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Characterization of cadmium-zinc telluride crystals grown by 'contactless' PVT using synchrotron white beam topography

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Abstract

Crystals of $Cd_{1-x}Zn_xTe$ grown by PVT using self-seeding 'contactless' technique were characterized using synchrotron radiation (reflection, transmission, and Laue back-reflection X-ray topography). Crystals of low ($x = 0.04$) and high (up to $x \approx 0.4$) ZnTe content were investigated. Twins and defects such as dislocations, precipitates, and slip bands were identified. Extensive inhomogeneous strains present in some samples were found to be generated by interaction (sticking) with the pedestal and by composition gradients in the crystals. Large (up to about 5 mm) oval strain fields were observed around some Te precipitates. Low angle grain boundaries were found only in higher ZnTe content ($x \geqslant 0.2$) samples.

PACS: 81.05 Dz; 61.72. Ff

Keywords: PVT; Cadmium-zinc telluride; Defects

1. Introduction

High quality crystals of cadmium-zinc telluride are required for fabrication of high performance IR and γ-ray detectors [1, 2]. Physical vapor transport (PVT) technique, which allows for a relatively low growth temperature without a presence of a second condensed phase, may offer an improved quality material relative to that grown from the melt. Recently we reported on growth and characterization of $Cd_{1-x}Zn_xTe$ crystals obtained by seeded and 'contactless' unseeded PVT techniques [3, 4]. The present work contains additional characterization of the latter performed using synchrotron white beam X-ray topography (SWBXT) in Laue transmission and Bragg reflection geometries [5]. The technique allows both for general characterization as well as imaging of defects in the crystals [5].

2. Experimental procedure

Materials and ampoule preparation, and growth of our cadmium-zinc telluride crystals are described

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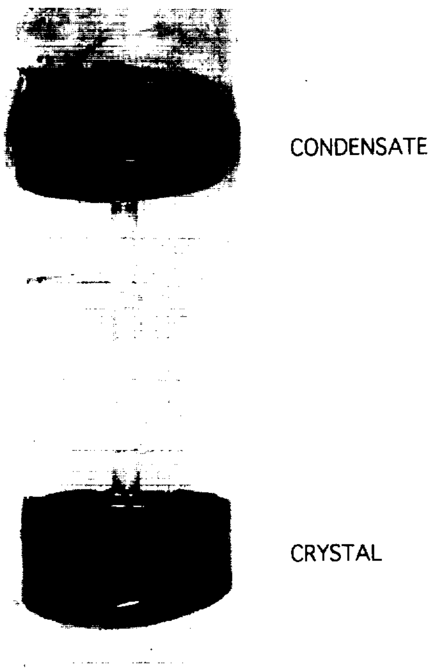


Fig. 1. Crystal in the ampoule after growth (grid in mm).

in Ref. [3]. Fig. 1 shows a typical ampoule with crystal after growth. The crystals were sectioned parallel to the pedestal and mechanically and chemo-mechanically polished (2 vol.% Br in ethylene glycol). To remove mechanical damage, at least 500 μm thick layer on both sides of the sample was etched out prior to subsequent characterization. The topography images were obtained using the White Beam Camera on the Stony Brook Synchrotron Topography Facility at the National Synchrotron Light Source. The samples were oriented using the back-reflection Laue technique and then imaged in the reflection and transmission geometries.

3. Results and discussion

The SWBXT technique allowed for identification and characterization of different types of defects present in the crystal: linear defects such as dislocations, planar defects such as twins and subgrain boundaries, and volumetric defects like precipitates. Non-homogeneous strains present in the samples were also characterized. Using the broad wavelength range of the synchrotron radiation, twinned domains in crystals can be identified by means of orientation contrast, i.e., the matrix

