

RESUMO - NOTAS / ABSTRACT - NOTES

We investigate the basic mechanism occurring in a diffusion-controlled growth of a  $Pb_{1-x}Sn_xTe$  crystal by the Vapor-Melt-Solid (VMS) technique. A physical model for the mass transfer by the vapor and liquid phases without parasitory convection is stated and solved with an accurate implicit moving-boundary finite-difference scheme. It is shown that growth by VMS in microgravity is expected to yeld very good-quality PbSnTe crystals, due to a mechanical stabilization of the liquid layer and enhancement of the streaming effect in the vapor phase. More accurate results concerning Bridgman growth of PbSnTe in the diffusion-controlled regime are also obtained. These preliminar investigations are intended to support an experiment being devised in cooperation with the Laboratory of Materials Science in Space of the USSR Space Research Institute to grow a PbSnTe crystal at MIR facilities.

OBSERVAÇÕES/REMARKS

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# THEORETICAL MODEL FOR THE SOLUTE DISTRIBUTION OF A POSTE GROWTH BY VMS IN MICROGRAVITY ENVIRONMENT

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We investigate the basic mechanism occurring in a diffusion-controlled growth of a  $Pb_{1-x}Sn_xTe$  single crystal by the Vapor-Melt-Solid (VMS) technique. A physical model for the mass transfer by the vapor and liquid phases without parasitory convection is stated and solved with an accurate implicit moving-boundary finite-difference scheme. It is shown that growth by VMS in microgravity is expected to yeld very good-quality PbSnTe crystals, due to a mechanical stabilization of the liquid layer and enhancement of the streaming effect in the vapor phase. More accurate results concerning Bridgman growth of PbSnTe in the diffusion-controlled regime are also obtained. These preliminar investigations are intended to support an experiment being divised in cooperation with the Laboratory of Materials Science in Space of the USSR Space Research Institute to grow a PbSnTe crystal at MIR facilities.

## 1. Introduction

Large and homogeneus ternary alloys single crystals are a must for high performance optoelectronic devices. One of the best candidates for infrared detectors and semiconductors lasers in the range of 5 to 12 µm is the narrow band-gap semiconductor Pb1-xSnxTe. However, carefully controlled growth of large crystals is necessary in order to attain the desired efficiency and lifetime figures in the final devices1. It is well--known that Bridgman-like growth in low gravity results in a crystal with a more homogeneus axial concentration and improved structural homogeneity2, 3. This is mainly due to the shift from convective growth on Earth to diffusion-controlled steadystate (DCSS) growth in Space. The same improvements are expected to take place when techniques using vapor transport are employed\*.

There are several ways to achieve DCSS growth on Earth by reducing the aspect ratio of the growing crystal. In Bridgman growth horizontally it is possible to sustain a DCSS growth for a 5 cm lenght crystal if the bore of the growth ampoule is reduced to 0.2 cm5. Another Earth-bound method to avoid normal freezing is the horizontal or vertical vapor-melt-solid (VMS) growth. In this method a thin stoichiometric liquid layer is formed at the interface between the solid and the vapor. Such liquid layer ahead of the crystallization front is the order of millimeters instead of the centimeters column of a Bridgman growth, reducing the Rayleigh or the Grashof numbers below critical values for vertical or horizontal growth respectively; in the vapor phase, mixing is greatly inhibited by the high axial transport rates (streaming) necessary to sustain the steady--state growth. Reducing the ampoule diameter to a few millimeters will not help to obtain crystals

for device fabrication, but with the employement of VMS method it is possible to get crystals with the same volume or even bigger than the normal Bridgman ones.

If it is possible to find a method for eliminating the buoyancy-driven convection on Earth, very little can be done about surface-tension convection. When analysing the ratio of Marangoni to Rayleigh numbers on Bridgman growth in space, It is seen that, even taking into account only the VT normal to the free surface, the surface-driven convection is an important factor for segregation. In contrast, the liquid layer in a typical VMS growth has a Bond number about 70 times smaller than a similar Bridgman growth.

