



**Crystal Growth of Ternary Compound
Semiconductors in Low Gravity Environment**

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Crystal Growth of Ternary Compound Semiconductors

A low gravity material science experiment has been prepared to be performed in the Low Gradient Furnace (LGF) in the Material Science Research Rack (MSRR) on International Space Station (ISS).

There are two sections of the flight experiment:

- (I) Investigation toward crystal growth by physical vapor transport (PVT): the growth of ZnSe and related ternary compounds, such as ZnSeS, ZnCdSe, and ZnSeTe,
- (II) Investigation on the melt growth of CdTe and CdZnTe by directional solidification.

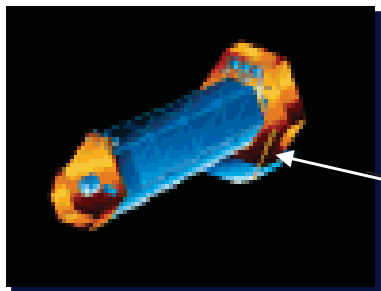


Crystal Growth Activities at NASA/MSFC

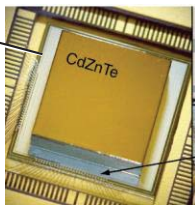
Technological significance:

Growth (melt and vapor) and characterization of II-VI and IV-VI compounds semiconducting materials, such as HgCdTe, HgZnTe (for IR detectors), CdS and ZnO (for UV detector), ZnSe, ZnSeTe (for green /blue laser), CdTe and CdZnTe (for x-ray, gamma ray detectors), PbTe, PbTeSe, and PbSnTe (thermoelectrics).

II	IV	VI
Zn	Sn	O
Cd	Pb	S
Hg		Se
		Te



**CdZnTe
FPA**



**X and gamma
ray telescope**

Contributors:

MSFC personnel:

- Dr. Sharon Cobb
- Dr. Donald Gillies
- Dr. Sandor Lehoczky
- Dr. Ching-Hua Su
- Dr. Martin Volz
- Dr. Dale Watring
- Dr. Frank Szofran

On-site contractors:

- Dr. Shari Feth
- Dr. Chao Li
- Dr. Konstantine Mazuruk
- Dr. N. Ramachandran
- Dr. Witold Palosz
- Dr. Yigao Sha
- Dr. Shen Zhu



II-VI semiconducting compounds grown at MSFC

Compounds	HgTe	HgCdTe	CdTe	CdZnTe	ZnTe	CdS	ZnSe	ZnS
Melting points (°C)	670	700	1092	1130	1292	1397	1526	1718
Melt growth								
PVT growth temperature (°C)			850		1000	985	1120	1150

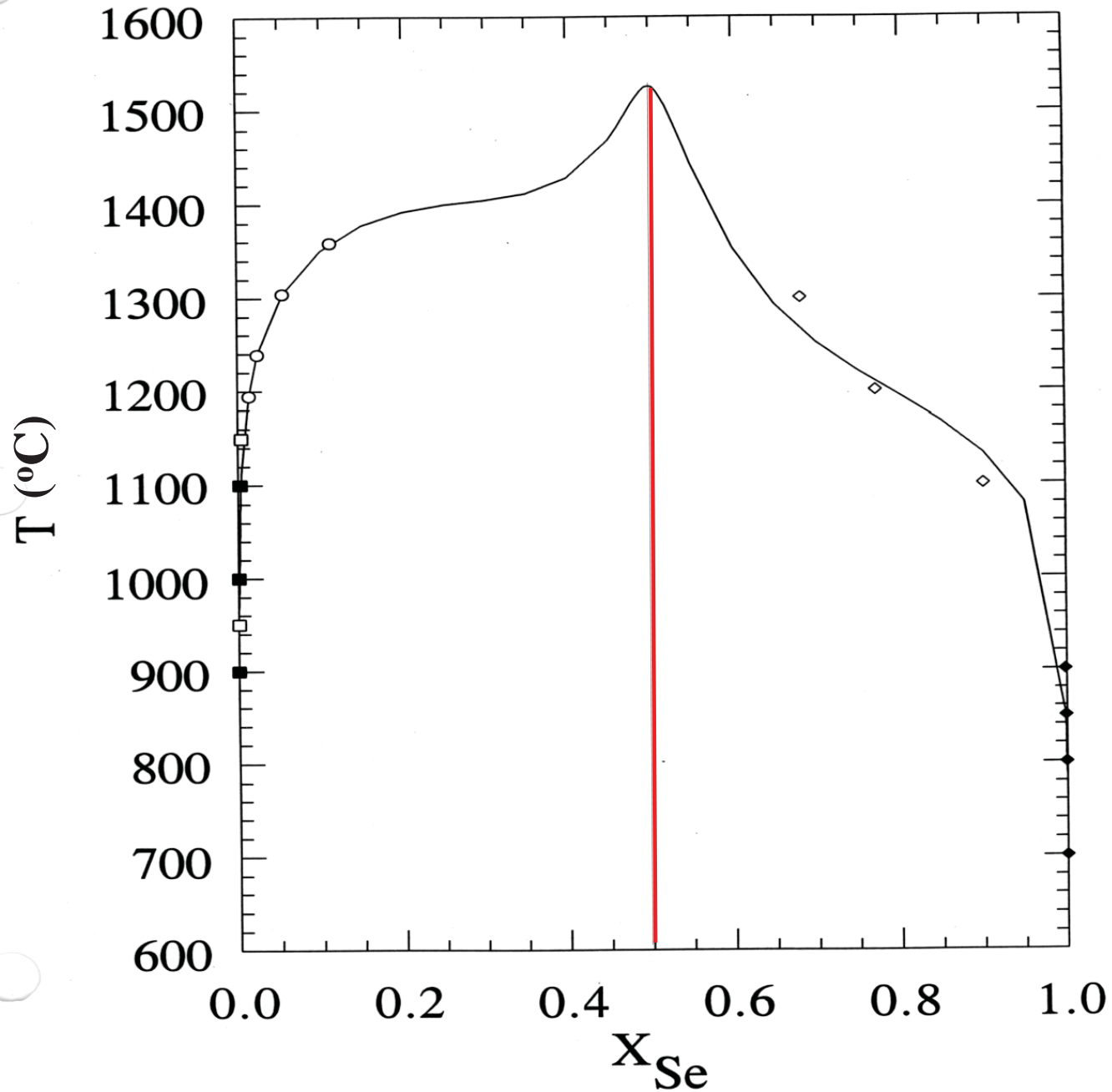


Scientific Objectives

1. To establish the relative contributions of gravity-driven fluid flows, both in liquid and vapor, to (1) the non-uniform incorporation of impurities and defects and (2) the deviation from stoichiometry and (3) the compositional variation observed in the grown crystals.
2. To assess the self-induced strain developed during processing at elevated temperatures and retained on cooling caused by the weight of the crystals.
3. The relation between fluid phase processes and the generation of defects in a grown crystal is an outstanding problem in materials growth. Studies in microgravity will be compared with modeling and will lead to a greater understanding of the processes involved.