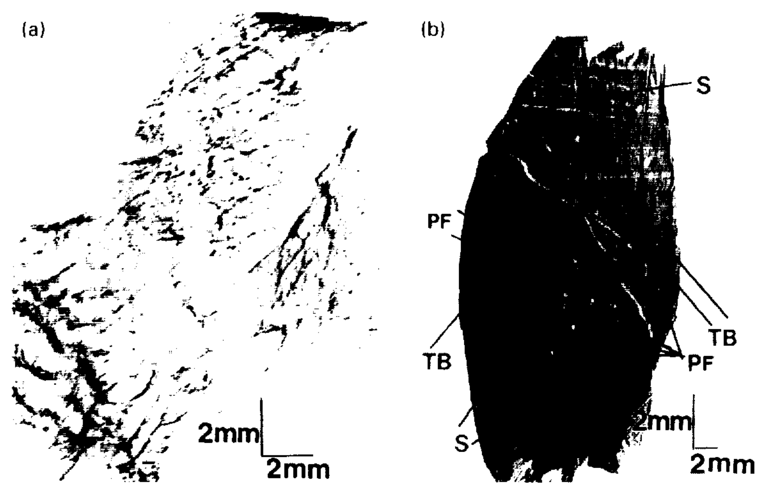


Fig. 2. SWBX reflection topographs of cadmium telluride. PF – precipitate contrast features, TB – twin boundaries, S – slip bands. (a) $z = 1$ mm; (b) $z = 7$ mm, $\boldsymbol{g} = (2\ 2\ 0)$.

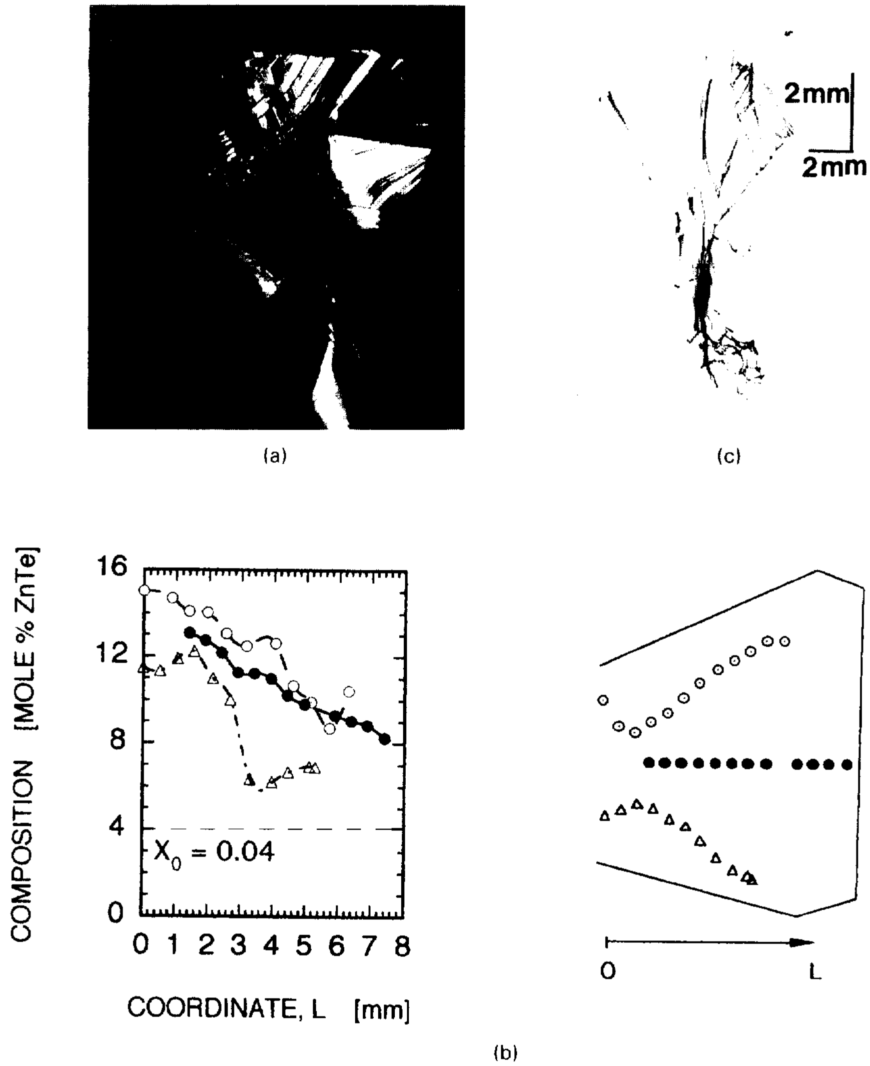


Fig. 3. Growth on the source, $x_0 = 0.04$. (a) crystal (grid in mm); (b) representative composition profiles (electron microprobe analysis results, left) and scanning paths (right) across the crystal; (c), SWBX reflection topograph, $g = (\bar{4}\,0\,\bar{4})$.

