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JOURNAL OF **CRYSTAL GROWTH**

Journal of Crystal Growth 174 (1997) 733-739

Seeded physical vapor transport of cadmium-zinc telluride crystals: growth and characterization

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Seeded physical vapor transport of cadmium–zinc telluride crystals: growth and characterization

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Abstract

Crystals of $\text{Cd}_{1-x}\text{Zn}_x\text{Te}$ with $x = 0.2$ and 40 g in weight were grown on monocrystalline cadmium–zinc telluride seeds by closed-ampoule physical vapor transport with or without excess (Cd + Zn) in the vapor phase. Two post-growth cool-down rates were used. The crystals were characterized using low temperature photoluminescence, atomic force microscopy, chemical etching, X-ray diffraction and electrical measurements. No formation of a second, ZnTe-rich phase was observed.

PACS: 81.05.Dz; 61.72.Qq; 78.60.Ya

Keywords: Physics vapor transport; Cadmium–zinc telluride

1. Introduction

Cadmium–zinc telluride is a ternary material with applications in IR and γ -detectors. $\text{Cd}_{1-x}\text{Zn}_x\text{Te}$ substrates allow for improved perfection and performance of IR detectors relative to CdTe-based devices [1]. Addition of ZnTe improves the energy resolution of CdTe-based γ -detectors [2]. Cadmium–zinc telluride crystals are

usually grown from the melt [3–5]. Improved growth conditions (lower growth temperature, good stoichiometry, reduced impurity level) and related improvement in the crystal quality may be achieved by physical vapor transport (PVT) techniques. Recently we conducted theoretical and experimental studies on the transport of (Cd, Zn)Te and “contactless” crystal growth by PVT [6–8]. In this paper we present our results on (Cd, Zn)Te bulk crystals grown by seeded PVT. The crystals were characterized by chemical etching, photoluminescence, atomic force microscopy, X-ray diffraction, and electrical measurements.

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2. Crystal growth

Crystal growth experiments were performed in fused silica ampoules of inner diameter 15–24 mm. The starting material was pre-synthesized and pre-treated as described in Ref. [7]. The source material was compacted and loaded into the growth ampoule with a seed crystal and excess Cd (when desired). After evacuation and back-filling with hydrogen, the ampoule was sealed. Other ampoule and growth parameters are shown in Table 1. During growth the furnace was translated relative to the (stationary) ampoule at a rate approximating that of the crystal growth. After growth the ampoule was cooled down either by turning the power off or by a slow cooling program (procedures A and B, respectively). In the first case (A) the temperature of the crystal dropped from about 1000 to 200°C in 6.5 h. During the programmed procedure (B), cooling down to 800°C occurred at a rate of 10°C/h, and was followed by a subsequent cooling at

Table 1
Experimental parameters

Source material	Pre-synthesized and sintered (Cd, Zn)Te powder [7]
Initial composition	0.04; 0.21
Amount of source	20–50 g
Excess component	0.05 atm H ₂ or 0.1–0.2 atm Cd (at T_{source})
T_{source}	1000°C
Temperature gradient at the crystal vapor interface	20–40°C/cm
Average crystal growth rate	1–4 mm/day

20°C/h. A typical ampoule, crystals after growth, and representative radial composition profiles of the crystals (obtained with a microprobe) are shown in Fig. 1a–Fig. 1d, respectively. Large monocrystalline grains can be obtained (Fig. 1c). The radial compositions are reasonably uniform across most of the boule (Fig. 1d). However, in agreement with our previous measurements,