Another reason to try VMS growth in space is to investigate physical transport rates by the vapor phase. Quantitative results from experiments with chemical vapor transport in space have shown an anomalous increase in the transport rates, and were interpreted loosely as being originated from thermogenerated laminar convection currents, whose effects would be masked by buoyancy in a normal gravity environment. In VMS, the vapor transport does not involve any chemical process, and therefore no anomalous increase in the transport rates should be observed if that hypothesis is correct. Also, the supression of convective currents in the vapor phase (other than

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streaming), that eventually could be present in Earth bound experiments, would result in an increase of axial homogeneity in the final crystal.

To fulfill this experiment we have a cooperation with the Laboratory of Materials Science in Space of the USSR Space Research Institute to grow a PbSnTe crystal at MIR facilities.

Section 2 in this paper shows a brief description of the VMS technique along with the results of Earth bound experiments. In Section 3 we analyze a simple diffusive Bridgman growth for the Pbi-xSnxTe system with the help of an accurate numerical technique; the results of that Section are fundamental for the understanding of the particular VMS features that we introduce in Section 4. We limit ourselves here to an unidimensional model to analyze the axial concentration distribution in the crystal, wich is very sensitive to vapor-melt transport rates, as well as to the degree of mixing. Studies of interface shapes are required for the description of radial segregation and bidimensional convection patterns and are under progress. Numerical details about the algorythms used will be published elsewhere.

## 2. The VMS Technique

The first detailed report about  $Pb_{1-x}Sn_xTe$  growth by VMS is from Kinoshita and Miyazawa<sup>6</sup>, who employed the horizontal assembly depicited in fig. 1.

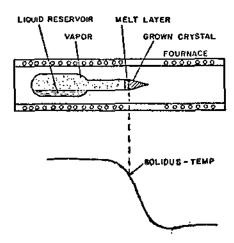


Fig. 1 - Horizontal VMS growth.

The liquid reservoir is maintained above the liquidus temperature (~10009C) and the vapor formed consists primarily of molecules of PbTe and SnTe. The vaporization takes place congruently, and the vapor is transported to the cold crystal growth region. A thin stoichiometric liquid layer is formed at the crystallization front, wich is maintained at the solidus temperature. The ampoule translation rate is empirically adjusted so that the balance between the condensation rate (wich depends on the vapor pressure and the normal thermal gradient in the vapor-liquid interface) and the solidification rate results in a layer thickness between 0.5 and 4mm in the steady-state<sup>T</sup> Typical growth velocities are in the range 0.1-1.0 mm/h.

In the expriments of Kinoshita and Miyazawa , the fournace temperature gradient at the solidus temperature was between 30 -609C/cm and with growth rates in the range 0.3-0.5 mm/h they obtained crystals 10 to 25mm in diameter and 5 to 10cm in length, some of them having axial uniformity within 5% along 60% of the crystal lenght. This is unattainable with a Bridgman growth under similar conditions.

The vertical VMS configuration, sketched in fig. 2, was developed by Ying An and Bandeira to fit the experimental facilities at INPE. It also permits a better stabilization of the liquid layer in the crystallization front when working in a normal gravity environment.

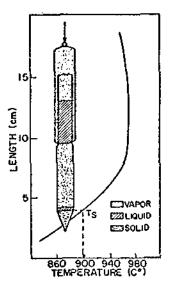


Fig. 2 - Vertical VMS growth.

Preliminary results have shown that with the vertical assembly it is possible to carry out a VMS growth twice as fast as compared to the horizontal arrangement.

A typical result for the mean axial concentration profile in the final crystal is shown in fig. 3, compared to a Bridgman growth with similar conditions. The success of the VMS technique in maintaining a DCSS growth in such a low growth rate is a clear indication of the high quality crystals so obtained. Microprobe measurements to map out the detailed radial concentration profile, as well as microphotographs and chemical etching to visualize defects and grain boundaries are under way.