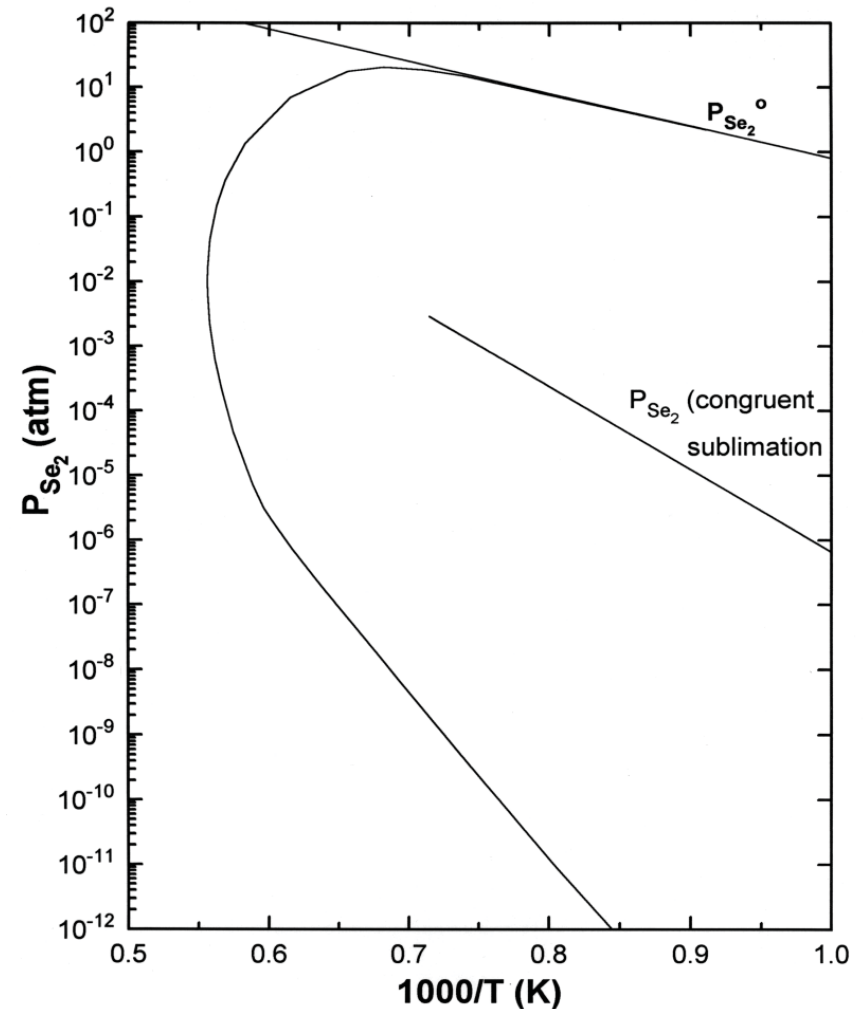
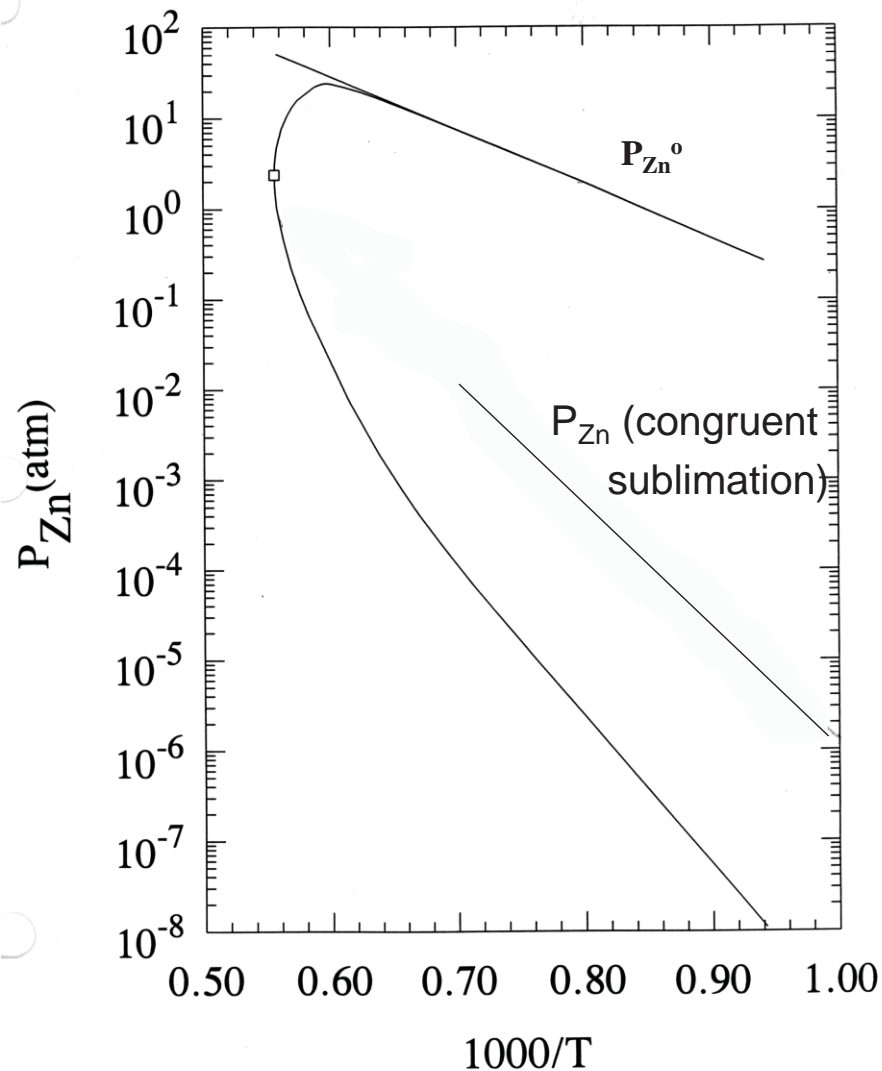
Phase Diagram of Zn-Se





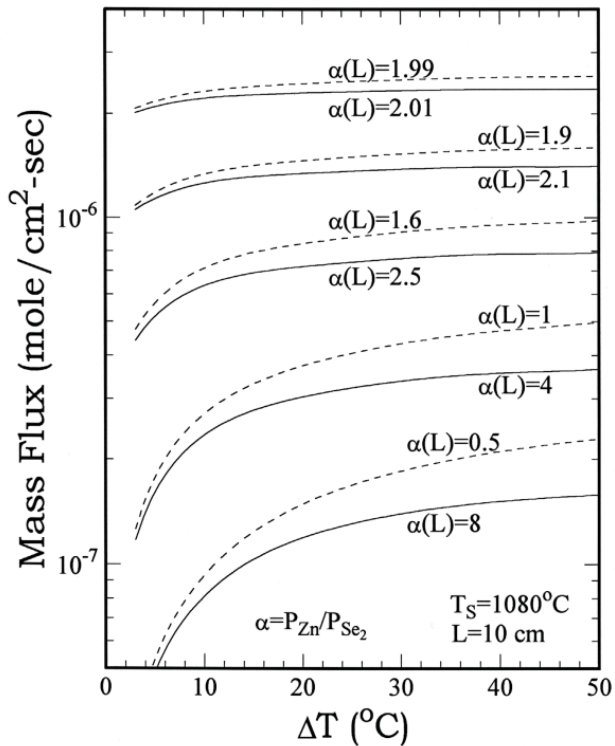
Partial pressures along the three-phase curve for ZnSe(s)

At 1156 °C, P_{Zn} varies from 8.4 to 1.3×10^{-4} atm, P_{Se_2} varies from 20 to 5×10^{-9} atm and $\alpha = P_{\text{Zn}} / P_{\text{Se}_2}$ varies from 1.7×10^9 to 6.5×10^{-6}

