (probably ZnTe) on the sample's surface and on the ampoule walls near the sample's location. It was formed owing to the interaction between the Zn and Te vapor. The Te was generated by CZT dissociation at the high temperature.

For all annealed samples, their resistance was defined using I-V curve measurements (Table I). Mainly it was very low



a)

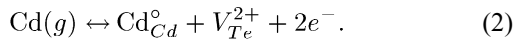
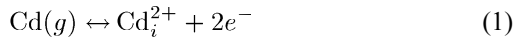


b)

Fig. 13. IRM images of CZT sample before (a) and after (b) annealing under a Cd vapor overpressure for 1 h at 1100 K.

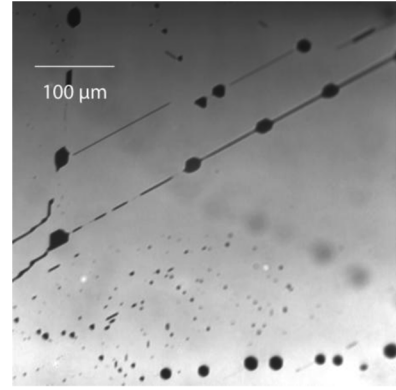
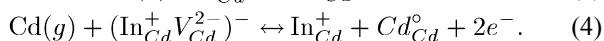
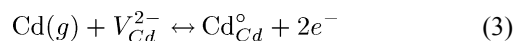
($10^3 - 10^4$ Ohm). However in some cases it was 2–3 orders of magnitude higher.

During the annealing under Cd saturated vapor pressure, Cd atoms are introduced into the lattice as doubly charged native donors (Cd_i^{2+} and V_{Te}^{2+}) as follows [14]:

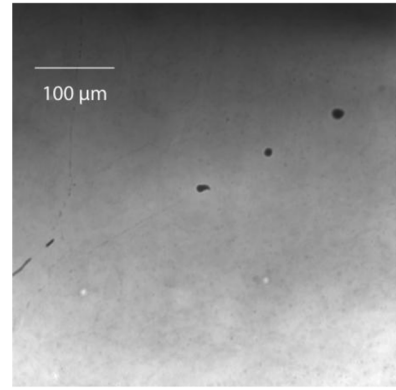


Therefore the samples become low-resistive and of n-type conductivity (for example, experiments #1, 5, 8, and 15). Of course, the annealing time and cooling rate also influence the resistivity value.

When the Cd overpressure is much lower than the saturated one (for example, experiments #9, 16, and 21), the quantity of native donors produced is less than in the previous case (1)–(2), and the concentration of native acceptors (V_{Cd}^{2-}) is higher (3). A portion of the Cd vacancies also form A-centres with foreign donors (4). Therefore the electron density is not as high, and the sample's resistivity increases:



a)



b)

Fig. 14. IRM images of CZT sample before (a) and after (b) annealing under a Cd vapor overpressure for 1 h at 1100 K.

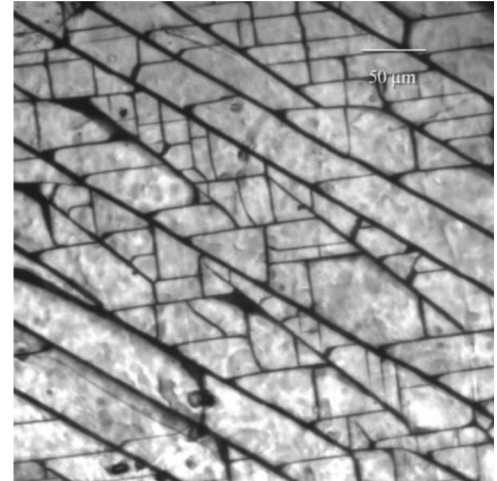


Fig. 15. Surface layer of a CZT sample illuminated by IR light after annealing under a Zn vapor overpressure at 1100 K for 60 min.

