A new approach to crystal growth of $Hg_{1-x}Cd_xTe$ by the travelling heater method (THM)

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Crystal growth by the travelling heater method (THM) is reported using a source material preparation process that is different from all methods used before. Non-stoichiometric (Hg,Cd)Te melts were homogenized and quenched to prevent macroscopic segregation effects. Inclusions of excess Te were removed during a first THM pass, resulting in stoichiometric solid alloys with a shift of the mole fraction towards higher CdTe contents. The amount of the shift, dependent on the Te excess and on the equilibrium temperature of the first THM run, was calculated and taken into account in the preparation of $x = 0.22$ and $x = 0.30$ $Hg_{1-x}Cd_xTe$ single crystals. Source material ingots, as well as THM single crystals, were characterized with special emphasis of the compositional homogeneity. Radial as well as axial homogeneity are comparable with the best results on THM crystals reported so far. The described method can be used in growing all materials for which THM is possible. However, quantitative calculation requires the exact knowledge of the particular ternary phase diagram.

1. Introduction

For more than two decades the narrow-gap semiconducting crystals of $Hg_{1-x}Cd_xTe$ (MCT) have been well accepted as IR detecting material. In recent years related alloys, among them $Hg_{1-x}Zn_xTe$, have been regarded as alternative candidates for the same application. The capability to cover a wide wavelength range from about 3 to 15 μm by only shifting the mole fraction renders these materials very attractive. On the other hand, the demands on small scale compositional uniformity at a desired level are extremely high. However, up to now the thermodynamical properties of these materials (high Hg vapour pressure of the stoichiometric melts, and wide separation between solidus and liquidus temperatures leading to segregation coefficients much higher than unity) have restricted the possible methods for bulk crystal growth. These thermodynamically driven problems can be solved, for example, by using the travelling heater method (THM) which has been already demonstrated by Triboulet et al. [1,2].

THM combines solution growth, allowing relatively low growth temperatures which result in low vapour pressures, and steady-state conditions with respect to the materials balance, well known from the principle of zone melting. It is these steady-state conditions between dissolution of the source material at the upper interface (see fig. 1) and crystal growth of an alloy of the same mole fraction at the lower interface which reduces the problem of segregation and may produce a uniformally distributed mixed crystal. It has been shown in a previous paper [3] that in THM a modified zone melting distribution of the axial composition is valid. Taking a homogeneous source ingot, the mole fraction in the THM-grown crystal will asymptotically decrease to the original value of the source. This feature shows the basic problem of THM for such alloys. In order to carry out a THM crystal growth run to produce a homogeneous single crystal, a macroscopically homogeneous ingot is needed as a source.

The simplest way to get the source ingot is the homogenization and successive quenching of a stoichiometric melt. However, in the case of HgTe-containing alloys this is somewhat dangerous because
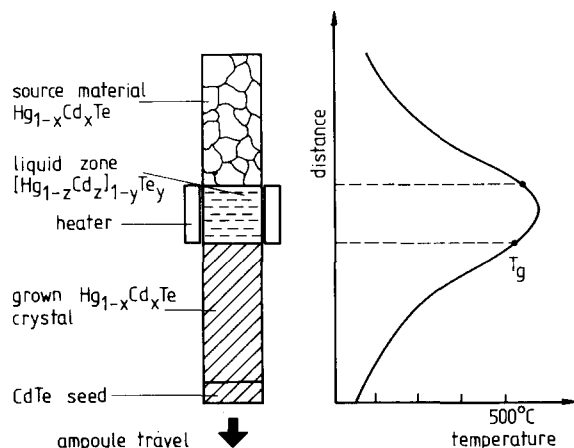
Fig. 1. Scheme of the vertical THM crystal growth arrangement used in the experiments. The temperature at the upper side of the Te-rich solution zone is slightly higher than the growth temperature T_g (530–550°C).

of the high Hg vapour pressure at the liquidus temperature of the desired liquid mixture (e.g. 3.5 MPa for $x = 0.22$ [4,5]) and may occasionally result in explosive failure.

