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Growth of cadmium–zinc telluride crystals by controlled seeding "contactless" physical vapor transport

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Growth of cadmium–zinc telluride crystals by controlled seeding "contactless" physical vapor transport

W. Palosz [a,*,1], K. Grasza [b,2], D. Gillies [a], G. Jerman [a]

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Abstract

Bulk crystals of cadmium–zinc telluride, 23 mm in diameter and up to 45 grams in weight were grown. Controlled seed formation procedure was used to limit the number of grains in the crystal. Most uniform distribution of ZnTe in the crystals was obtained using excess (Cd + Zn) pressure in the ampoule.

1. Introduction

Cadmium–zinc telluride crystals can be used as substrates for HgTe-based infrared detectors and as room-temperature γ-ray detectors. By appropriate selection of ZnTe content in the crystals, the lattice constants of the substrate and of the active epilayer can be matched giving improved crystallographic perfection and related performance of the devices [1]. Nuclear detectors fabricated from cadmium–zinc telluride show improved, relative to CdTe, energy resolution [2].

Crystals of (Cd,Zn)Te can be grown by physical vapor transport (PVT) at relatively low temperatures and in the absence of a second condensed phase. Under such conditions, material with improved crystallographic perfection and purity relative to those in melt grown crystals may be obtained. In this work we investigate the growth of (Cd,Zn)Te crystals by a self-nucleation "contactless" PVT technique using a special seed selection procedure. This method has proved successful in growing large, high quality crystals of the related material, CdTe [3,4]. However, growth of a ternary crystal by PVT poses distinctive thermochemical and kinetic complications as compared to related binary systems [5,6]. The current work has been undertaken in order to evaluate the capabilities and limitations of the "contactless" techniques for growing ternary, cadmium–zinc telluride bulk crystals.

2. Experimental procedures

Cadmium–zinc telluride ($Cd_{1-x}Zn_xTe$) source materials were synthesized as described in our earlier

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Table 1
Crystal growth experimental parameters

Initial composition of the source, X_0	0.04, 0.21
Range of T(source):	
without excess (Cd + Zn)	880°C–930°C
with excess (Cd + Zn)	990–1050°C
Excess component pressure (at T(source))	0.03–0.7 atm H_2 or 0.1–0.2 atm (Cd + Zn) + 0.04 atm H_2
Duration of seed selection stage	2–5 days
Average crystal growth rate	1–2.5 mm per day
Post-growth cool-down	T(source) → 100°C in 3 h

work [6]. 50–60 g of pre-synthesized source was thermally pre-treated [6], then sealed and pre-compacted at about 850°C under hydrogen atmosphere to a relative density of about 70%. The compacted material was loaded (with pre-determined amount of elemental cadmium when desired) into a growth ampoule of the type used earlier [3]. The ampoule was assembled, evacuated, and back-filled with the desired amount of hydrogen and sealed. Crystal growth experimental parameters are listed in Table 1.

Composition profiles were obtained using electron microprobe analysis techniques. The spatial resolution of the measurements was 40 micrometers. The combined composition (X) uncertainty (resulting from the beam current instabilities, local surface non-planarity, and misalignment of the sample and standards relative to the beam direction) was estimated at about 3% or less.

3. Experimental results

3.1. Growth without excess constituent element

Fig. 1 illustrates consecutive stages of the growth process [3,4]. The initial source (Fig. 1a) undergoes a local recrystallization in the (non-uniform) thermal field, what leads to formation of a cone with a crystal grain (seed) at its tip (Fig. 1b). Further development of the cone and seed leads to a contact and attachment of the seed crystal to the pedestal (Fig. 1c). After a proper change of the thermal field (by displacing the ampoule [3]) the seed separates from the source (Fig. 1d) and the crystal growth proceeds (Fig. 1e). Photographs of the source and of the seed developed on top of the source (stage (b) in Fig. 1) are shown in Fig. 2a and 2b, respectively. The seed is bound primarily by {110} and {111} facets. Composition profiles across the seed are shown in Fig. 3a