and twinned oriented regions select different wavelengths for diffraction and give rise to images which are shifted with respect to each other depending on the particular reflection. This can be used to reveal the orientation relationships between the matrix and twinned regions. Dislocations can be identified when the distortion field around a dislocation diffracts kinematically giving rise to enhanced diffracted intensity compared with the surrounding perfect region which diffracts dynamically. Sub-grain boundaries comprise of dislocations usually arranged in a tilt fashion and can be identified by means of orientation contrast. The contrast of precipitates depends on the strain field associated with a precipitate. Normally a spherical precipitate has a radial strain field. Images of such precipitates consist of two dark, kinematic lobes positioned in the ± directions of the reciprocal lattice vector (diffraction vector, g) for a given reflection (topograph), separated by a region of significant lattice rotation [6]. Precipitate contrast is more complex if the strain field is non-radial. Lattice strains/distortions can be discerned in the synchrotron topographs as darker, irregular lines of variable thickness and contrast.

3.1. Non-homogeneous strains

Two sources of non-homogeneous strains were found in the samples. Fig. 2a and Fig. 2b show images of our CdTe crystal at a distance of $z = 1$ and 7 mm from the pedestal, respectively. The sample close to the pedestal (Fig. 2a) shows severe lattice distortions, which are essentially absent in the further part of the crystal (Fig. 2b). Apparently the large inhomogeneous strains were caused by sticking of the crystal to the pedestal and a resulting stress (caused by a difference in thermal expansion coefficients of fused silica and crystal) developed upon cooling the ampoule after growth.

Another source of lattice strain is illustrated in Fig. 3. The crystal (Fig. 3a) has formed on the source (without excess Cd) by local resublimation and with no contact with the walls of the ampoule [4]. However, the composition of the crystal is very non-uniform (Fig. 3b) what apparently leads to severe distortions of the crystal lattice (Fig. 3c). Similar lattice distortions were observed in other crystals with non-uniform distribution of zinc in the lattice (Fig. 4a and Fig. 4b). Growth under very low undercooling or excess Cd pressure improves compositional homogeneity of the crystals [7, 8]. With sufficient Cd pressure, high compositional uniformity and essentially elimination of the lattice distortion can be achieved (Fig. 4a and Fig. 4c).

3.2. Precipitates

Precipitates (P) were observed in all crystals independent of their composition and growth conditions. The precipitates were found to be tellurium-rich (> 90% Te) as determined by the SEM/EDS analysis. Some were associated with an oval contrast precipitate feature (PF) around them. Relatively small (less than 100 μm) and larger (up to 800 μm in diameter) features were observed in cadmium telluride (Fig. 2b and Fig. 5a). Generally larger ones (up to a few μm in diameter, Fig. 4b,

