

3.2 - LEAD-TIN-TELLURIDE CRYSTAL GROWTH BY VMS METHOD IN MICROGRAVITY

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Abstract: Large homogeneous ternary alloy single crystals are a must for high performance optoelectronic devices such as diode lasers and infrared detector arrays. One practical choice for infrared detectors and semiconductor lasers in the wavelength range of 5 to 12 μm is the narrow band-gap semiconductor $\text{Pb}_{1-x}\text{Sn}_x\text{Te}$.

It is well known that, if $D / v < L$ (where D is the diffusion coefficient, v the growth rate and L the crystal length), Bridgman growth in low gravity can result in crystals with more homogeneous axial concentration. This is due to the shift from convective growth on Earth to diffusion controlled steady-state (DCSS) growth in space. The only way to achieve DCSS growth on Earth is by reducing the growth aspect ratio (liquid height / ampoule diameter). In Bridgman growths it is possible to sustain a DCSS growth for a 50 mm length crystal if the bore of the growth ampoule is reduced to 2 mm. Another Earth bound method to avoid convection 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 vapor¹. Such liquid layer, ahead of the crystallization front, has a thickness of the order of millimeters instead of the centimeters typical of Bridgman growth, reducing the Raleigh and Grashof number below critical values for vertical or horizontal growth respectively.

While it is possible to find a method to eliminate buoyancy driven convection on Earth, very little can be done about surface-tension convection (Marangoni flow), which can give rise to axial and radial concentration inhomogeneities.

When analyzing the ratio of Raleigh to Marangoni numbers (Bond number) in Bridgman growth in space, it is seen that even taking into account only the thermal gradient normal to the free surface, the surface tension driven convection is still an important factor for segregation. The same does not apply for VMS, where the Bond number is about 70 times smaller than that for Bridgman. To grow a crystal by VMS in microgravity conditions will be an excellent way to evaluate the mechanisms that give rise to radial and axial concentration inhomogeneities on melt grown crystals^{2,3}.

Other possible advantages of VMS method in space are:

1. A constant aspect ratio during DCSS is maintained because the melt layer formed over the solid always has the same height. In Bridgman growth the melt height decreases.
2. Starting of solidification at the tip of the ampoule, avoiding the growth of bicrystals. To test this, some VMS growths have been made at INPE, in a vertically destabilized

configuration (melt below the crystal). At the start the melt condenses in to a very thin liquid layer that hangs in the tip of the ampoule even in the upside down configuration. In the above experiment it was noted that the crystal perfectly follows the container walls. In Bridgman growth a sort of containerless growth can develop in microgravity which, although favoring stress free crystallization, can enhance surface tension driven flows.

The surface tension between the melt and the solid is enough to hold the thin melt layer in place at all times. In Bridgman growth there is a possibility of lack of contact between the melt and the solid at the interface in some stages of the flight, due to the free space left for volume expansion when the charge changes from solid to liquid during the melting.



a



b



c

Pictures of a PbSnTe VMS upside down growth. Picture a shows the ampoule and charge before growth; picture b shows an interrupted growth with the partially grown crystal at the tip of the ampoule and the remainder of the melt in the reservoir; picture c shows a fully grown crystal.

3. Due to the thin melt layer, a stagnant diffusion layer is created by geometrical factors. For low speed growth and high diffusion coefficients, the possibility of complete mixing for Bridgman growth in microgravity cannot be disregarded.

As far as we know, the VMS method has not yet been used in space. Although DCSS growth is achieved in Bridgman growths in low-gravity, the favorable geometry of VMS growth tends to minimize the convective mixing, allowing a better understanding of axial and radial concentration profiles.

The following characterization techniques are available at INPE:

- Electron probe microanalysis to check axial and radial solute concentrations
- Hall and capacitance measurements at low temperatures
- X-rays analysis (Laue, diffractometry and rocking curves)
- Electronic and optical microscopies to study morphological details
- Optical spectroscopic measurements (PL, Brillouin, Raman, etc.)

Furthermore, hydrodynamic theoretical models can be developed. Photovoltaic detectors can be made on the space grown materials at INPE by Molecular Beam Epitaxy (MBE), and their figures of merit compared with those of detectors made with Earth grown substrates.