† - The actual liquid layer thickness is difficult to measure, but is an essential parameter for the homogeneity of the final crystal. See Section 4.

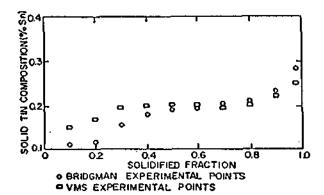


Fig. 3 - Axial concentration profile for Bridgman and VMS growth. The fournace translation rate is  $2 \times 10^{-5}$  cm/s. Crystal lenght is 4cm.

In order to hold the molten material inside the liquid reservoir in low gravity, a closed ampoule with a certain number of small holes in it was enviaseged. We are currently carrying out tests in horizontal quartz ampoules with holes about 1mm² of area each. For melt colums of 0.5cm (F=30dyn on Earth) the system worked very well, without any sign of leakage. Taken 30dyn as the limit for 1mm² holes it seems to be possible to hold typical PbSnTe melts in space even for high G-jitters.

# 3. DCSS Bridgman growth of Pbi-xSnxTe

The basic features of a DCSS growth is present in the simple Bridgman configuration sketched in flg. 4. The solidification from the melt with a given constant interface velocity  $R = dx^2/dt$  in the pure diffusive regime gives a concentration profile that, in the limit of large L, and for constant interfacial segregation, is given by the Smith, Tiller and Rutter (STR) theory  $^9$ .

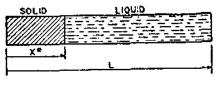


Fig. 4

For finite values of L, the desired steady-state regime is only achieved for large values of the growth velocity R (such that D/R << L, where D is the diffusivity of liquid SnTe in liquid PbTe). In the limit of large D, the liquid is almost completely mixed and the final concentration profile in the solid will follow the normal freezing curve (Pffan's regime) the specially devised to avoid convection in the melt show that, for a 5cm mglt height, steady-state is achieved for R > 5mm/h. The value of the mutual diffusion constant D of SnTe in PbTe is estimated to be  $5 \times 10^{-5} \text{cm}^2/\text{s}$ .

To compare DCSS growth on Earth with those in Space, it is necessary to rely in a more accurate calculation than the STR theory. Corrections due to differences between the growth rate and the fournace translation rate, the dependence of the equilibrium segregation factor on the concentration at the interface, and corrections due to the finiteness of the melt column must be taken into account. Rather than pursuing an approximated analytical approach to incorporate those corrections in the framework of the STR theory, we decided to solve numerically the free-boundary diffusion equation that governs the concentration field  $\mathbb{C}(x,t)$  of SnTe in the liquid:

$$D \frac{\partial^2 C}{\partial x^2} = \frac{\partial C}{\partial t}$$
 (1a)

$$\frac{\partial C}{\partial x} (x = L, t) = 0$$
 (1b)

$$\frac{\partial C}{\partial x} (x = x^{\pm}, t) = -qRC(x = x^{\pm}, t)$$
 (1c)

$$C(x,0) = C_0 \tag{1d}$$

$$T_{i}[C(x^{*},t)] = T_{p}(x^{*},t)$$
 (1e)

Eq. (1a) is the normal diffusion equation for a fixed diffusion constant D, in a reference frame attached to the ampoule. In (1b) we impose no net flux at x=L. (1c) is the flux condition at the interface at  $x=x^{\pm}$ , where the segregation factor k=1-q can be concentration-dependent and  $R=dx^{\pm}/dt$ , the interface velocity, is time-dependent. The interface  $x^{\pm}$  is supposed initially at the ampoule tip, and the melt is homogeneous at t=0 with a Sn molar fraction  $C_{\rm O}$ .