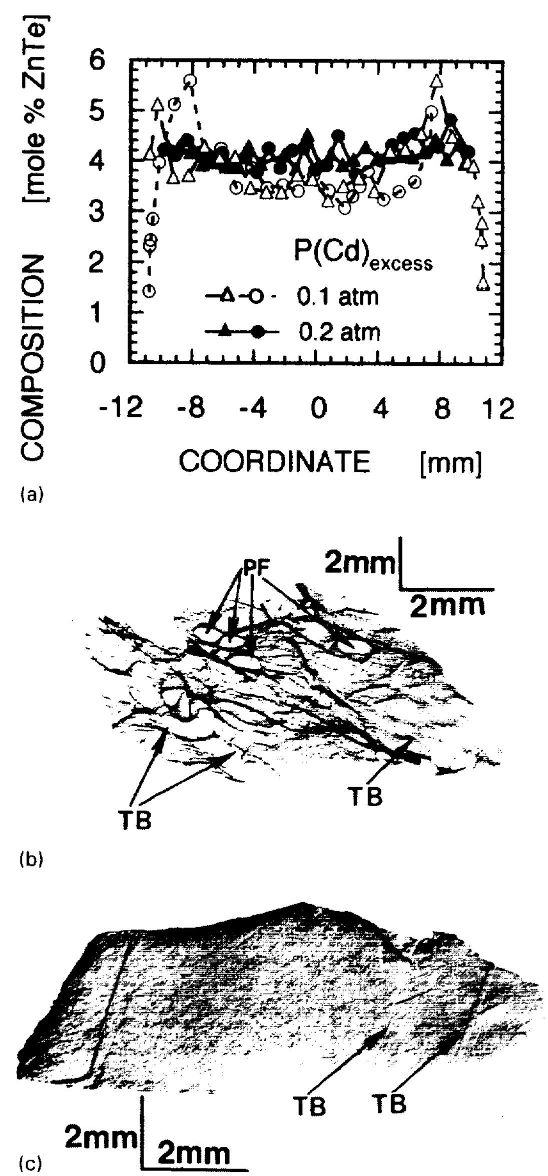


Fig. 4. Lateral composition profiles of (Cd, Zn)Te crystals and SWBX reflection topographs of their representative samples. $x_0 = 0.04$. PF – precipitate contrast features, TB – twin boundaries. (a), lateral composition profiles; (b) $P(Cd)_{excess} = 0.1$ atm, reflection topograph; (c), $P(Cd)_{excess} = 0.2$ atm, reflection topograph, $g = (5\ 3\ \bar{1})$.

Fig. 5b, Fig. 6a, Fig. 6b, and Fig. 7) were observed in cadmium-zinc telluride samples. Fig. 6a and Fig. 6b show reflection (top) and transmission (bottom) images of samples of $x = 0.04$ and 0.20,