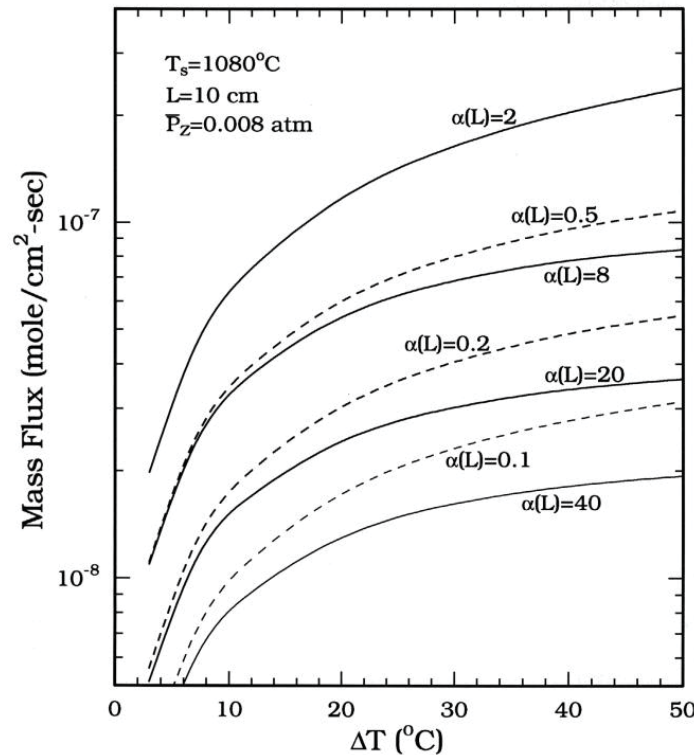




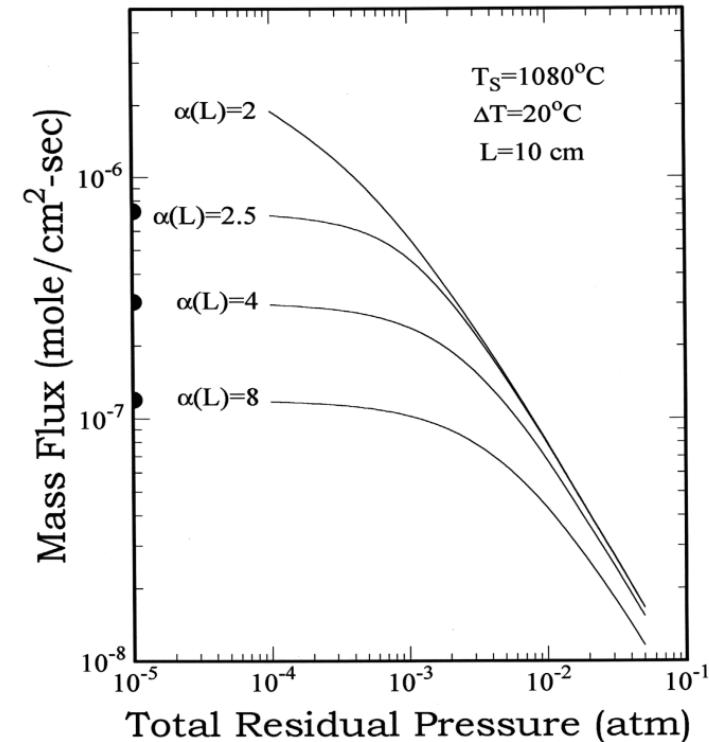
One-Dimensional Diffusion model of PVT



Calculated mass flux of ZnSe as a function of ΔT and different values of $\alpha(L)$. The source temperature was 1080°C. Solid lines are for $\alpha(L) > 2$ and dashed lines are for $\alpha(L) < 2$.



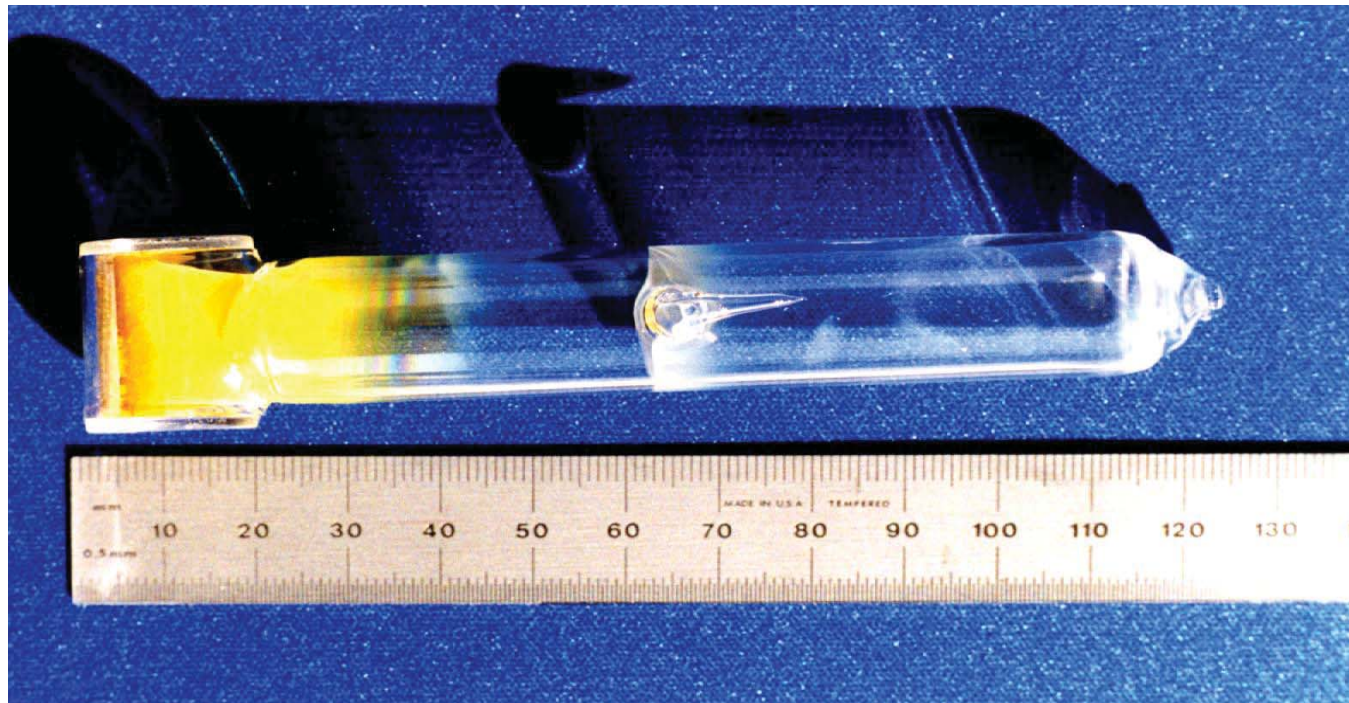
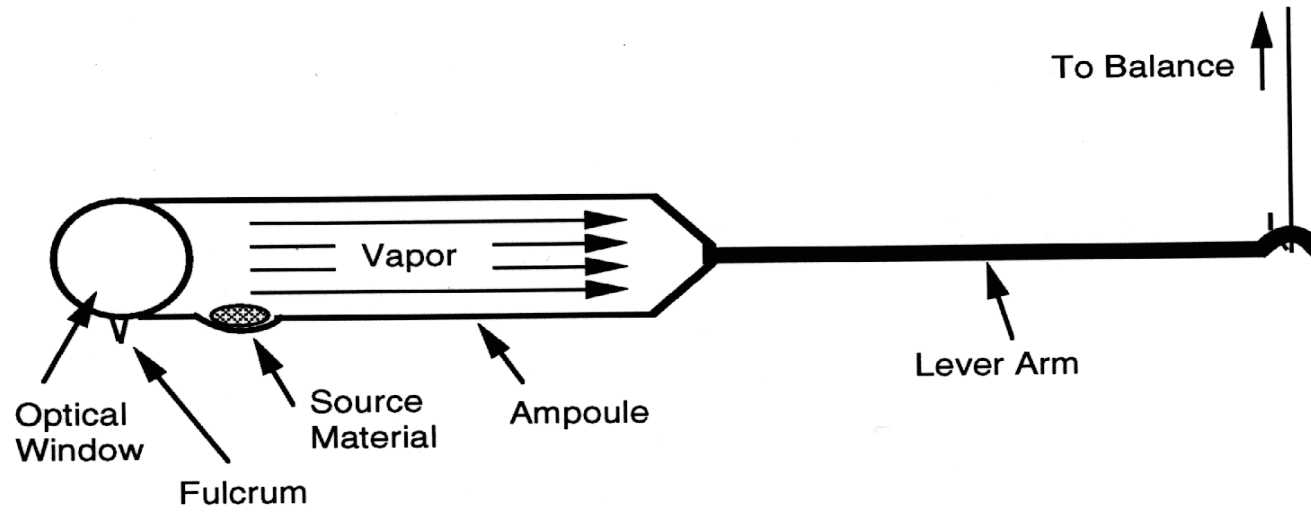
Calculated mass flux of ZnSe as a function of ΔT under the same conditions except a residual gas pressure of 0.008 atm is present in the system.