The location of the interface,  $x^*(t)$ , is given by the equation (1e), where  $T_F$  is the fournace temperature profile (that is moving with a constant velocity in the reference frame where eqs.1 are written) and  $T_{\parallel}$  (C) is the liquidus curve taken from the phase diagram of the PbTe-SnTe system. That is to say, the interface is in a position such that its temperature, imposed by the fournace profile, agrees with the predicted equilibrium temperature from the phase diagram. We are thus assuming that the growth rate is sufficiently small to be described by a sucession of quasi-equilibrium steps. This approach was also employed by Clayton et al<sup>12</sup> for the study of a Bridgman growth of HgCdTe; they devised a Taylor expansion to solve eqs.1 by finite differences, and considered the case of a constant q. Since their algorythm showed some instabilities in the final transient of the growth, we adopted a totally implicit second-order in space first-orde in time finite difference scheme. The proof that such an algorythm is unconditionally stable for the linearized form of eqs.1 will be published elsewhere.

The equilibrium segregation factor k = 1-q is also taken from the PbTe-SnTe phase diagram. Relations (1c) and (1e) introduce smooth nonlinearities, and the actual location of the interface is found by a simple bissecsion technique.

The quantity of relevance here is  $C_s(x) = kC(x^k)$ , that gives the axial solid concentration profile. A plot of  $C_s(x)$  obtained for a 5cm length crystal

is given in fig. 5 for V=1mm/h and fig. 6 for V=10mm/h, where V is the fournace translation rate (constant). The experimental points are taken from ref. 5. The diffusion constant, the only unknown parameter in these simulations, was adjusted by least squares to fit the experimental data, and we found  $D=(4.9 \pm 2.1) \times 10^{-5} \text{cm}^2/\text{s}$  for V=1mm/h and  $D=(11.1 \pm 3.1) \times 10^{-5} \text{cm}^2/\text{s}$  for V=10mm/h. A set of carefull controlled experiments is being devised at our laboratories to clarify that discrepancy, wich can be due to residual convection or to quenching in the final region of the growth. The interface velocity profile is also shown in figs. 5 and 6. We see that it is not true, as is frequently claimed, that the difference between the interface velocity and the fournace translation speed is very small in this system due to the proximity between the solidus and liquidus lines in the PbTe-SnTe phase diagram: this fact only means that the corrections in the axial concentration profile are small, and that this is indeed the case is also indicated in figs. 5 and 6.

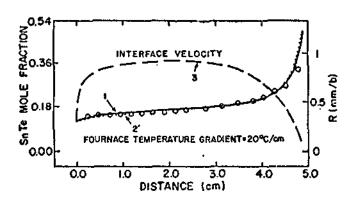


Fig. 5 - Axial concentration profile for a Bridgman diffusive growth. The fournace translation rate is V=lmm/h. Curve 1 is obtained by solving the diffusion equation when the speed of advance of the interface is constant (=V). Curve 2 takes into account the actual interface velocity profile shown in curve 3. The experimental points (o) are from ref. 5.

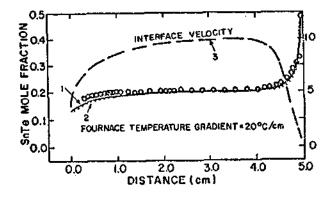


Fig. 6 - Same as fig. 5, when the fournace translation rate is V=10mm/h.

For space-based experiments, the growth time must be carefully controlled. In fig. 7 we show the growth profile in time. From these curves we see that, to achieve a given fraction of the complete growth, it may be necessary far more than that same fraction of the expected time L/V. For instance, if we are to grow a Bridgman crystal with a melt column of 5cm at V=1mm/h, we need 50 hours to grow 4.5cm if a fournace gradient of 409C/cm is employed; with a fournace gradient of 209C/cm, to achieve a 4.75cm crystal we need 63 hours. If not carefully designed, the growth will not take place in a region of the fournace with constant temperature gradient, and the final region of the crystal can be solidified at a very distinct regime from that of the bulk crystal.