Triboulet et al. [1] have demonstrated that the source ingot need not consist of a homogeneous alloy but can be constituted of two segments of the binary compounds in which the cross-sections fix the mole fraction of the ingot to be grown. This approach has given good results with a variety of different mole fractions and diameters of 30 mm which are in principle not restricted by the method. Nevertheless, there are several drawbacks, such as the very high accuracy necessary in mechanical adjustment, which not only influences the average mole fraction of the composed cross-section of the ingot, but also has to prevent any cavity between the source and the wall of the ampoule. Additionally, this excessive handling results in the risk of contamination. On the other hand, it is difficult to understand the situation at the dissolving interface. The liquid solution zone cannot establish equilibrium conditions with both segments, CdTe and HgTe, at the same temperature. Hence, there has to be a shift between the two parts of the segment interfaces, bringing the HgTe interface to lower and the CdTe interface to higher temperatures, and in this way creating a discontinuity in the upper solid–liquid interface. This influences, at least in the starting region, the axial distribution of the mole fraction and would require an excessive program of optimization to repeat the published results. The same would be true with the method of Schmitz et al. [6], which is different in preparation but leads to a similar situation with cylindrical segments.

There is another procedure, incremental quenching, which was originally designed for single crystal growth [7] but recently has been used to produce source ingots for THM growth [8]. However, by melting stoichiometric $Hg_{1-x}Cd_xTe$ material, the risk of explosion is present once more.

For these reasons we have taken a new approach to prepare the macroscopically homogeneous ingot necessary for THM crystal growth. The basic idea is to start with a Te-rich $Hg_{1-x}Cd_xTe$ melt, so as to lower the Hg vapour pressure, followed by quenching and then removal of Te inclusions by a first THM pass.

2. Principle

The principle of the source material preparation under consideration is depicted in fig. 2. The excess Te added to the stoichiometric composition of $Hg_{1-x_0}Cd_{x_0}Te$ has to be high enough to significantly lower the
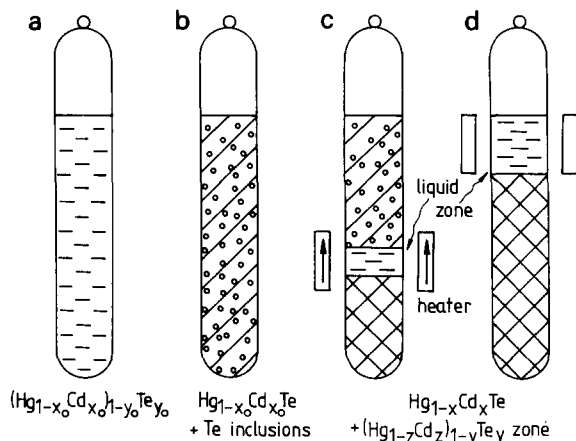
Fig. 2. Principle of the source material preparation for THM crystal growth. In order to lower the Hg pressure, excess Te is used in the original ternary liquid (a) which crystallizes as a second phase during quenching (b). In a first THM-like procedure, excess Te is collected under the influence of the travelling heater (c, d).
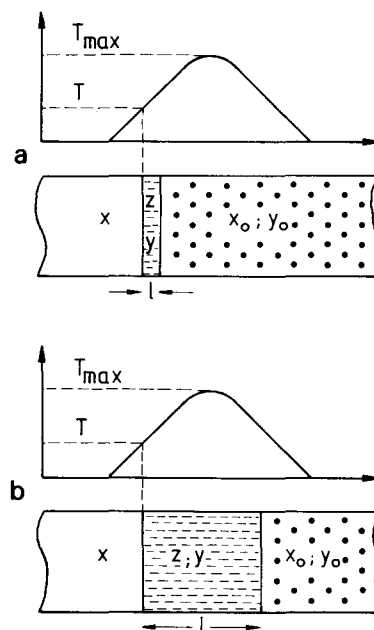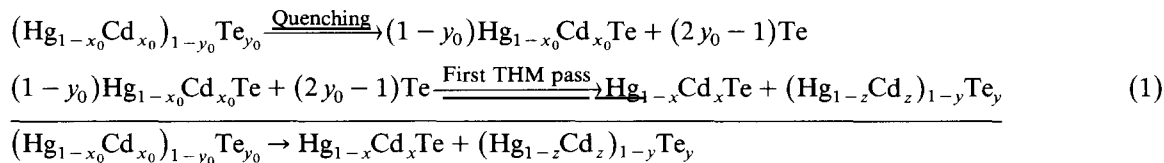
Fig. 3. Detailed picture of the process of forming a liquid solution zone $(Hg_{1-z}Cd_z)_{1-y}Te_y$ from the Te inclusions being homogeneously distributed in the $Hg_{1-x_0}Cd_{x_0}Te$ ingot.