In conclusion, our proposal to grow a $Pb_{1-x}Sn_xTe$ crystal by VMS technique in low-g seeks the understanding of radial and axial concentration inhomogeneities in melt grown crystals due to the residual buoyancy convection and the gravity-independent Marangoni convection. We also hope that more can be learned about vapor transport mechanisms in low-g.

PRELIMINARY CHARACTERISTICS FOR THE VMS GROWTH IN LOW-g

01. Material: $(Pb_{1-x}Sn_x)_{1-y}Te_y$ for $x = 0.20$ and $y = 0.50$
02. Ampoule size: overall length = 80 mm; diameter = 10 mm
03. Ampoule material: electronic grade SiO_2
04. Internal ampoule pressure at room temperature: $< 10^{-6}$ torr
05. Internal ampoule pressure at growth temperature: < 230 torr (< 0.3 atm)
06. Isothermal region temperature: $950^\circ C \pm 10^\circ C$ in 10 cm
07. Solidification temperature: $896^\circ C$
08. Temperature gradient: $\approx 20^\circ C/cm$
09. Growth rate: $\approx 1 \times 10^{-4}$ cm/s
10. After growth rate: $\approx 4^\circ C/min$ until $500^\circ C$
11. Growth time: ≈ 10 hours
12. Temperature stability: optimum $\pm 0.1^\circ C$; acceptable $\pm 1^\circ C$
13. Safety precautions: quartz ampoule enclosed in sealed stainless steel cartridge (length = 120 mm; diameter = 15 mm). The internal pressure is low and even in the case of ampoule breakage, the toxic lead and tellurium vapors at high temperatures will not be harmful because PbTe does not dissociate and will condense on surfaces with temperatures around $500^\circ C$.

References:

- 1 - C. Y. An e I. N. Bandeira: Crescimento de monocristais de $Pb_{1-x}Sn_xTe$ por transporte de fase vapor, com formação de uma interface de crescimento líquido/sólido. Revista de Física Aplicada e Instrumentação, 1, 52, 1985.
- 2 - M. Fabbri, I. N. Bandeira and L. C. M. Miranda: A model for mass transfer in VMS crystal growth, J. Crystal Growth 104, 435, 1990.
- 3 - L.L. Regel, A. Y. Oleinikov, A. M. Turchaminov, O. V. Shumaev, I. N. Bandeira, C. Y. An and P. H. O. Rapp: Growth of PbSnTe crystals in high-gravity, J. of Crystal Growth, 119, 94, 1992.

3.3 - PROPOSTA PARA IMAGEAMENTO DE EMISSÕES ATMOSFÉRICAS A PARTIR DA ISS

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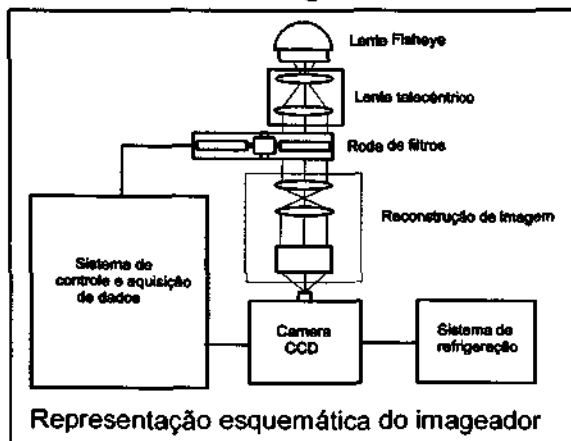
Finalidade: O projeto visa o imageamento das emissões luminescentes provenientes de reações fotoquímicas na região da mesopausa e da baixa termosfera (70-120 km).

O instrumento a ser embarcado no ISS (em palete), é uma câmara CCD com filtros e sistema óptico para registrar imagens das distribuições espaciais das emissões atmosféricas de hidroxila, oxigênio e de sódio. Os resultados destas observações serão de grande utilidade nos estudos da dinâmica e fotoquímica da alta atmosfera.

Os resultados das observações serão utilizadas em duas linhas de pesquisas:



1. Ondas internas de gravidade.



Ondas internas de gravidade são ondas atmosféricas com períodos maiores que ~ 4 minutos, onde a força de gravidade é importante.

Comprimentos de onda horizontais ~ 1 a >1000 km.