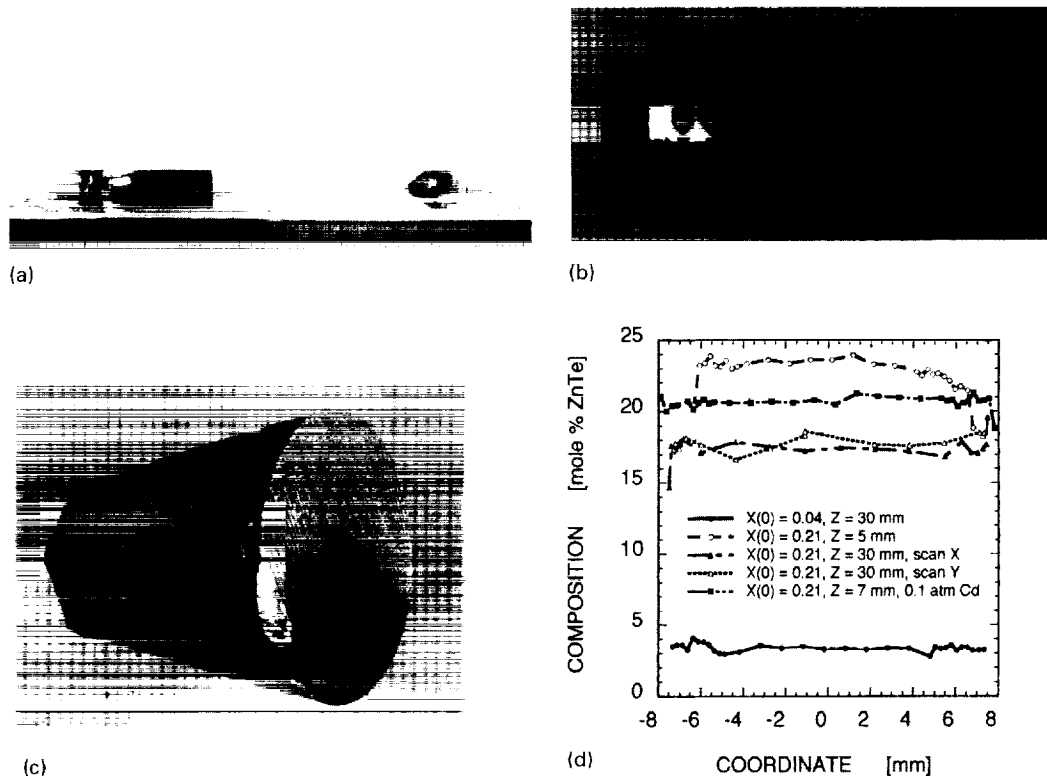


Fig. 1. (a) Ampoule after growth; (b), (c) crystals; (d) radial composition profiles. Z is the distance from the seed crystal.

crystals grown without excess Cd showed axial compositional nonuniformities [6–8].

3. Characterization

3.1. Etching

Selected samples of the grown crystals were chemo-mechanically polished with 2% Br in ethy-

lene glycol and etched using Inoue [9] and/or Nakagawa [10] etch. It was found that etch pits in zinc-cadmium telluride are more difficult to obtain than in pure CdTe, particularly for higher ZnTe-content samples. The crystals have EPD values usually about 10^5 cm^{-2} or less (Fig. 2), in some cases up to about $5 \times 10^5 \text{ cm}^{-2}$. Subgrains were observed in crystals of low ZnTe content cooled slowly (procedure B) after growth (Fig. 2b). No