vapour pressure and in this way to prevent any risk of explosion. After homogenization, the non-stoichiometric melt has to be quenched fast enough to prevent axial macroscopic segregation with respect to the mole fraction x_0 and to make sure that all second phase inclusions become homogeneously distributed. The ampoule with the quenched ingot inside is then transferred to the THM furnace for the first THM run without opening the ampoule. During this stage the ring furnace produces an axial temperature profile (compare with fig. 1) inside the ingot and collects all the Te inclusions to form a permanently increasing solution zone. The solid ingot crystallized from the solution zone is well suited to act as a source for THM crystal growth.

Fig. 3 depicts the process during the first THM run, which collects the randomly distributed inclusions of excess Te. At the start, the separated inclusions become liquid under the influence of the temperature field and behave like a liquid droplet in a temperature gradient [9]. Each droplet has a hotter and a colder side, leading to a solubility with respect to the MCT matrix material which is higher or lower, respectively. The concentration gradient resulting inside the droplet is the force for diffusion. As a result the liquid phase moves towards the direction of the temperature gradient. Regarding it as a one-dimensional problem, the liquid droplet becomes a very thin liquid solution zone, as shown in fig. 3a. This is the situation of the well-known travelling solvent method (TSM) first described by Pfann [10]. While moving the ring heater with respect to the ingot, with a travel rate low enough to allow the diffusion to take place, the thin solution zone collects more and more Te inclusions and becomes longer and longer. In some later stage the liquid zone is long enough (pictured in fig. 3b) to have the maximum of the axial temperature profile inside the solution zone. This is the case of THM growth with a solution zone increasing up to the point when the zone reaches the end of the ingot.

The symbols in fig. 3 are used to write the equations of the solubility reactions:

$$\left(Hg_{1-x_0}Cd_{x_0}\right)_{1-y_0}Te_{y_0} \xrightarrow{\text{Quenching}} (1-y_0)Hg_{1-x_0}Cd_{x_0}Te + (2y_0 - 1)Te$$

$$(1-y_0)Hg_{1-x_0}Cd_{x_0}Te + (2y_0 - 1)Te \xrightarrow{\text{First THM pass}} Hg_{1-x}Cd_xTe + (Hg_{1-z}Cd_z)_{1-y}Te_y \qquad (1)$$

$$\overline{\left(Hg_{1-x_0}Cd_{x_0}\right)_{1-y_0}Te_{y_0} \rightarrow Hg_{1-x}Cd_xTe + (Hg_{1-z}Cd_z)_{1-y}Te_y}$$

where x_0 and y_0 are the mole fractions in the original non-stoichiometric melt, z and y are the mole fractions in the liquid zone during the first THM pass, and x is the mole fraction in the resulting ingot to be taken as source for the THM crystal growth.

When estimating the solubility equilibria, the quenched ingot (fig. 2b) as an intermediate product need not to be regarded in detail provided it is macroscopically homogeneous with respect to the mole fraction and the distribution of inclusions. Of course, the average mole fraction x_0 is the result of a microscopical variety of different mole fractions ranging almost to the binary compounds HgTe and CdTe as shown below. However, while dissolving the macroscopically inhomogeneous MCT matrix, the liquid will produce the same solution zone as it would do with a microscopically homogeneous matrix of x_0. It is, to a lesser degree, a similar situation to the macroscopic two-segment method of Triboulet [1]. This principle was first described to produce an in-situ solution zone for THM temperature profile measurements [11]. It is different from using the elements as the starting point for THM crystal growth, "cold THM", as developed by Triboulet et al. [12]. The authors themselves stated that their method failed to grow homogeneous (Hg,Cd)Te.