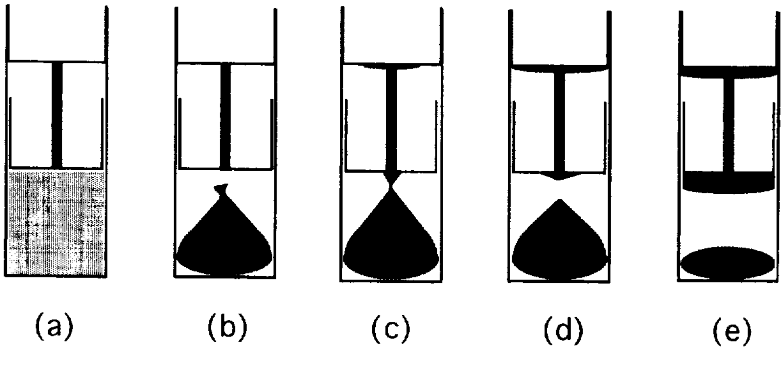

Fig. 1. Schematic representation of the consecutive stages of the nuclei development and crystal growth.

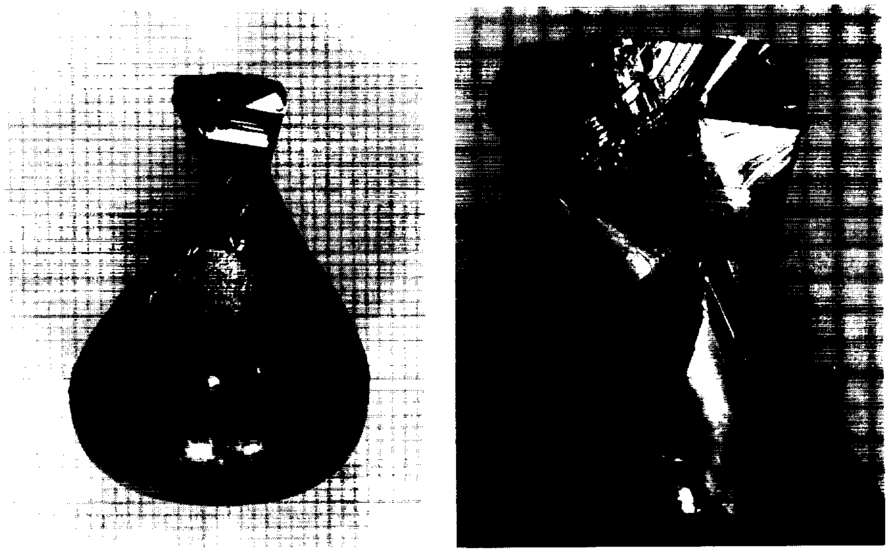

Fig. 2. The source (a) and the crystal (b) formed on the source tip. $X_0 = 0.04$, no excess constituent element.