Calculated mass flux of ZnSe as a function of residual gas pressure for source temperature at 1080°C and different values of $\alpha(L)$.



Simultaneous measurements of mass flux and partial pressure





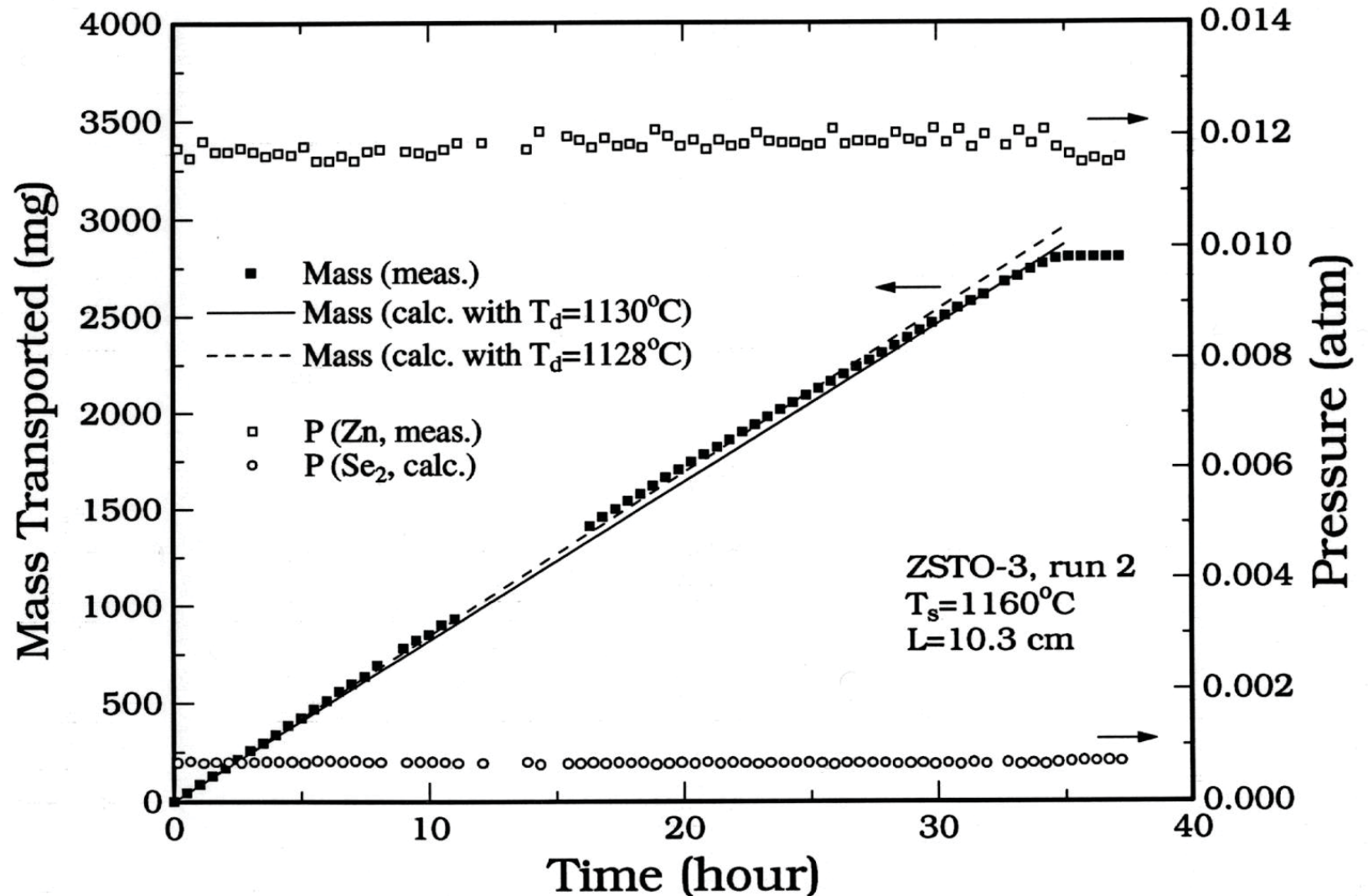
Simultaneous measurements of mass flux and partial pressure