3. Theoretical

It is the aim of this section to describe the relation between the mole fractions x_0 of the original composition and x of the resulting single-phase ingot and its dependence on other parameters. As explained above, it is only necessary to regard the balance reaction of eq. (1). From the schematic in fig. 4 it can be seen that in some intermediate stage of the first THM pass the liquid solution zone dissolves an average mixture of $(Hg_{1-x_0}Cd_{x_0})_{1-y_0}Te_{y_0}$ at the upper interface and crystallizes single-phase $Hg_{1-x}Cd_xTe$. The solution zone has composition $(Hg_{1-z}Cd_z)_{1-y}Te_y$ and length l and may be regarded as consisting of two parts: a zone of the pure solvent Te, with the length l_0, and the geometrical difference $(l - l_0)$ with stoichiometric material $Hg_{1-z}Cd_zTe$. This involves the usual assumption that the solution is the sum of the individual volumes of the solvent and the solute. With a constant cross-section and flat interfaces we can treat the one-dimensional problem and find the length l_0 of the fictive pure solvent zone proportionally increasing with the distance d the dissolving interface has moved:

$$l_0 = d\left(1 + \frac{1-y_0}{2y_0 - 1}f\right)^{-1}, \qquad (2)$$

with

$$f = M_{MCT}\rho_{Te}/\rho_{MCT}M_{Te},$$

where f is the ratio of the molar volumes of MCT and Te (M is the molar mass and ρ is the mass density).

For a given material the increase of l_0 with d only depends on the degree of non-stoichiometry of the original composition y_0. The length l occupied by the solution zone depends not only on l_0 but also on the interface temperature T, determining the solubility, which should be described in terms of $y(T)$. If the
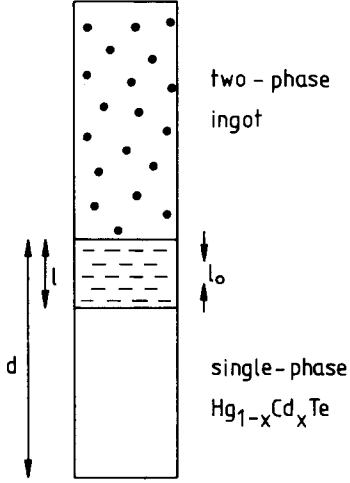
Fig. 4. Schematic sketch of the arrangement showing the different zone lengths used in the theoretical treatment. While l is the actual length of the liquid solution zone, l_0 is the hypothetical length of a zone consisting only of the pure solvent Te.
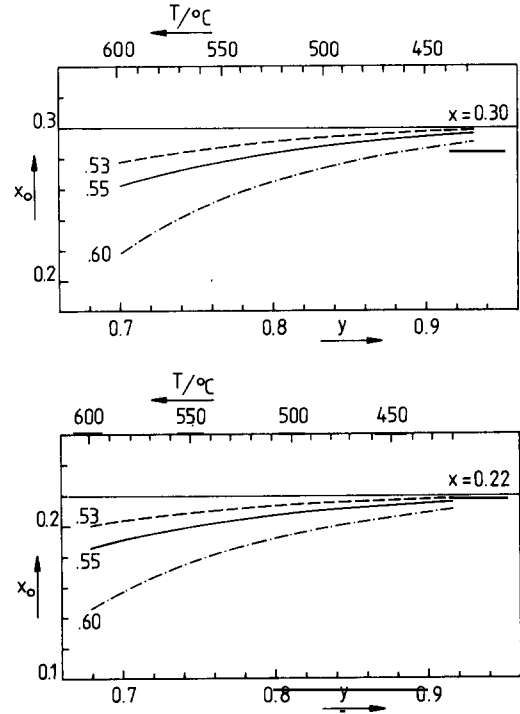

Fig. 5. Calculated plot of the mole fractions x_0 of the original ternary composition $(Hg_{1-x_0}Cd_{x_0})_{1-y_0}Te_{y_0}$ necessary to produce stoichiometric ingots of fixed mole fractions x. The upper diagram shows conditions for $x = 0.30$ ingots, the lower one for $x = 0.22$. The curve parameter describes the amount of Te (y_0) in the ternary liquid prior to quenching.

interfaces are held at a constant temperature, $y(T)$ is also fixed at a constant value and the total solution zone length also will be proportionally increasing with the axial distance:

$$l = \left(1 + \frac{1-y}{2y-1}f\right)l_0 = \frac{1 + \frac{1-y}{2y-1}f}{1 + \frac{1-y_0}{2y_0-1}f}d. \tag{3}$$

Although the interface positions are shifted away from the maximum of the axial temperature profile, their temperature can be held constant by increasing the maximum temperature. At the end of the first THM run, l describes that length of the total ingot which has to be rejected.