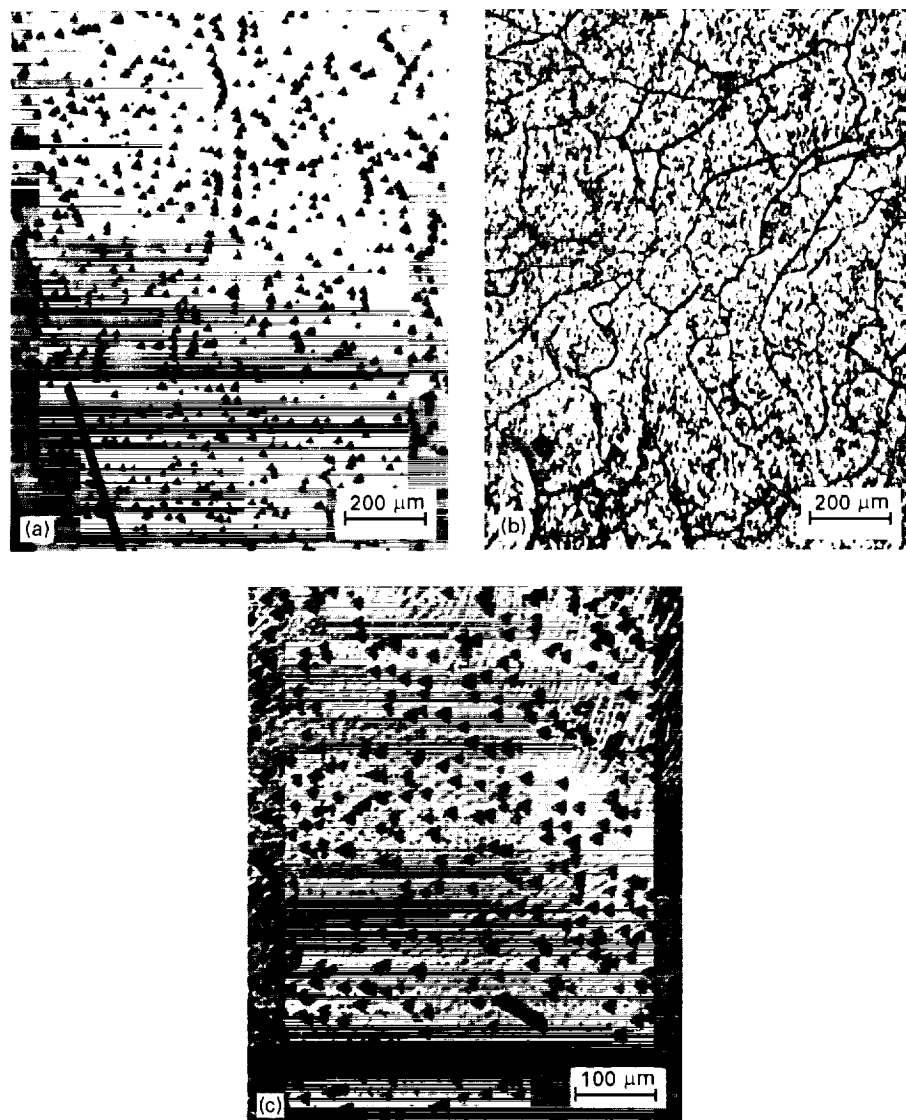


Fig. 2. Etch pits. (a) $x = 0.04$, excess (Cd + Zn), cool-down A; (b) $x = 0.04$, no excess (Cd + Zn), cool-down B; (c) $x = 0.14$, no excess (Cd + Zn), cool-down A.

specific dependence of the etch pit density and pattern on crystal composition and excess (Cd + Zn) in the vapor was found.

3.2. Photoluminescence (PL)

Low temperature PL measurements were performed on crystal samples which were cooled down to 10.6 K by APD Cryogenic Inc. system equipped with dual HC-4 MK1 helium compressors. The 514.5 nm line, with a power density of 15 mW/mm² from an ILT 5500A air-cooled argon ion laser was selected for excitation. The PL spectra were recorded using a Spex 1877D Triplemate Spectrophotometer and a liquid nitrogen cooled CCD detector. In the spectrograph stage of the spectrometer, a 30 μm slit and a 300 grooves/mm grating were employed. A spectrum typical for our (Cd, Zn)Te samples is shown in Fig. 3a. Considering the origin of the emission lines, the PL spectra can be divided into three regions (from right to left, Fig. 3a): (i) the bound exciton recombinations (BE) [11, 12]; (ii) donor–acceptor pair (DAP) transitions [13]; and (iii) emission associated with crystal defects (DEF) [13, 14]. The exciton nature of the edge peak (BE) of the crystals was confirmed by a super-linear dependence of the PL amplitude of the edge peak on excitation intensity. While in binary materials these are usually sharp peaks (no kinetic energy broadening) in ternary crystals some disorder-in-

duced broadening occurs. The position of the exciton peak was used to determine the composition of the Cd_{1-x}Zn_xTe samples using the empirical formula [13]

$$\epsilon(\text{BE}) = 1.589 + (0.65 \pm 0.01)x + (0.19 \pm 0.02)x^2. \quad (1)$$