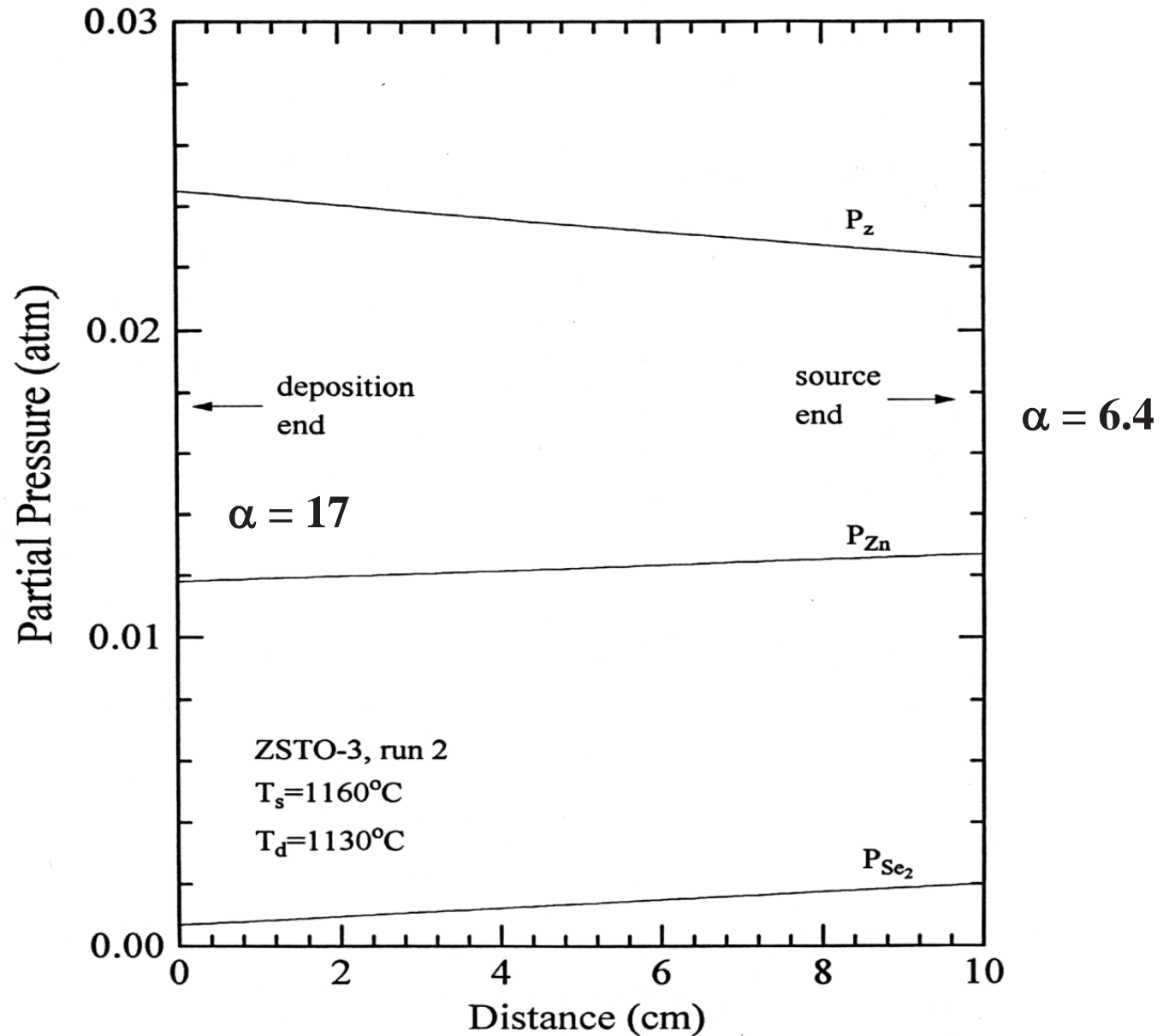
Simultaneous measurements of mass flux and partial pressure

- P_{Zn} measured over the deposit; α (deposit) = 17.
- The measured P_Z value was used in the calculation.





Calculated partial pressure profiles for ZSTO-3 run 2





Summary of one dimensional diffusion analysis

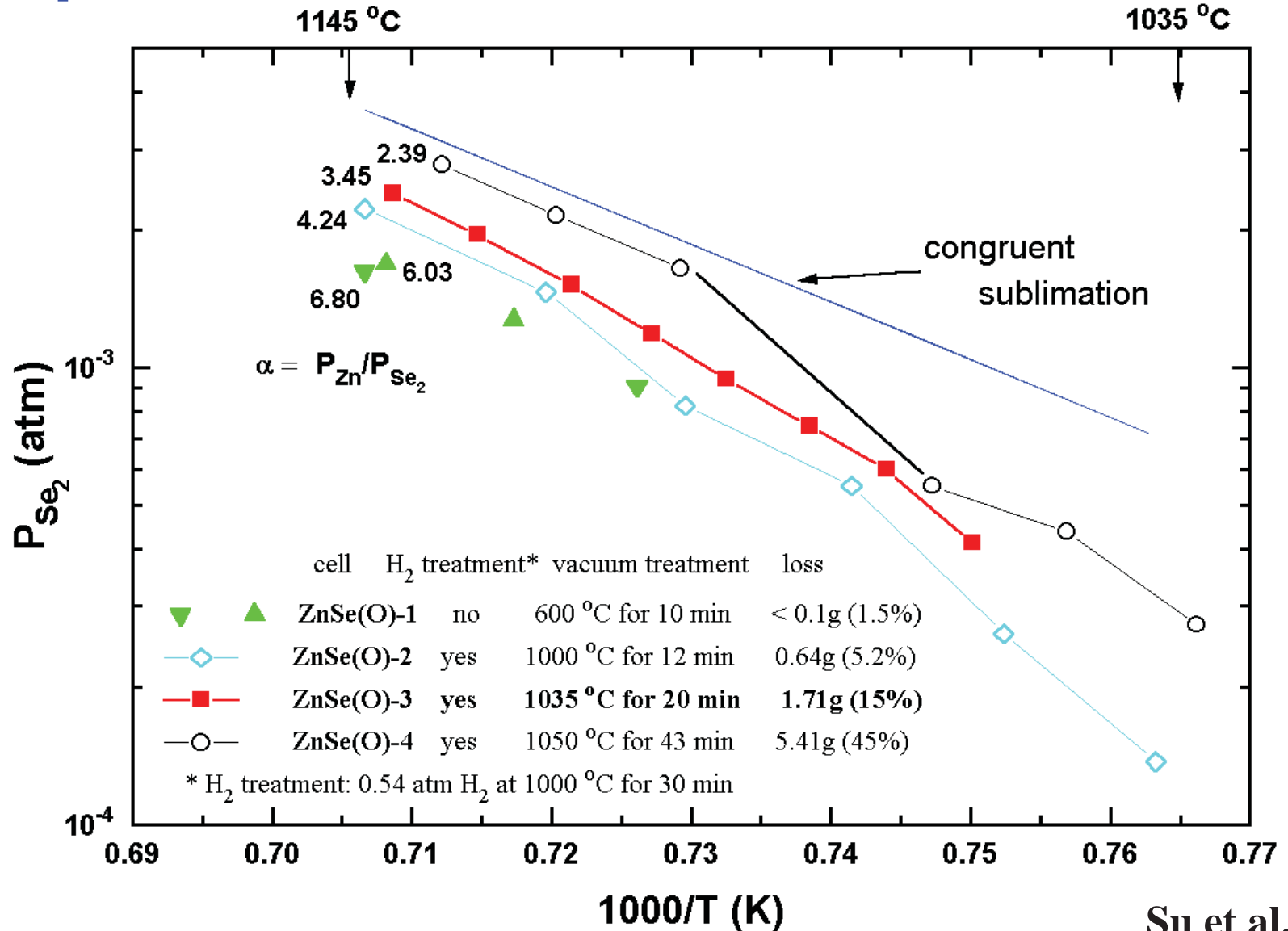
- Four experimentally adjustable parameters, **the source temperature, the deposition temperature, the partial pressure ratio over source and the residual gas pressure**, determine the diffusive mass flux of a PVT system.
- However, two of these four parameters, the **partial pressure ratio over source and the residual gas pressure**, are more critical than the others. As will be shown, these two parameters are critically dependent on the proper **heat treatments** of the starting materials for optimum mass flux.