Regarding the relation between x_0 and x, it has to be remembered that in the MCT system the segregation coefficient $k = x/z$ is always much higher than unity. This means that every Te inclusion while being collected and forming a part of the solution zone binds more HgTe than CdTe compared with the solid alloy. The higher amount of HgTe necessary to produce the solution zone will be lacking in the solid to be crystallized from it. This explains why the ingot grown during the first THM pass is always higher in x than the original composition x_0. For a given mole fraction x_0 the amount of the shift $(x - x_0)$ depends on the quantity of inclusions defined by y_0 and the solubility $y(T)$ of the solution zone.

If all the prerequisites specified above are fulfilled, all mole fractions in the balance equation (eq. (1)) become independent of time and local position and the particle balance can be calculated, leading to

$$x_0 = \frac{1}{1-y_0}\left\{ xy_0 - \frac{2y_0-1}{2y-1}[xy - z(1-y)]\right\}.$$ (4)

With $x = kz$, z can be replaced by x/k and eq. (4) changes to

$$x_0 = \frac{x}{1-y_0}\left[y_0 - \frac{2y_0-1}{2y-1}\left(y - \frac{1-y}{k}\right)\right],$$ (5a)

or

$$x = x_0(1-y_0)\left[y_0 - \frac{2y_0-1}{2y-1}\left(y - \frac{1-y}{k}\right)\right]^{-1}.$$ (5b)

While eq. (5b) gives the resulting mole fraction x compared with the original mole fraction x_0, eq. (5a) is better suited for practical use. It predicts which x_0 will produce an ingot with the mole fraction x desired for the actual THM process.

Brice [5] has given a numerical description of the Hg–Cd–Te phase diagram including the solubility data $y(T, x)$ and the segregation coefficient $k(x)$ which must be known before the conditions for the procedure can reliably be set. These data have been used to produce fig. 5 for the most interesting MCT applications which require $x = 0.22$ and $x = 0.30$ alloys. The different curves in fig. 5 assume different mole fractions y_0. The higher the Te content y_0 in the original non-stoichiometric melt, which is beneficial in lowering the Hg vapour pressure, the larger the shift from the original mole fraction x_0 towards the desired value x. To achieve the desired mole fraction x with a high accuracy, conditions which cause only a small shift, i.e. relatively low interface temperatures and a small original excess of Te, are preferred.

Although the method can be used for the entire MCT system, the numerical description [5] has been given only for the range limited by $0 < z < 0.3$, $0.63 < y < 0.93$ and $420 < T < 600°C$. This is the only reason why this paper is restricted to low CdTe contents and does not deal with the higher mole fractions of interest for other applications.

4. Experimental

Thick-wall ampoules (wall thickness 3.5 mm) with an inner diameter of 16 mm and internally coated with graphite were filled with 6N-grade elements, according to the overall composition $(Hg_{1-x_0}Cd_{x_0})_{1-y_0}Te_{y_0}$ $(0.53 \leq y_0 \leq 0.60)$ and a total mass of about 150 g. After sealing the ampoule under high-vacuum conditions, synthesis was carried out in the horizontal position, followed by a 24 h homogenization step at temperatures only slightly higher than the liquidus temperature of the particular composition. The furnace was mounted on a horizontal axis but could be moved into a vertical position while keeping the ampoule with the molten material inside. The ampoule was then lowered at rates as high as 25 mm/min through a high temperature gradient so as to "quench" the non-stoichiometric melt.

The sealed ampoule was then transferred to the THM furnace for the first THM pass. To meet the need of a constant interface temperature (about 580°C for $x = 0.22$ or 600°C for $x = 0.30$) with a permanently increasing zone length in a linear temperature profile at both sides of the maximum (see fig. 1), use was made of a coordinated program between heating power, time and translation rate of the ampoule. With typical ampoule travel rates of 2 mm/day the first THM run took two months. After removing the ampoule by solving it in concentrated hydrofluoric acid, a small face was mechanically polished along the

periphery of the ingot so as to allow axial concentration measurements by electron probe X-ray microanalysis (EPMA) using the Cd Lα line.