The peak at about 0.05 eV below the bound exciton position can be attributed to the donor–acceptor pair transitions, the presence of which is primarily due to extrinsic defects in the lattice [13]. The third, broad emission region lies about 0.17 eV below the bound exciton line. Since we used undoped and high purity materials, the peak is apparently associated with cadmium vacancy related defects [13, 14].

The composition of the crystals determined from the position of the bound-exciton peak is in a good agreement with independent measurements made using energy dispersive X-ray spectroscopy and wavelength dispersive X-ray spectroscopy techniques, and with our previous results. Good compositional uniformity of the crystals was obtained with excess Cd in the vapor or a reverse temperature gradient in the source region [6–8]. PL spectra of several samples from each crystal were taken. In most cases, the DAP peak was small, the peak intensity ratio $J_{\text{BE}}/J_{\text{DAP}}$ was over 100. The DEF peaks were usually higher, the ratio $J_{\text{BE}}/J_{\text{DEF}}$

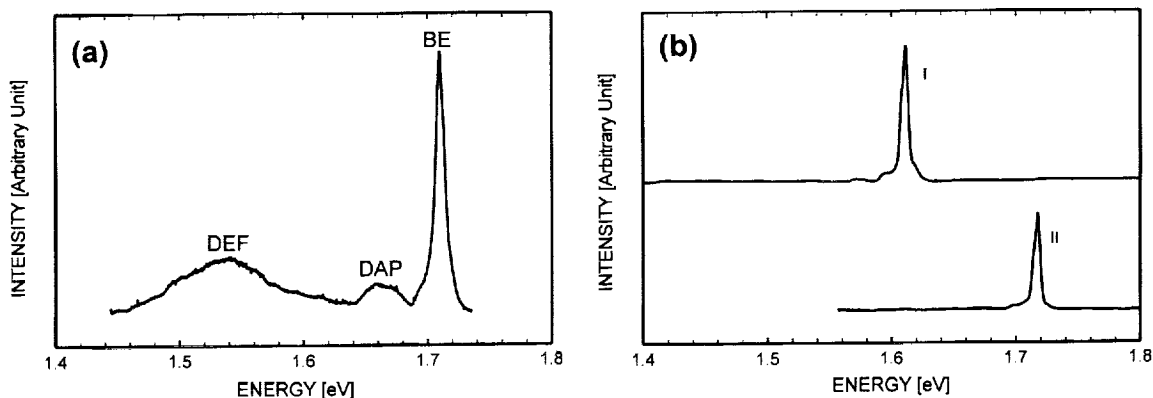


Fig. 3. Photoluminescence spectra. (a) general features: bound exciton (BE), donor–acceptor pair (DAP), and deep impurity and crystal defects (DEF) transitions; (b) I: $x = 0.04$, no excess (Cd + Zn), II: $x = 0.19$, excess (Cd + Zn).