Optimum Heat Treatments of Starting Materials

P_{Se_2} in equilibrium with ZnSe for various optical cells

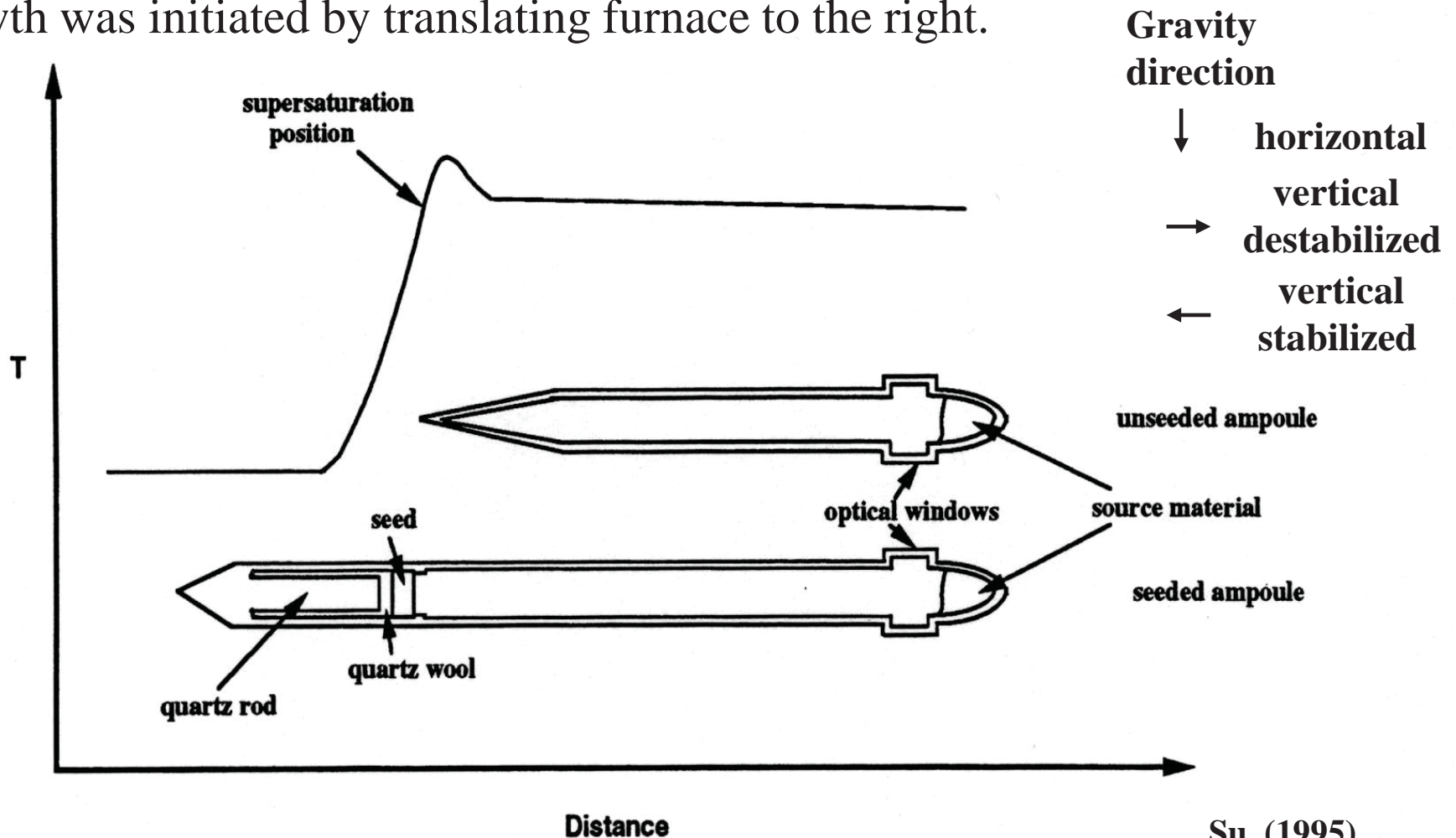
H_2 reduction and vacuum treatments for ZnSe(O)-3 established to be the optimum procedure





Crystal Growth by Physical Vapor Transport: temperature profile and initial ampoule positions

- The growth ampoules can be equipped with optical windows to confirm the stoichiometry of the starting material before growth.
- The thermal profile, with a maximum in the middle, was provided by a three-zone furnace with an adiabatic zone between central and cold zones.
- Growth was initiated by translating furnace to the right.



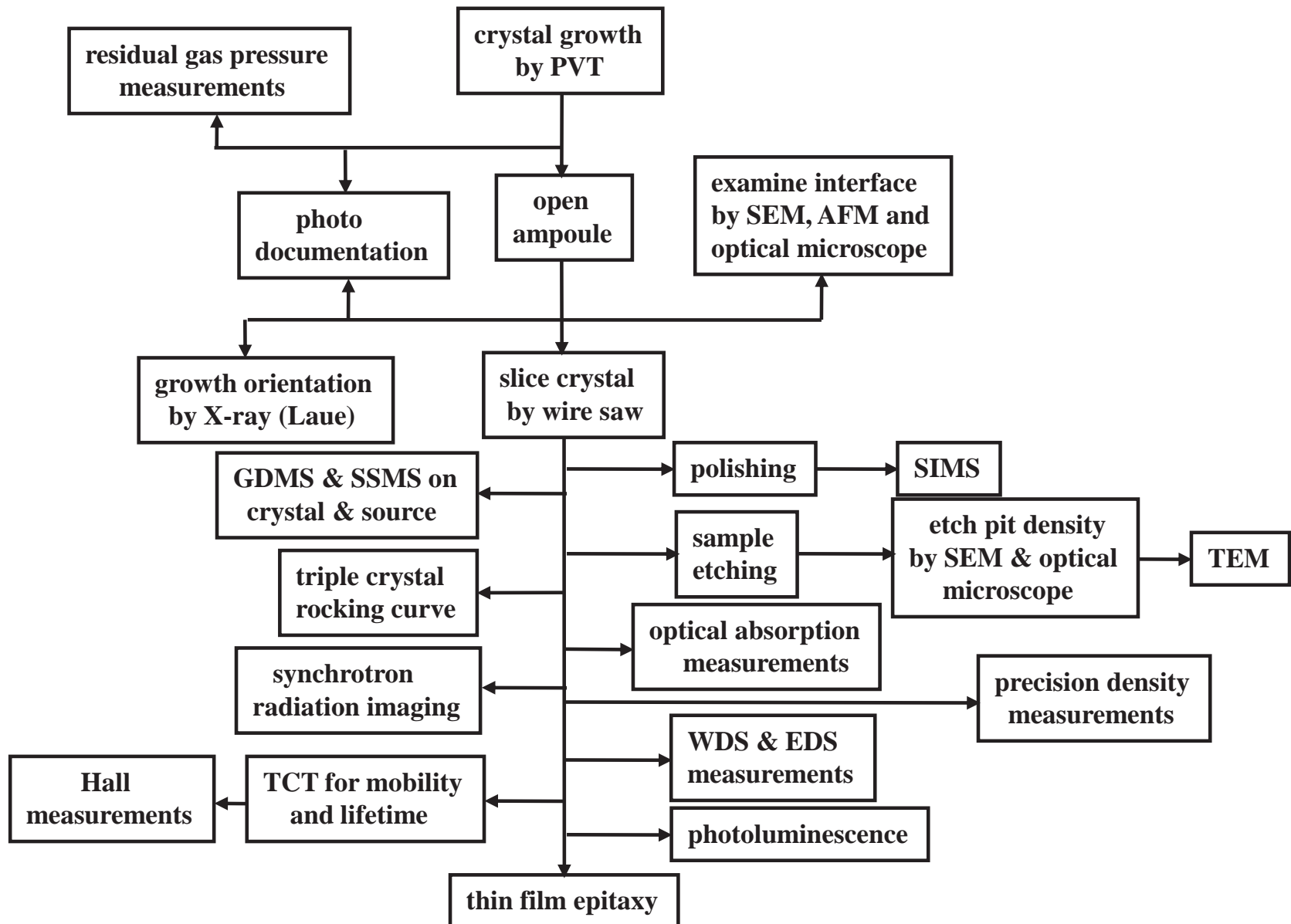


PVT Growth of ZnSe and Related Ternary Compounds

1. Self-seeded growth of ZnSe in vertical (stabilized and destabilized) and horizontal configurations
2. Seeded growth of ZnSe in vertical and horizontal configurations
3. Self-seeded growth of Cr-doped ZnSe in vertical and horizontal configurations
4. Self-seeded growth of ZnSeTe in vertical and horizontal configurations
5. *In-situ* and real-time optical monitoring of seeded growth in a horizontal configuration