The annealing under Te overpressure results in the formation of a large quantity of intrinsic acceptors (V_{Cd}^{2-} and Te_i^-) (see, for example, experiments #4, 7, 13, 18, and 20):

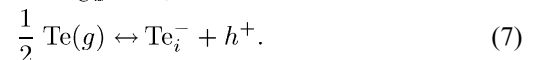
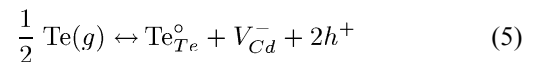


TABLE I
CZT SAMPLES' ANNEALING CONDITIONS AND THEIR RESISTANCE AFTER
THIS TREATMENT

Samples' number	Component	T_{CZT} , °C	$T_{\text{comp.}}$, °C	Time, hours	Cooling type	R, Ohm
1.	Cd	823	776	1	Q	$2 \cdot 10^4$
2.	Zn	823	772	1	Q	$2 \cdot 10^6$
3.	Vac	823	>815	1	Q	$\sim 10^3$
4.	Te	812	765	1	Q	$3 \cdot 10^3$
5.	Cd	~790	~750	0.4	Q	10^3
6.	Zn	823	~790	0.33	S	$2 \cdot 10^6$
7.	Te	823	723	0.33	Q	10^4
8.	Cd	823	~800	0.17	Q	10^3
9.	Cd	623	~400	1	Q	$4 \cdot 10^8$
10.	Zn	600	450	1	Q	$3 \cdot 10^4$
11.	Zn	716	665	10	S	$5 \cdot 10^4$
12.	Cd	603	535	24	S	$2 \cdot 10^3$
13.	Te	733	680	3	Q	$4 \cdot 10^3$
14.	Cd	833	790	1.7	S	$9 \cdot 10^5$
15.	Cd	743	712	5	Q	$4 \cdot 10^3$
16.	Cd	600	445	12	S	$4 \cdot 10^5$
17.	Cd	740	627	12	S	$4 \cdot 10^3$
18.	Te	700	630	8	Q	$4 \cdot 10^3$
19.	Zn	600	515	12	S	$2 \cdot 10^4$
20.	Te	725	648	12	S	$8 \cdot 10^3$
21.	Cd	815	700	5	Q	$7 \cdot 10^6$
22.	Zn	825	685	1	Q	$3 \cdot 10^6$

S- slow cooling rate (1-2 K/min), Q – quenching in the air

As shown by the high-temperature measurements under Te overpressure, such treatment creates a lot of holes that are easily observed in the material [15].

The treatment under Zn overpressure gives higher resistivity for the samples, in most cases, compared to annealing under Cd or Te saturation (for example, see experiments #2, 6, and 22). The reason is not clear yet. We can only surmise that the higher Zn vacancy content in CZT crystals associates more readily with the In_{Cd}^+ (higher vacancy concentration in ZnTe crystals compared to CdTe does not allow the production of n-type material under Zn overpressure, even at 1200 K [16]).

We should mention that our research at this phase of experimentation focused on elimination of Te inclusions in CZT crystals only.

IV. CONCLUSIONS

Treating high-temperature detector-grade ~ 3 -mm thick CdZnTe samples under different stoichiometry conditions, we demonstrated the possibility of drastically diminishing the Te inclusions' size and density during short-time annealing (60–150 min) at $T \sim 1100$ K under Cd-, Zn-, or Te-vapor overpressure or in vacuum. Below 900 K, the necessary annealing time exceeds 40–60 h. We conjectured that the dissolution of the inclusions occurs either via the dislocation net and/or by interaction with both native- and foreign-point defects. Annealing under single Zn vapor overpressure engenders a “cracked” surface possibly due to the mismatch between lattice

parameters of the surface layer, more or less enriched by Zn, and the sample's bulk volume.

However, in some cases it is not possible to eliminate all inclusions even during long-term and high-temperature treatments. We conclude that annealing is a complicated procedure that depends not only on the temperature, time, and type of overpressure, but also on the structure of the crystal, the nature of its point defects, the dislocation network, and the dopant content.

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