The spherical tip of the ingot and the solution zone were removed using a wire saw, and the cylinder between them became the source for the THM crystal growth process. The ingot was not etched but carefully rinsed in boiling acetone and ethanol prior to re-loading for the final THM run. Crystal growth was carried out in thin-wall ampoules (wall thickness 1–1.5 mm) which have to perfectly suit the diameter of the source ingot. Silica tubes for the ampoules were chosen according to the exact diameter of the particular source. Seeds were ground on a small turning lathe. Use was made of CdTe seeds (either (111)$_A$ or (111)$_B$ orientation) and Te as a solvent, in a one-zone furnace with axial temperature gradients as high as 85 K/cm in the liquid adjacent to the phase boundary. The growth temperatures were about 530 °C for $= 0.22$ and about 550°C for $x = 0.30$ which is somewhat lower than in Triboulet et al.'s paper [1]. In a previous contribution [13], we have found such a low growth temperature favourable with respect to the structural perfection of the MCT crystal. As a consequence, the translation rate of the furnace with respect to the ampoule, which determines the growth rate under steady-state conditions, was chosen as low as 1 mm/day. The grown crystals were re-measured by electron probe microanalysis along the periphery and then cut into 1 mm thick slices perpendicular to the growth axis.

5. Results and discussion

Fig. 6 demonstrates the microscopic inhomogeneity of a quenched ingot prior to the first THM pass. To enable improved electron probe microanalysis measurements, without the additional problem of Te inclusions, this was occasionally carried out with stoichiometric $Hg_{1-x_0}Cd_{x_0}Te$ ($x_0 = 0.215$) grown under similar quenching conditions. The microscopic structure consisted of dendrites having compositions that range from nearly pure HgTe to high CdTe contents. Spacing of the dendrites depends on the quenching rate. Fast quenching leads to cavities at the end of the ingots which must be avoided in source material intended for THM crystal growth. As mentioned in section 4, "quenching" was carried out by simply lowering the ampoule with a rate as high as 25 mm/min. Using even higher translation rates (e.g. 40 mm/min), which had been successfully applied to thin wall ampoules before, cavities along the axis of the last-to-freeze part occur. By measuring a large number of points on a slice, the average mole fraction was found to correspond to the original composition of the melt. This is not true for the first part of the quenched ingot.

With the usual quenching rate, according to the ampoule travel rate of 25 mm/min, a transient in the first-to-freeze region is to be expected similar to the distribution function derived by Smith et al. [14]. Nevertheless, sometimes there is a segregation behaviour within the very first part which is not well understood by means of Smith's function. This corresponds to that section of the ingot where the individual inclusions have not yet formed an extended solution zone. This also explains why the theoretical prerequisites assumed previously are not strictly valid for the transient region. In fig. 7 the axial distributions in ingots after the first THM pass are depicted. It is again the first-grown part of an ingot where the deviation from axial homogeneity is marked. We do not have a satisfactory explanation why the initial x change is sometimes opposite (see fig. 7). It should depend on the spontaneous process of quenching and the undercooling that is necessary for nucleation. For this reason the first 15 mm or so of the ingot had to be removed.