Flow chart of sample characterization plan





Gravity Effects on the Grown Crystals

Effects were studied by comparing the following characteristics of horizontally and vertically grown ZnSe crystals in :

- Grown crystal morphology : contactless growth for the horizontal configuration.
- Surface morphology of the grown crystals was examined by SEM and AFM. (growth was terminated by stopping furnace translation, lowering the source temperature by 10 °C and then cooling the thermal profile at the same rate)
- Segregation and distribution of defects and impurities in the grown crystals was determined by photoluminescence, SIMS and precision density measurements.



Gravity Effects on the Grown Crystals

Morphology of the as-grown crystals:

- I. Self-seeded ZnSe: Crystals grown in the horizontal configuration grew away from the ampoule wall with large (110) facets tend to align parallel to the gravitational direction. Crystals grown in the vertical configuration grew in contact with the wall to the full diameter.
- II. Seeded ZnSe: the as-grown seeded crystals for the horizontal and vertical configurations showed similar characteristics in the morphology as described above for the self-seeded growth.

As-grown surface morphology:

- I. As-grown surface of horizontally grown crystals was dominated by (110) terraces and steps (identified to be (221) in one case).
- II. As-grown surface of the vertically grown
 - (a) Crystals showed granular structure with nanotubes (200nm OD, 75nm ID, 25nm in height for one case on ZnSe) on the top.
 - (b) Some crystals showed a network of high plateau with each island 30 – 70nm in diameter and 3.5nm in height. Numerous nuclei were observed with diameter 20 - 50nm and height of 1 - 7nm on top of these islands.

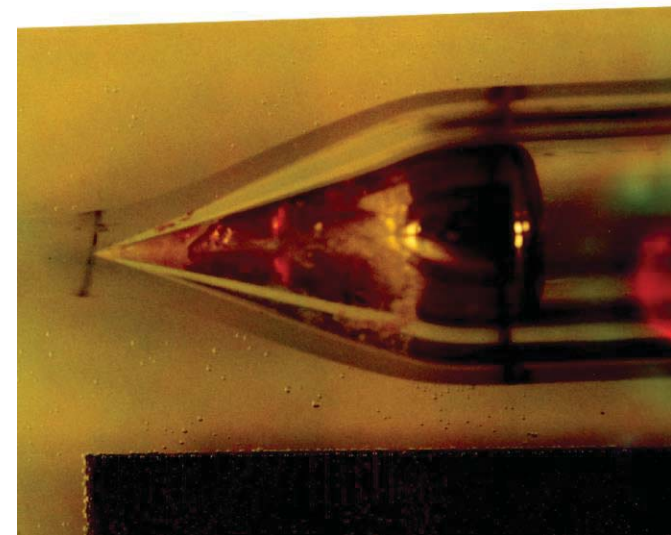
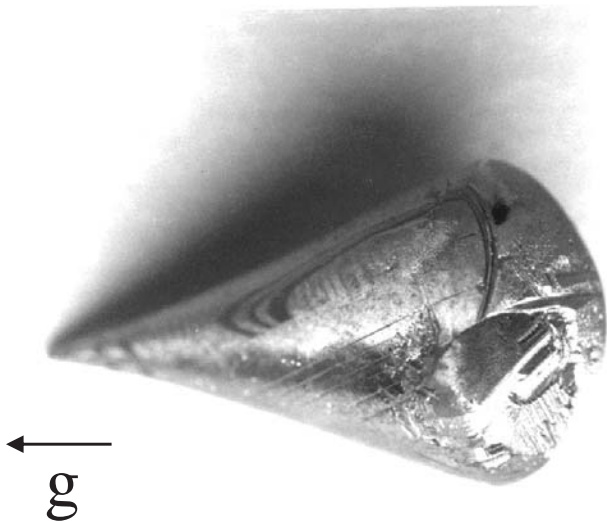
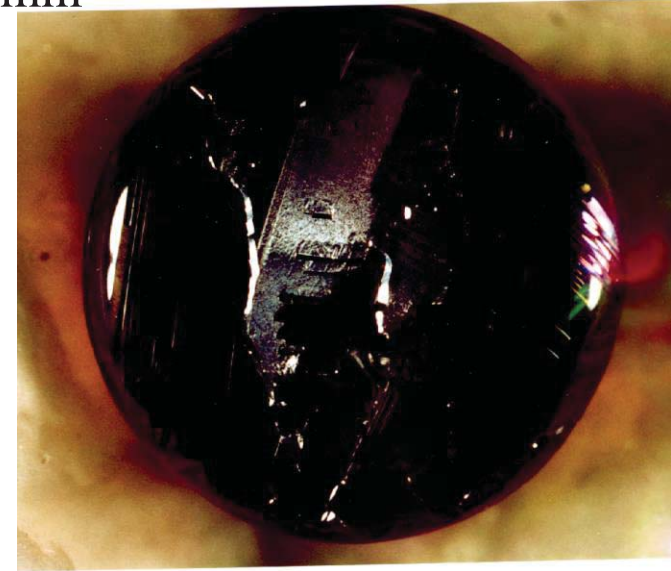