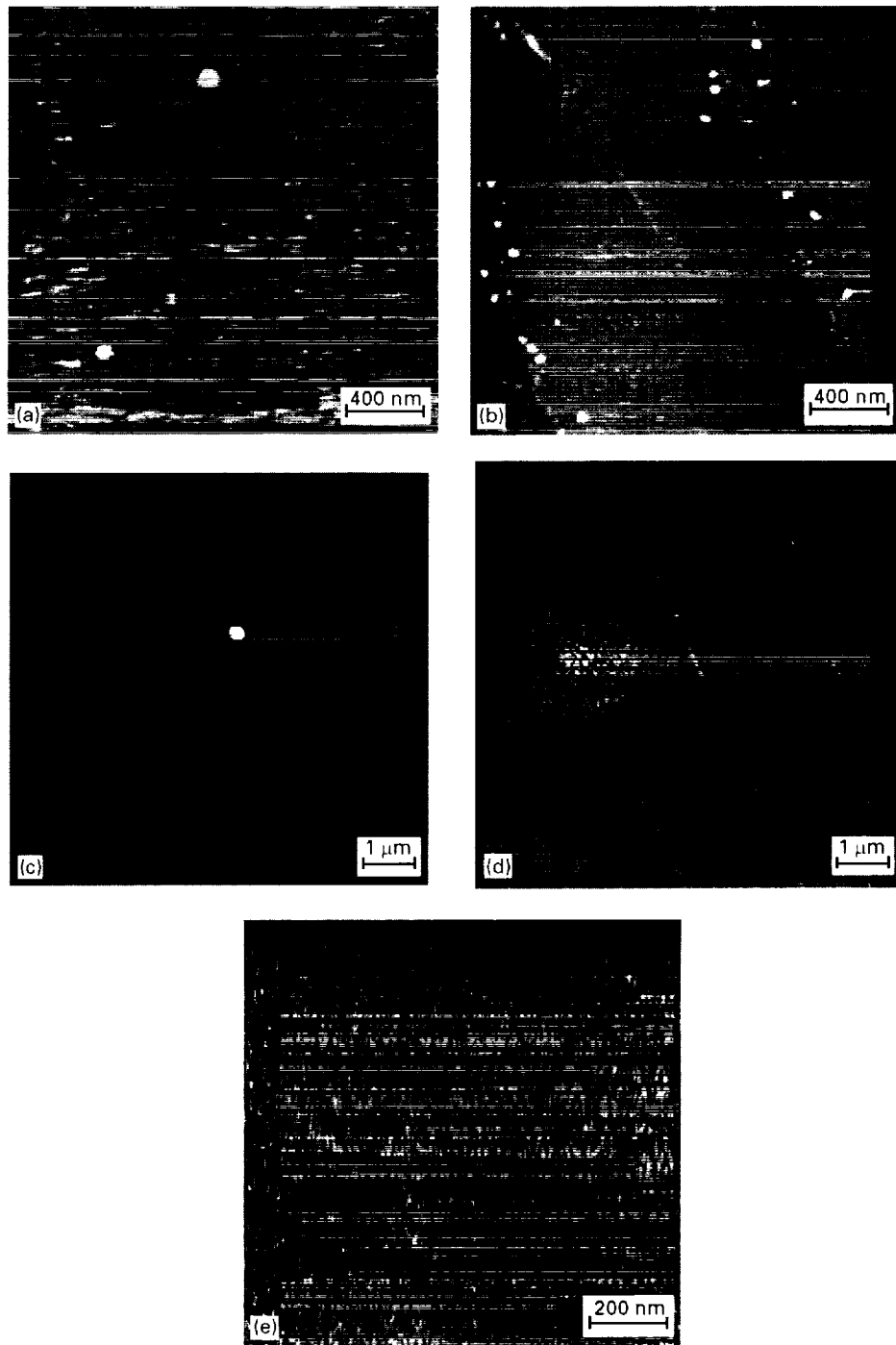


Fig. 4. Atomic force microscopy images. (a)–(d) precipitates; (e) voids. (a), (b), (c) and (e) constant height mode; (d) constant force mode.