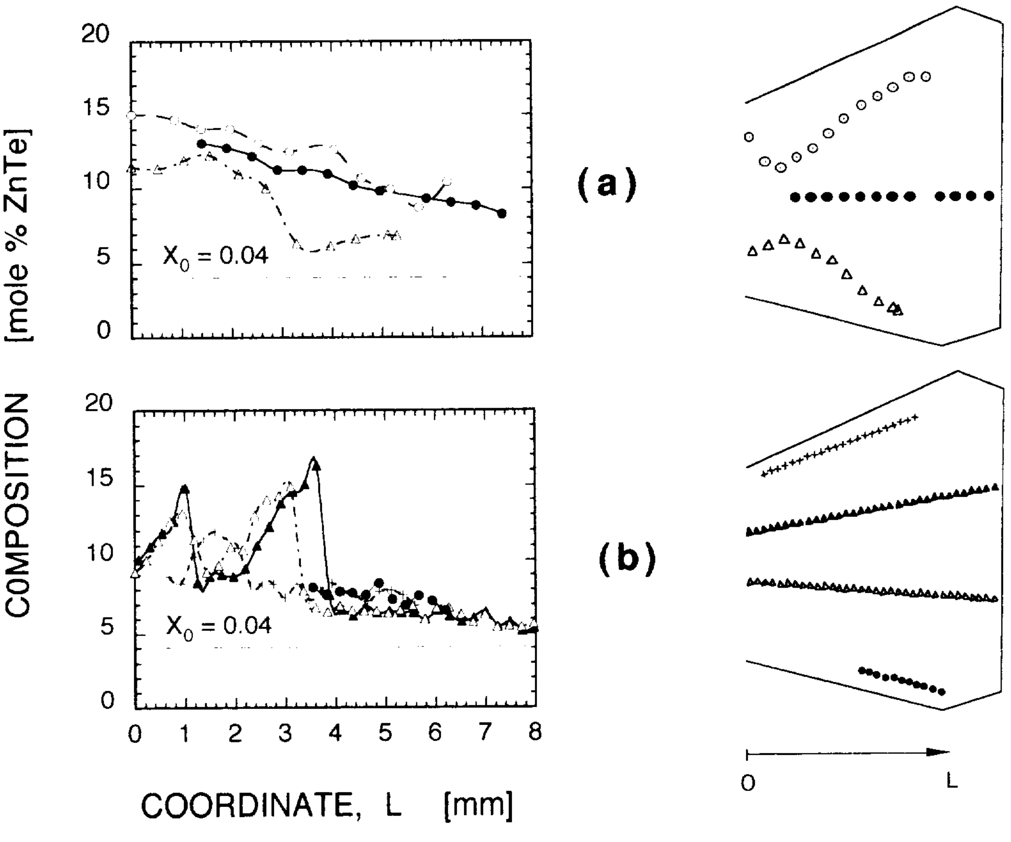

Fig. 3. Composition profiles in the seed crystal shown in Fig. 2: (a) on the surface; (b) in the bulk mid-plane. Scanning paths are shown on the right. L is the position along the path.

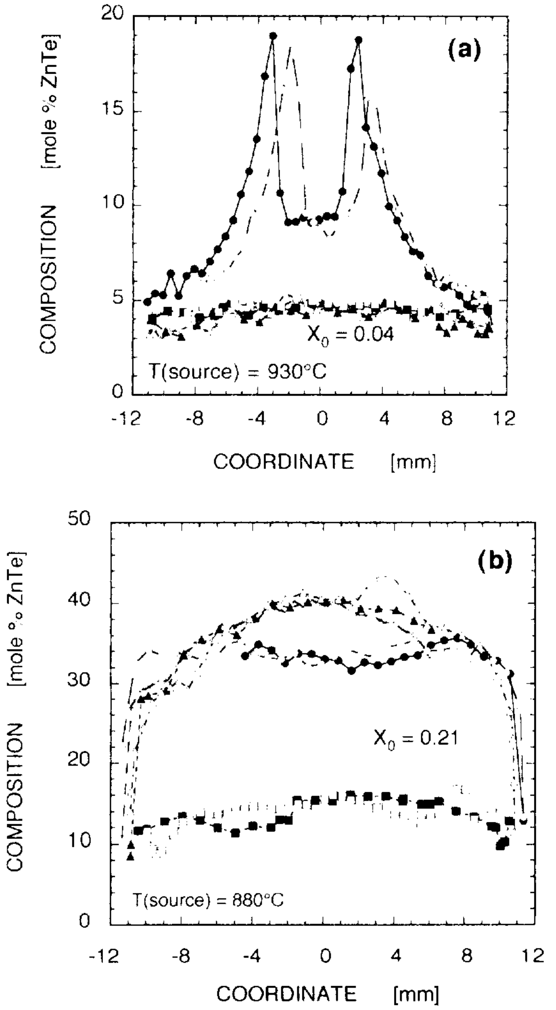

Fig. 4. Lateral composition profiles of the crystals grown without intentional addition of constituent element(s) to the ampoule: z is the distance from the pedestal, full symbols and open symbols depict scans along x and y, respectively. (a) $X_0 = 0.04$: (●,○) $z = 0.3$ mm; (▲,△) $z = 6$ mm; (■,□) $z = 12$ mm. (b) $X_0 = 0.21$: (●,○) $z = 0.3$ mm; (×) $z = 1.5$ mm; (▲,△) $z = 3.5$ mm; (■,□) $z = 12$ mm.