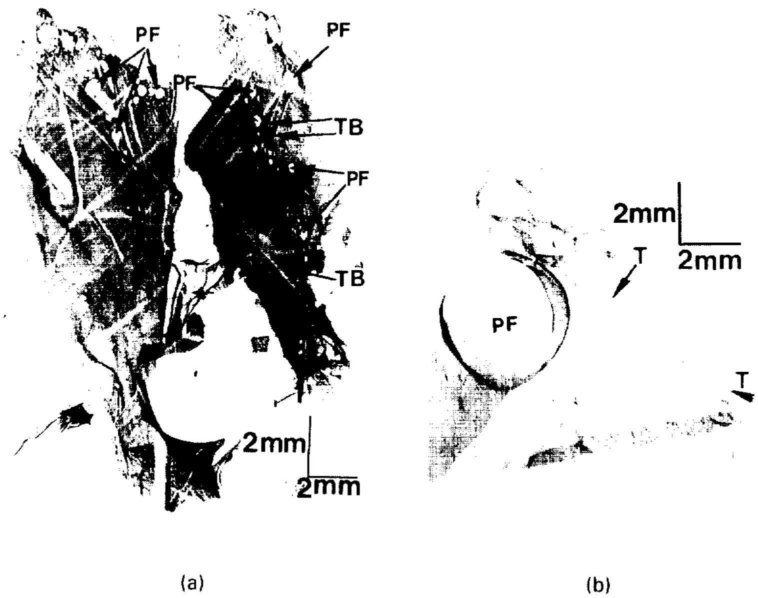


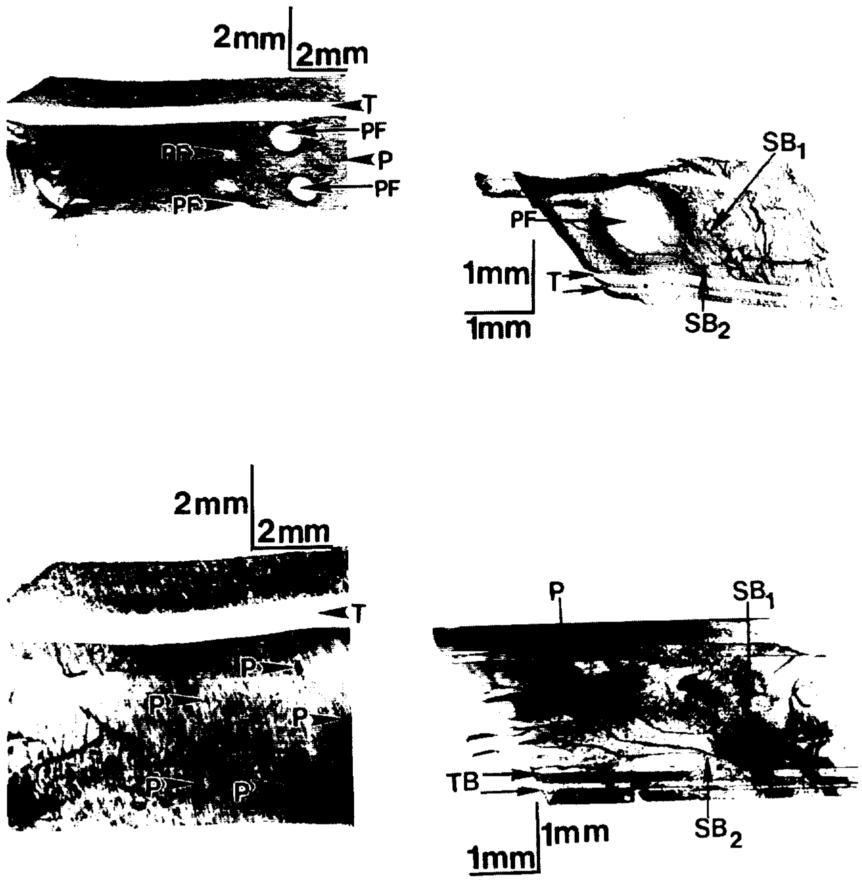
Fig. 5. SWBX reflection topographs. PF – precipitate contrast features, T – twin bands, TB – twin boundaries. (a) CdTe, $\boldsymbol{g} = (5\ 3\ 1)$; (b) (Cd, Zn)Te, $x_0 = 0.04$, $\boldsymbol{g} = (\bar{3}\ \bar{3}\ \bar{5})$.

respectively. The oval contrast features in the upper figures (reflection mode) are centered in locations corresponding to Te precipitates observed in the lower micrographs (transmission mode) (Fig. 6a and Fig. 6b). The strain field is apparently caused by high density of dislocation loops formed around Te precipitates [9]. Relatively limited number of the contrast features in the samples suggests, that the long range strain field forms only around larger size precipitates. Based on our earlier studies of (Cd, Zn)Te crystals grown by PVT under similar conditions, the largest precipitates are about 1–2 μm in size [3]. That means, that distortion of the lattice caused by the precipitates may be three orders of magnitude larger than the size of the precipitate alone.

In general, formation of a second phase of the constituent element in a crystal (Cd, Zn, or Te in cadmium-zinc telluride) can be due to two basic mechanisms [10]. Retrograde solubility of constituent element(s) leads to supersaturation and formation of precipitates (typical size a few nanometers) upon cooling to lower temperatures. Mechanical entrappment of non-congruent melt by the growing crystal surface leads to formation of inclusions (which may reach a few μm and more in size). Under our experimental conditions, partial pressures of Zn, Cd, and Te are about one order of magnitude or more below the respective saturation pressures. Therefore formation of a macroscopic (a few μm or more in thickness) liquid layer on the growing crystal surface is not possible, and has not been observed. In view of the above the tellurium features observed in our crystals seem to be precipitates formed after growth. However, the mechanism of formation of these large precipitates is not clear at this moment.

3.3. Linear defects

Except for the seed crystal (Fig. 3c), all larger size grains show a presence of 180° twins (T – twins, TB – twin boundaries, Fig. 2b, Fig. 4b, Fig. 4c and Figs. 5–8). That implies, that the probability of occurrence of a twin feature increases with the increase in the size of the grain. In some samples slip bands (S) regions were observed (Fig. 2b and Fig. 7). They do not cross the twin boundary lines,



(a) (b)

Fig. 6. SWBX topographs. P – precipitates, PF – precipitate contrast features, T – twin bands, TB – twin boundaries, SB_1, SB_2 – subgrain boundaries. (a), $x_0 = 0.04$, $P(Cd)_{excess} = 0.2$ atm; (b), $x_0 = 0.2$, $P(Cd)_{excess} = 0.1$ atm. Top – reflection geometry (left, $\boldsymbol{g} = (\bar{1}\,\bar{3}\,\bar{5})$); right, $\boldsymbol{g} = (6\,0\,\bar{2})$), bottom – transmission geometry (left, $\boldsymbol{g} = (1\,3\,\bar{1})$; right, $\boldsymbol{g} = (\bar{1}\,1\,3)$).

what indicates that the twinning occurred earlier than the slip process and that the twin boundaries act as a barrier to dislocation motions [11, 12]. The above observations support earlier literature conclusions [13, 14], that twinning is primarily the growth phenomenon, and not the result of the crystal deformation under stress.