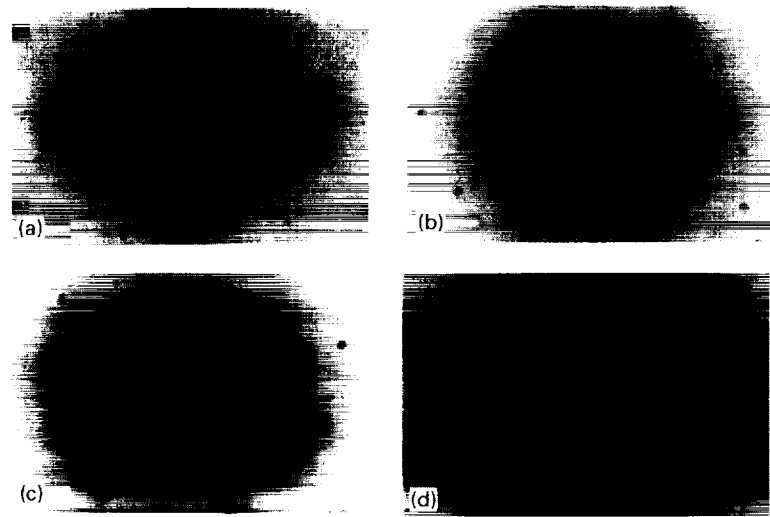


Fig. 5. Laue patterns. (a) $x = 0.04$, cool-down A; (b) $x = 0.04$, cool-down B; (c) $x = 0.14$, cool-down A; (d) $x = 0.20$, cool-down B.

exceeded 100 only in some samples. PL spectra of our better crystals are shown in Fig. 2b. No apparent effect of excess (Cd + Zn) pressure on PL spectra of the grown crystals was observed.

3.3. Atomic force microscopy

The morphology of freshly cleaved $\{110\}$ surfaces of selected samples was investigated with the AFM technique. It was found that the cleaved surfaces are very smooth, with the (average) roughness typically in the 0.1–0.3 nm range. This indicates good compositional microhomogeneity of the crystals. Submicron precipitates were found in some samples (Fig. 4a–Fig. 4c). The precipitates are usually small, with a size of 20–50 nm (Fig. 4a). An increased number of the precipitates was observed in slowly cooled (procedure B) crystals of low ($x = 0.04$, Fig. 4b) but not high ($x = 0.2$) content of ZnTe. Sometimes precipitates a few hundred nanometers in size were found (Fig. 4c). Larger size precipitates (above 100 nm) were found in crystals grown under Cd-excess pressure. Their presence is observed primarily in low ZnTe-content crystals and is accompanied by an occasional occurrence of a much larger size feature (about 1 μm in diameter, Fig. 4d). Sub-micron voids were sometimes observed at locations 20–30 mm from the seed crystal

(Fig. 4e). They might have been caused by an insufficient interfacial temperature gradient resulting in morphological instabilities.

3.4. Additional characterization

X-ray Laue diffraction patterns for low- and high-ZnTe content and for both post-growth cool-down rates are shown in Fig. 5a–Fig. 5d. The Laue patterns show good crystallinity without a second (ZnTe-rich, Refs. [15, 16]) phase. The resistivity of most crystals is in the range 10^3 – 2×10^5 Ωcm and is independent of the growth conditions.

4. Summary

Relatively large crystals of cadmium–zinc telluride have been grown on seeds by physical vapor transport. Both low ($x = 0.04$) and high ($x = 0.2$) ZnTe-content crystals were obtained. Subgrain boundaries were found to form only in low ZnTe-concentration crystals when slow post-growth cool-down is applied. PL spectra and AFM measurements show that high quality crystals can be obtained by this method. No apparent second phase (ZnTe-rich phase) has been detected even after a slow post-growth cool-down of the crystals.

A small excess of (Cd + Zn) in the vapor phase (0.1–0.2 atm) improves compositional homogeneity without any meaningful effect on the crystal morphology.

Acknowledgements

The authors are pleased to acknowledge the support of this work by the National Research Council Associateship Programs (W.P.), and by the Microgravity Science and Applications Division of the National Aeronautics and Space Administration through the Marshall Space Flight Center and NASA/Fisk Center for Photonic Materials and Devices.

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