and 3b on the surface and in the bulk of the seed, respectively. The composition of the seed is highly non-uniform and up to four times higher than the

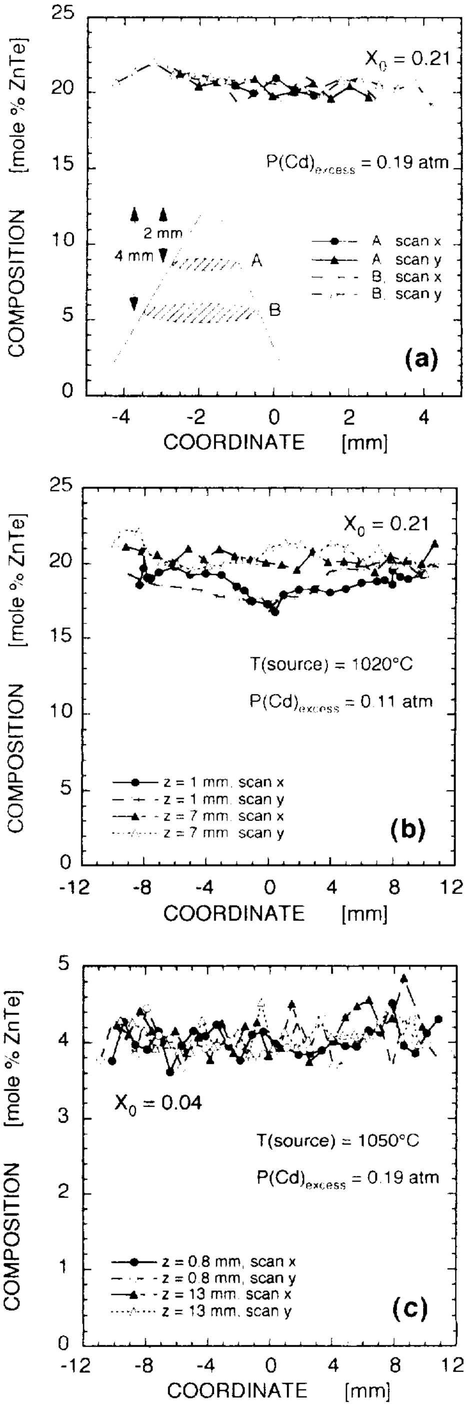

Fig. 5. Lateral compositions of the source tip (seed) and crystals grown under excess (Cd + Zn) pressure. (a) The source, $X_0 = 0.21$ (the inset shows the investigated cross sections of the source); (b) crystal, $X_0 = 0.21$; (c) crystal, $X_0 = 0.04$.

average composition of the source $X_0 = 0.04$. The observed non-uniformity of the ZnTe distribution after the source compacting/recrystallization is consistent with the results of our earlier work [5,6]. Fig. 4a shows lateral composition profiles of a crystal grown from $X_0 = 0.04$ source without intentional excess constituent in the vapor phase. The composition profiles of the first grown part of the crystal ($z = 0.3$ mm, where z is the distance from the pedestal) are highly non-uniform and reflect the compositional non-uniformity of the seed: the composition in the middle of the cross section corresponds to the location where the seed attached itself to the pedestal (stage (c) in Fig. 1). Subsequent, laterally grown parts of the crystal enriched in ZnTe (the peaks in Fig. 4a) are apparently due to high concentrations of zinc on top of the source [6]. The composition becomes relatively uniform in further parts of the crystal (Fig. 4a, $z = 6$ and 12 mm). Fig. 4b shows lateral composition profiles of a crystal grown from $X_0 = 0.21$ source. The compositional non-uniformities in the initially grown part of the crystal (Fig. 4b, $z = 0.3$ mm) are relatively less pronounced then corresponding profiles for $X_0 = 0.04$ (Fig. 4a). However, substantial lateral non-uniformities are observed in different parts of the crystal. In addition, axial composition non-uniformity of the crystal is very high, the composition varies (Fig. 4b) from 200% to 50% of the initial composition of the source ($X_0 = 0.21$).