A typical result of the radial distribution of the mole fraction in a slice cut perpendicular to the growth axis after the first zone pass is shown in fig. 8. This slice was taken from near to the final position of the zone and corresponds to the end of the cylinder used as a source for the THM process of an $x = 0.30$ crystal. Although slices from these last-to-freeze parts of the ingots are sometimes single-crystalline, they were never used for device fabrication as the radial distribution did not meet the requirements. The mole
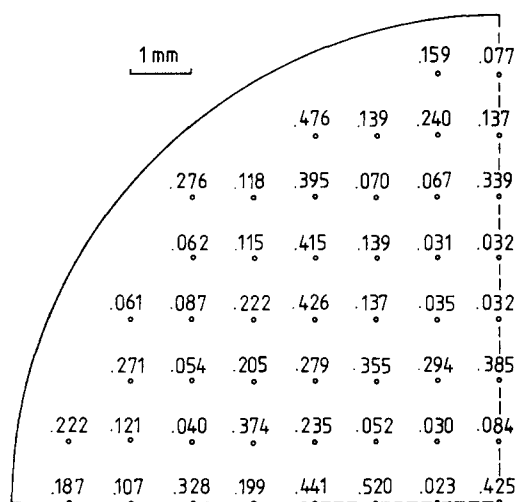
Fig. 6. Microscopic distribution of the mole fraction x on a quarter of a 16 mm diameter slice of an ingot quenched from a stoichiometric $Hg_{1-x_0}Cd_{x_0}Te$ melt ($x_0 = 0.215$). Electron probe microanalysis measurement using Cd Lα radiation.
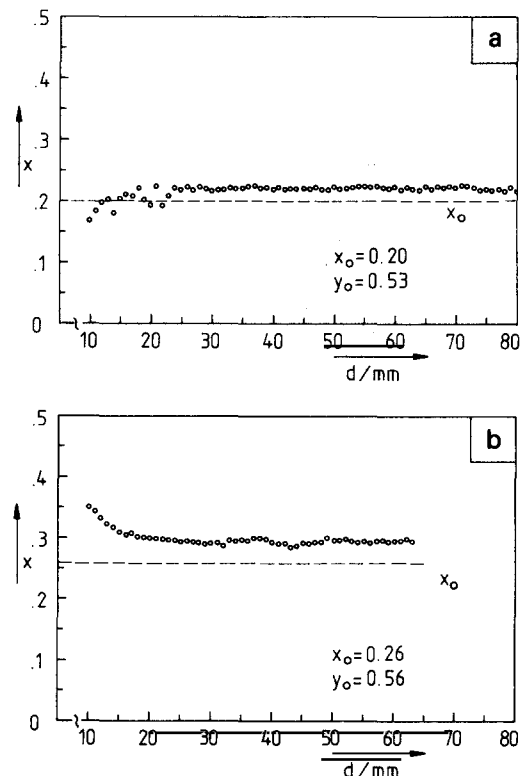
Fig. 7. Axial distribution of the mole fraction x along a $Hg_{1-x}Cd_xTe$ ingot after the first zone pass to produce THM source material for (a) $x = 0.22$ and (b) $x = 0.30$ crystals.

fractions of the slice of fig. 8 range from about 0.29 near the centre to about 0.31 at the periphery. From the radial distribution, a curvature of the growing solid–liquid interface can be determined which is concave with respect to the crystal. As is well known, the isotherms in ring furnaces used in THM crystal growth considerably change their shape, depending on the axial distance from the centre of the furnace [15]. This explains why the curvature of the phase boundary in the process under consideration will continuously change with zone length. As a consequence, the growing interface has a convex shape at the beginning and a concave one at the end of the first THM pass, resulting in corresponding radial distributions of the mole fraction. Only a relatively small number of slices in between may have a homogeneous mole fraction needed for device processing. However, to act as a source for the subsequent THM process, the total ingot is much more homogeneous than in all other methods used so far.

Fig. 9 and fig. 10 demonstrate the axial and radial distributions of the mole fraction in MCT after the THM crystal growth process. Inhomogeneities across the slices do not exceed the standard deviation of EPMA ($|\Delta x| \leq 0.003$) while the axial distribution shows good homogeneity, $|\Delta x| \leq 0.005$. No attempt to suppress the transient distribution ranging from the seed to the first 10 to 15 mm of the crystal was made. This would in principle be possible by using the starting-charge operation, as has been already demonstrated [1].

Besides the homogeneity of the MCT crystals, it has been found that the reproducibility of growing single crystals of well-defined orientation ($[111]_A$ or $[111]_B$ as growth axis) is the main advantage of THM.

Fig. 9. Axial distributions of the mole fraction x along $Hg_{1-x}Cd_xTe$ crystals grown on CdTe seeds. Source ingots ($x = 0.22$ and $x = 0.30$) were grown by a first zone pass.
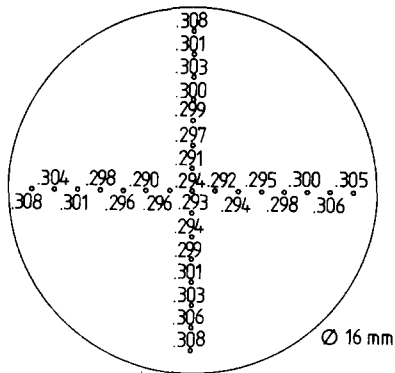