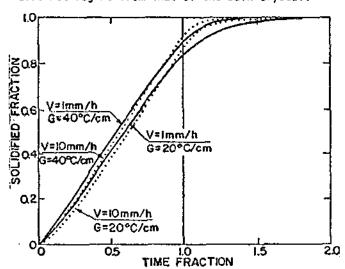
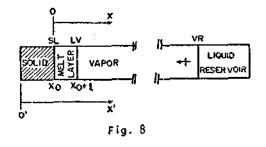


Fig. 7 - SOLIDIFIED FRACTION  $x^4/L$  versus TIME FRACTION t/(L/V). V is the fournace translation rate and G is the thermal gradient at the interface.

## 4. DCSS growth regime in VMS

Concentration homogeneity in a Bridgman growth crystal requires supression of convective currents in the melt and high growth velocities to achieve a steady-state diffusive regime within the finite length of the crystallization column. We will now show that, with the VMS technique, lower growth velocities are allowed to attain the DCSS regime, and convection currents are inhibited (in the melt, because of its small Rayleigh number, and may be of minor importance in the vapor because of the predominant streaming transport effect).

Consider a scheme of a VMS assembly, depicited in fig. 8.



Vapor is continuouslly generated by the liquid reservoir and condensed in the liquid layer. We define Y as being the volume contraction in the condensation process (or expansion during evaporation) at a given pressure and temperature:

$$\gamma = \frac{\text{molar volume of vapor}}{\text{molar volume of liquid}}$$
 (2)

For simplicity, we assume  $\gamma$  constant and homogeneus during the whole process: datailed profiles for y can be introduced without difficulties in the general equation derived below. We also take a constant width & for the liquid layer and neglect diffusion in the solid and density differences between the solid and liquid.

In the stationary frame 0'x' the differential equation governing diffusion of SnTe is given by 14:

$$\frac{\partial}{\partial t} (\mu C) + \nabla \cdot (\iota C \vec{v}) = \nabla \cdot (\mu D \nabla C)$$
 (3)

, where

- molar concentration of the mixture (PbTe+\$nTe)

= molar fraction of \$nTe

- mutual diffusion coefficient

⇒ molar average velocity = Cva+dvb= -vi

d = molar fraction of PbTe = 0  $v_a = mean velocity of SnTe molecules in the$ 0'x' frame

 $\vec{v}_b$  = mean velocity of PbTe molecules in the 0'x' frame

We now suppose that the drift flows of PbTe and SnTe are equal, and therefore  $\vec{v}_{a} = \vec{v}_b = \vec{v}$ , since d+C=1; this assumption being not rigorous because of the different molecular masses of PbTe and SnTe. Expressing eq. 3 in the moving frame 0x, for constant-velocity moving interfaces and stationary µ, v and D profiles we obtain

$$\mu D \frac{\partial^2 C}{\partial x^2} + \left[ \frac{d(\mu D)}{dx} + \mu (R + v) \right] \frac{\partial C}{\partial x} + \left[ \frac{d(\mu v)}{dx} + R \frac{d\mu}{dx} \right] C = \mu \frac{\partial C}{\partial x}$$
(4)

, where R is the speed of the moving fronts.

The last term in the l.h.s. of eq. 4 can be shown to be zero by application of the continuity equation to stationary profiles  $\mu(x)$ ,  $\nu(x)$  and D(x) in the moving frame.

The main features of VMS can now be deduced from eq. 4 taking the case of homogeneus media:

- in the liquid layer and the liquid reservoir, take D=D<sub>L</sub>=constant, u=constant, v=0. We obtain for these regions the well known diffusion equation of the STR theory:

$$D_{L} \frac{\partial^{2} C}{\partial x^{2}} + R \frac{\partial C}{\partial x} = \frac{\partial C}{\partial t}$$
 (5)

- in the vapor region, take  $\gamma$  =constant, D=D $_{
m v}$ = constant,  $\mu = constant$ ,  $v = (\gamma - 1)R = constant$ :

$$Ov \frac{\partial^2 C}{\partial x^2} + \gamma R \frac{\partial C}{\partial x} = \frac{\partial C}{\partial t}$$
 (6)