3.2. Growth under excess (Cd + Zn) pressure

Vaporization of cadmium–zinc telluride is essentially a non-congruent process: mole fraction of the more volatile component (Cd) is higher in the vapor than in the solid. Preferential vaporization of cadmium relative to that of zinc leads to a higher concentration of CdTe on the crystal–vapor relative to that at the source–vapor interface. As a consequence, progressive accumulation of the less volatile component (ZnTe) at the source–vapor interface leads to subsequent composition gradients in the source and in the growing crystal [6]. This process can be suppressed by an appropriate adjustment of the composition of the vapor: local thermodynamic equilibria combined with mass continuity requirements under steady state conditions lead to such redistribution of the vapor species that congruent transfer of the material becomes possible [5,6]. As expected, growth under excess metal pressure conditions leads to improved homogeneity of our cadmium–zinc telluride crystals. Fig. 5a shows lateral composition profiles of two cross sections of the source top after compacting/recrystallization (stage (b), Fig. 1) of $X_0 = 21$ material under excess metal pressure conditions. The composition of the cross sections is quite uniform and close to the initial composition of the source X_0. Lateral composition profiles of crystals grown under excess (Cd + Zn) pressure are shown in Figs. 5b and 5c. Both axial and lateral distributions of ZnTe in the crystals are fairly uniform and close to X_0. This is a considerable improvement relative to crystals grown without intentional non-stoichiometry of the vapor (Figs. 4a and 4b).

3.3. Crystals

A representative crystal grown in this work is shown in Fig. 6a. In general, the (Cd,Zn)Te crystals are similar in appearance to CdTe crystals grown by

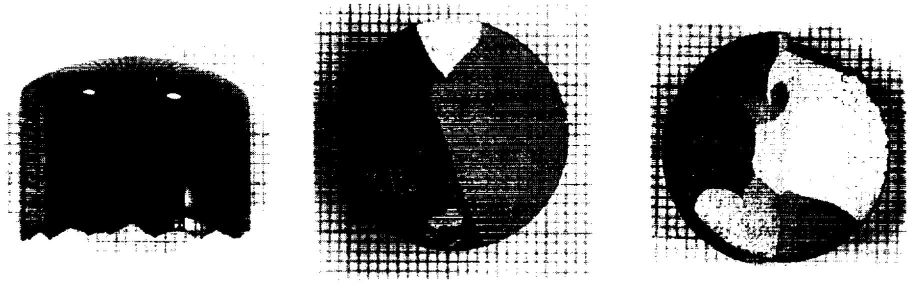

Fig. 6. Crystals. (a) Overall view. (b), (c) Cross sections of a crystal of $X \approx 0.04$ (no excess pressure) and $X \approx 0.2$ (excess P(Cd + Zn)), respectively.