Fontes apenas parcialmente conhecidas: efeitos orográficas (interação ventos - montanhas); grandes tempestades; correntes elétricas na ionosfera.

Atingem amplitudes máximas em alturas ~ 100 km.

Dissipação de energia em ~ 100 km desempenha papel importante na balança térmica da atmosfera.

2. Camadas neutras esporádicas

Camadas esporádicas ionizadas, em alturas ~100 km, são conhecidas desde os anos 30, através dos seus efeitos sobre radiopropagação.

Camadas esporádicas neutras, compostas de Na, K, Li, Fe, foram descobertas em 1978 pelo radar de laser do INPE.

Apesar de estudos extensivos no Brasil, EE. UU., Alemanha e Japão, ainda não existe certeza sobre a origem destas camadas.

Imageamento das emissões produzidas pela interação dos átomos de sódio em camadas esporádicas e ozônio atmosférico ajudará a determinar o mecanismo responsável. A emissão resulta da cadeia de reações:

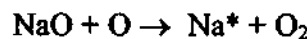
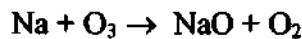


Imagem do céu na linha de emissão de hidroxila

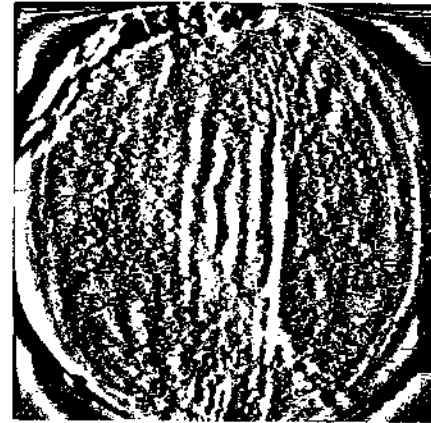
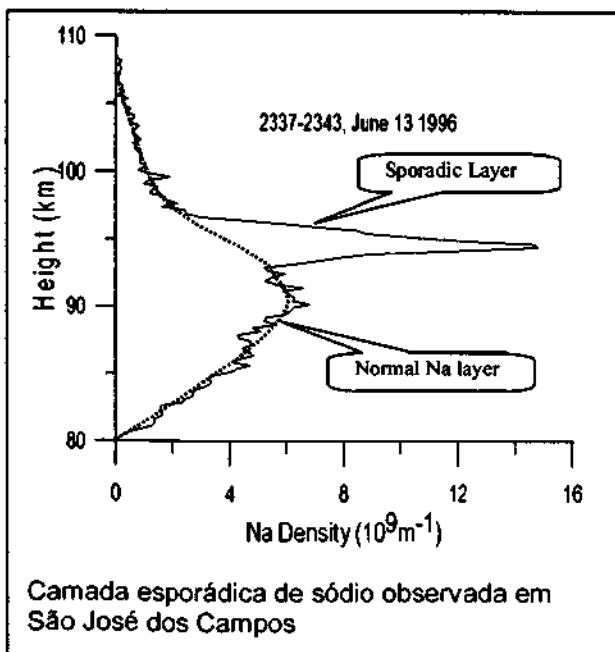


Imagem processada, mostrando presença de ondas de gravidade.



Uma das principais hipóteses sugeridas para explicar a formação das camadas esporádicas envolve a concentração de íons metálicos pelo cisalhamento dos ventos associados com ondas atmosféricas. Espera-se que a observação simultânea da existência de ondas e a formação de camadas esporádicas possa ajudar a esclarecer o mecanismo da sua formação.

3.4 - STATUS OF THE FIRE PROJECT

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Abstract - 'The Fire InfraRed Experiment (FIRE) will make the first observations of infrared synchrotron emission (at 35 and 150 microns) from high energy processes in solar flares. FIRE will be an instrument of the French-Brazilian Microsatellite (FBMS), scheduled to be launched earlier in 2002. FIRE excellent sensitivity and high time resolution (33 milliseconds) make it extremely complementary to the recently installed SST ground-based solar submillimeter telescope, and to the next high energy HESSI mission. The FBMS mission will provide the spacecraft and a launch by Brazilian VLS rocket (with "piggyback" on Ariane-5 as an alternative). FIRE is a joint project from CRAAE (joint center between Mackenzie, INPE, USP, UNICAMP), INPE, LEP/GSFC/NASA and Observatoire de Paris.