Fig. 8. Radial plot of a slice cut perpendicular from the end of a $Hg_{1-x}Cd_xTe$ ingot after the first zone pass. The minimum of the mole fraction in the middle of the slice indicates a curved growth interface.
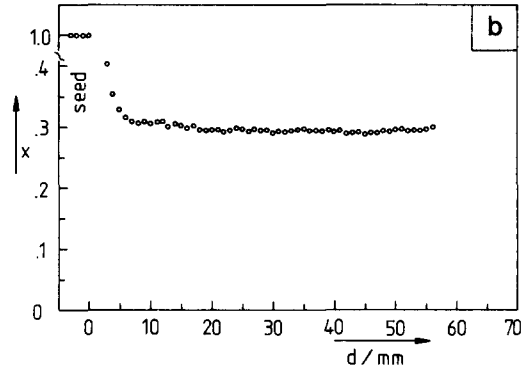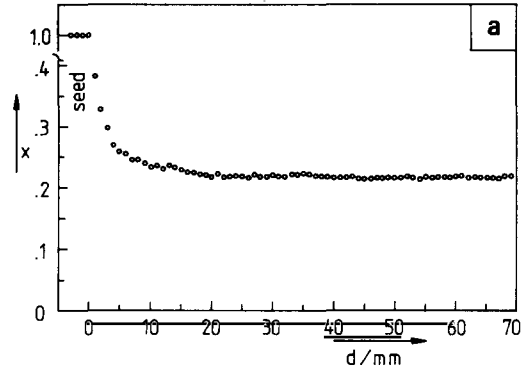
We have no argument to start device processing with {111} surfaces only, but in every case grain boundaries should be excluded. Crystal growth direction ⟨111⟩ was chosen by analogy to liquid phase epitaxy, which is in terms of the process of crystallization the same situation as in THM. However, it should be preferable to fix the parameter of surface orientation at least during optimizing device fabrication.
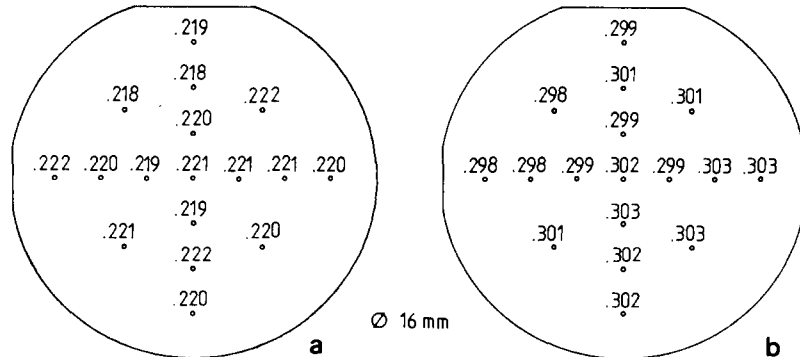

Fig. 10. Typical radial plots of the mole fraction x along 16 mm diameter slices of $Hg_{1-x}Cd_xTe$ single crystals.

The $Hg_{1-x}Cd_xTe$ crystals obtained during this study were characterized by X-ray double crystal topography and a chemical etching technique [16]. They show subgrain structures consisting of large grains (up to 1 mm^2) misoriented by angles less than 30 arc sec. Strong long-range lattice deformations are not in evidence. The dislocation density is below 10^5 cm^{-2}. Details of the investigations of the structural perfection have been reported elsewhere [13]. Contrary to the results obtained by Colombo et al. [8], we have not found marked differences with both directions, $[111]_A$ and $[111]_B$. Results on device properties which are not included in this study have been reported by Möllmann et al. [17].

6. Conclusions

The process of (Hg,Cd)Te crystal growth by THM based on a modified source material preparation starting from a Te-rich ternary has been described. This approach is well suited to produce axially as well as radially homogeneous MCT single crystals for device fabrication. The calculation of the shift in the mole fraction that occurs during the first pass of the solution zone necessitates the knowledge of the solubility data of the ternary phase diagram under consideration. Although this study has been restricted to 0.22 and 0.30 mole fractions of MCT, the method can be used for all materials produced by THM crystal growth.

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