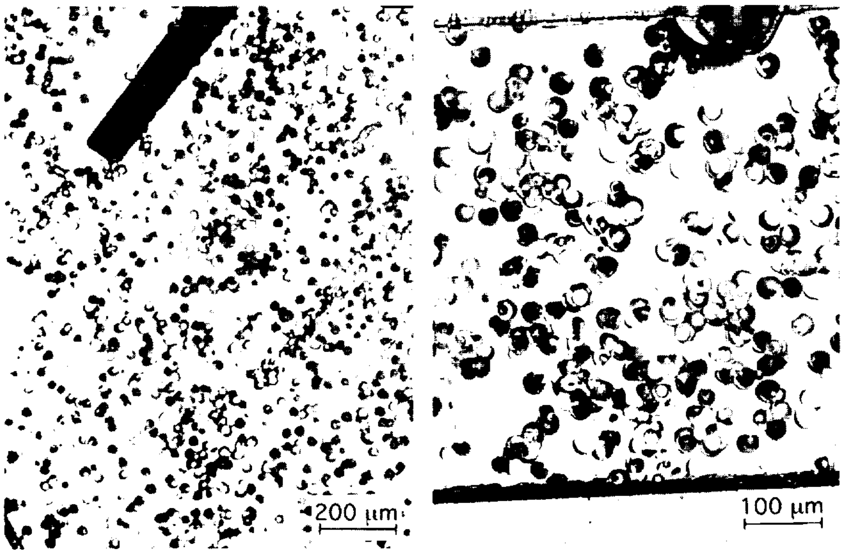

Fig. 7. Photomicrographs of etched (Nakagawa etch [7], Cd($\bar{1}\bar{1}\bar{1}$) surface) crystals of $X = 0.04$ grown (a) without, and (b) with excess (Cd + Zn) pressure.

the "contactless" technique [3]. The nuclei selection and control is, however, more difficult and more grains may form during growth of cadmium–zinc telluride crystals. Large single-crystalline grains can, however, be obtained (Figs. 6b and 6c). EPD values are about 10^5 cm^{-2} (Fig. 7a and 7b). No apparent difference in EPD values was found between crystals grown with and without excess (Cd + Zn). Crystals grown with excess metal were relatively brittle and had a tendency to break into a few pieces during cutting by a wire saw. Similar tendency to cracking was found in CdTe crystals grown under excess Cd pressure [3]. Cracking of (Cd,Zn)Te crystals observed by Ruault et al. [8] has been interpreted as a result of a phase separation/second phase formation at lower temperatures [9]. Laue back-reflection diffraction patterns of our crystals do not, however, show any traces of a second phase in the material (Fig. 8a and 8b).

4. Conclusions

The technique of "contactless" PVT with nucleus pre-formation can be used to grow bulk $Cd_{1-X}Zn_XTe$ crystals both with lower ($X \approx 0.04$) and higher ($X \approx 0.2$) zinc telluride concentrations. With a careful control of the process, large single-crystalline grain(s) can be obtained. Our experience with other ternary systems indicates that further im-

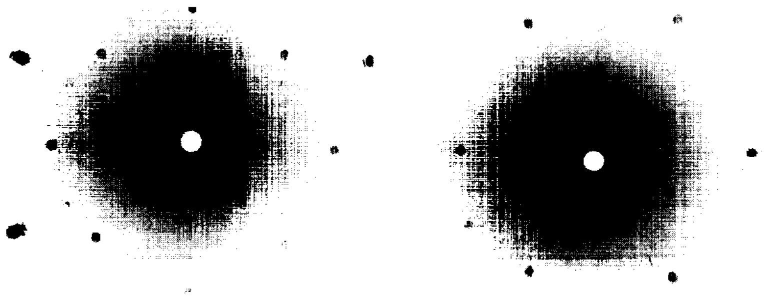

Fig. 8. Laue back-reflection diffraction patterns. (a) $X = 0.04$, (001) surface; (b) $X = 0.2$, (111) surface.

provement (larger grains and even single crystals) is possible with a more elaborate seed-selection process and better adjustment of the growth parameters and procedures. The best compositional homogeneity can be obtained for crystals grown under excess (Cd + Zn) pressure in the ampoule. The associated increased brittleness of the crystals could, possibly, be reduced by growth under modified thermophysical conditions (if caused by growth instabilities) or by a post-growth annealing under different (Te-rich?) atmosphere (if caused by excess metal in the lattice). Without the intentional addition of a constituent element a reasonably uniform distribution of zinc telluride in a part of the crystal can also be obtained for low ZnTe content in the source. Preliminary crystallographic characterization of the crystals shows a moderate etch pit density and no apparent presence of a second phase. More extensive crystallographic characterization of the crystals using synchrotron radiation will be presented in a separate publication [10].

Acknowledgements

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