Two types of subgrain boundaries, probably generated by stresses (due to possible local compositional nonuniformities) were observed in crystals of high concentration of zinc ($x \geqslant 0.2$). The first type (SB_1 in Fig. 6b and Fig. 8) forms cell structures comprised of an array of subgrain boundaries having the width of 50–200 μm with regions of very low dislocation (D) density. The subgrains must have been formed by the mechanism of dislocation glide and climb [15]. The formation of the second type of subgrain boundaries, which are long and straight (SB_2, Fig. 6b), may arise from polygonization of slip dislocations [16]. The boundaries might have formed during growth or post-growth cooling period (T(source) → 100°C in 3 h).

Dislocation densities of the better samples (Fig. 1b, Fig. 4c, Fig. 6a and Fig. 7) are in the low $10^4/cm^2$ range what is consistent with etch pit density results reported earlier [4].

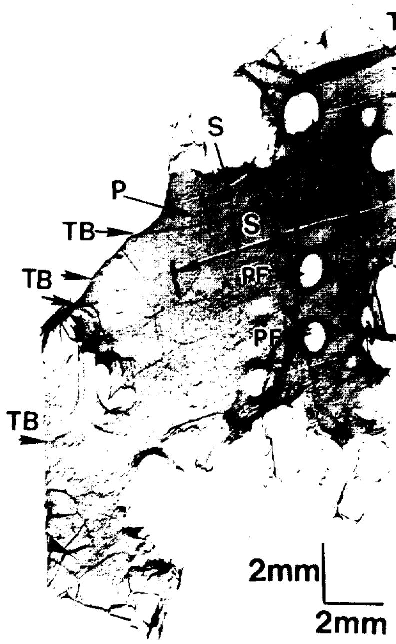


Fig. 7. SWBX reflection topograph, $g = (4\ 2\ 6)$. P precipitates, PF precipitate contrast features, TB twin boundaries, S slip bands. $x_0 = 0.04$, $P(Cd)_{excess} = 0.2$ atm.

4. Summary

Cadmium telluride and cadmium-zinc telluride crystals grown by 'contactless' PVT have been characterized using synchrotron white beam X-ray topography. Large inhomogeneous strains present in some samples were found to be caused by interactions with the ampoule (sticking to the pedestal) and by composition gradients (non-uniform distribution of ZnTe) in the material. A presence of an occasional oval strain field (up to a few μm in size) was observed in a number of samples. The strain fields were found to occur around (larger) Te-precipitates and to be about three orders of magnitude larger than the precipitates alone. Twin grains present in the crystals were formed during growth and not by thermal stresses caused by temperature and/or composition gradients. Low angle boundaries were formed occasionally in crystals of higher ($x \geqslant 0.2$) concentration of zinc telluride.

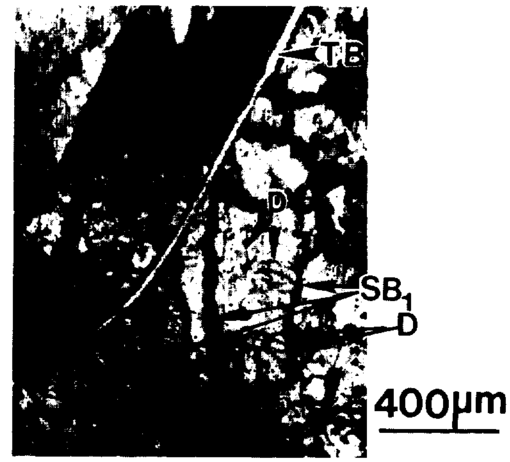


Fig. 8. Magnified transmission topograph of a region with subgrain boundaries, $g = (0\ 2\ \bar{2})$. TB twin boundaries, SB_1 subgrain boundaries, D dislocations.

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