Eq. 5 has a characteristic diffusion length  $L_{\perp}D_{\perp}/R$ , and eq. 6 has  $L_{\nu}=D_{\nu}/(\gamma R)$ . We see that,

for large values of  $\gamma$ , the diffusion length is reduced by the effect of the drift flow (streaming), and this is wy VMS allows for low growth rates compatible with a steady-state diffusive regime. For the case of an infinite liquid reservoir, loaded initially with a constant molar fraction  $C_{\rm O}$  of SnTe, it is easy to obtain the steady-state profiles and check that the SnTe molar fraction in the solid will indeed be Co after the initial transient.

To study the dependence of the axial concentration profile on  $\gamma$  and  $\ell$ (the liquid layer thickness) for the actual case of a finite reservoir, we solved eq. 4 by a totally implicit moving-boundary finite-difference (FD) scheme. The FD equations were obtained by a control-volume approach 15, wich properly takes into account the discontinuities introduced by the D,  $\nu$  and  $\mu$  profiles.

Fig. 9 shows a typical result from those simulations, for a growth velocity R=0.25mm/h and a Pbo.s Sno.2 Te charge. The mutual diffusion constant in the liquid is taken  $D_L = 5 \times 10^{-5} \text{cm}^2/\text{s}$  in all cases, and the equilibrium segregation constant k is 0.65. The length of the vapor column is 10cm, and the thickness of the liquid layer and the liquid reservoir are adjusted to yeld a 3cm-length crystal. Those were the parameters employed in the experimental setting of ref. 16.

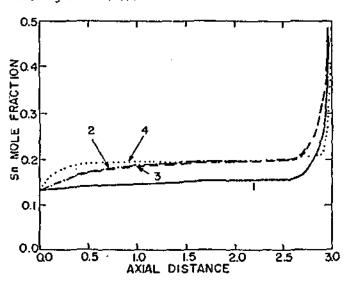


Fig. 9 - Predicted profiles for the axial concentration distribution in a crystal grown by VMS under various conditions. See text for discussion.

Curve 1 in fig. 9 shows the case where  $D_V=D_L$ and Y=1, thus resembling a Bridgman growth; expected, at such low growth rate all the fluid media is homogeneusly mixed by diffusion and consequently a normal freezing profile is obtained In curve 2 we maintain Dy=D1 but use  $\gamma=200$  , for a layer thickness £=3.5nm; we see that a steadystate at C=0.2 is attained for L>1.5cm, Increasind D, to  $5\times10^{-3}$  (=1000,), a value wich is certainly closer to the actual diffusion constant in the vapor, we obtain curve 3, showing clearly that the main influence on the attainement of the steady-state is through the vapor transport rate

 $\gamma$ , the exact axial profile being not sensible to  $D_{\gamma}$  in this range of parameters. An effect that is not easily seen in the model equation is the sensitiveness of the axial profile with respect to the layer thickness  $\mathcal{L}$ . In curve 4,  $\mathcal{L}$  is reduced to 1.0nm while maintaining all other parameters fixed ( $D_{\gamma}=100D_{\gamma}$ ,  $\gamma=200$ ); we now obtain a steady state profile for L>1.0, a high-quality growth that is more in accordance with the experimental findings 3 6 16.

More accurate profiles for  $\gamma$ ,  $0,\mu$  and  $\nu$  can be introduced in eq. 4 and solved by our FD algorythm in an easy manner, but those calculations cannot be reinforced until we have more accurate techniques for the growth visualization and crystal characterization.

## 5. Conclusion

It is apparent from the above results that a VMS growth in space would benefit from mechanical stabilization of the liquid layer, supression of convective currents in the vapor phase (other than streaming) and, in a speculative fashion, an increase in the laminar unidirectional vapor~transport rates.

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