Morphologies of Self-seeded Vertically Grown ZnSe Crystals

ZnSe-25: vertically stabilized

ZnSe-31: vertically destabilized

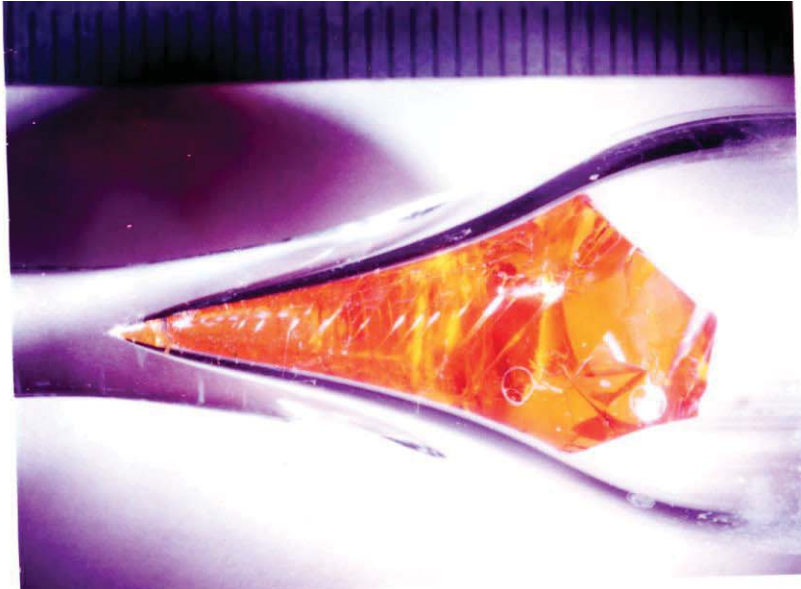
ampoule ID: 15mm



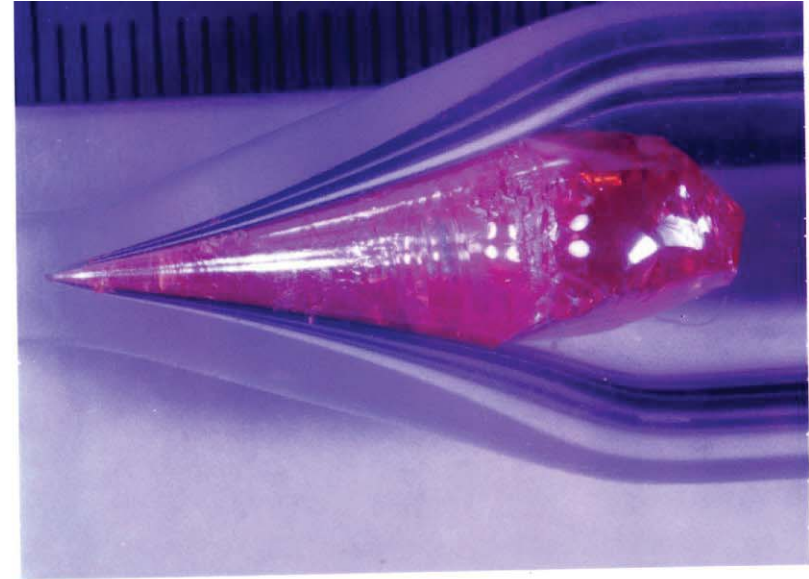


Morphologies of Self-seeded Horizontally Grown ZnSe Crystals

ZnSe-44



ZnSe-43

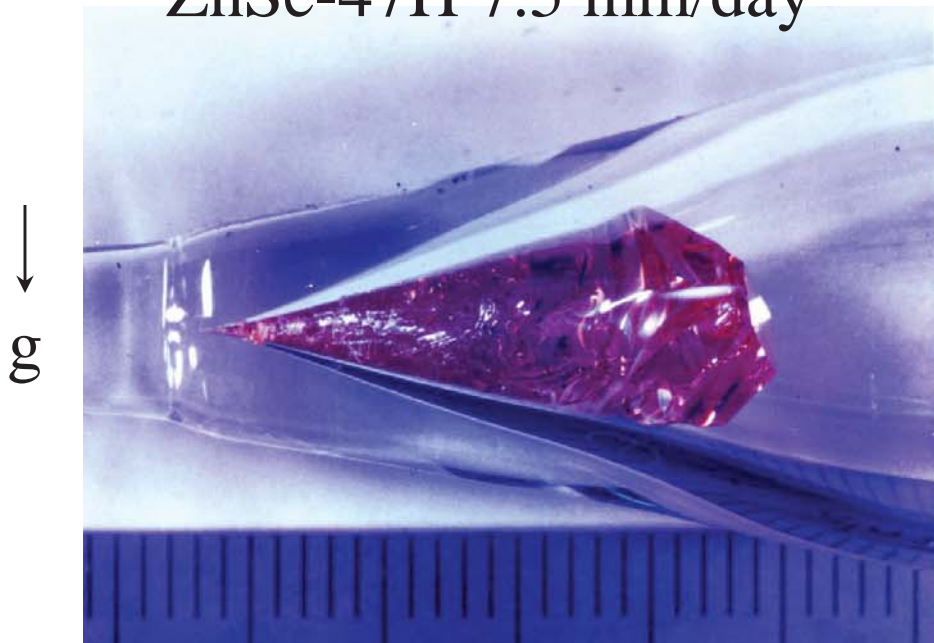




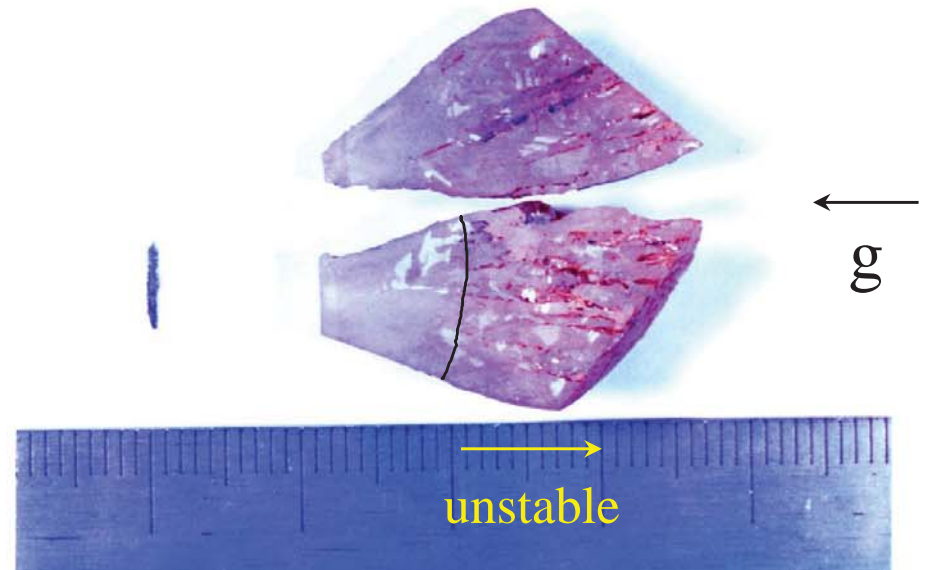
Gravity Effects on As-grown crystal morphology

- For furnace translation rates higher than the mass flux:
 - In the horizontal configuration the crystal maintained the growth surface morphological stability by (1) self-adjusting the degree of supersaturation to increase the mass flux or/and (2) by reducing the cross section area of the grown crystal.
 - In the vertical configuration the crystal growth surface became morphologically unstable with voids and pipes embedded.

ZnSe-47H 7.5 mm/day

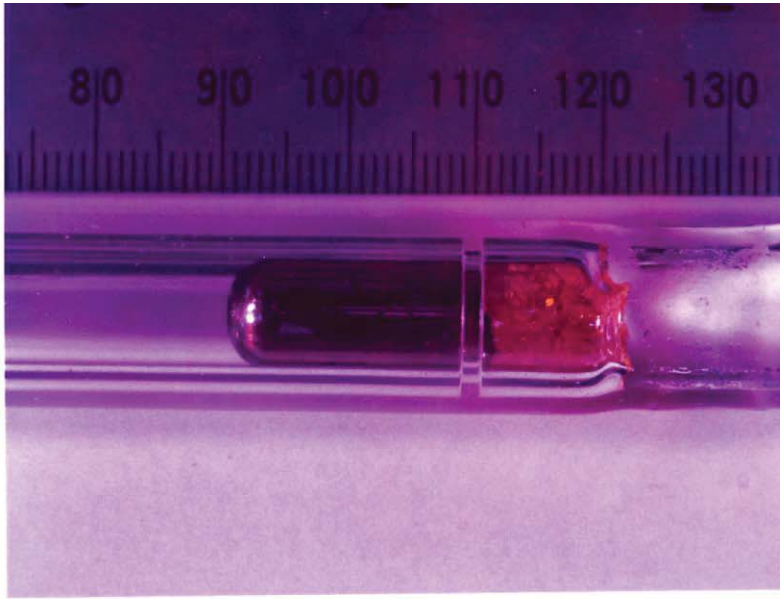


ZnSe-35V 11.4 mm/day

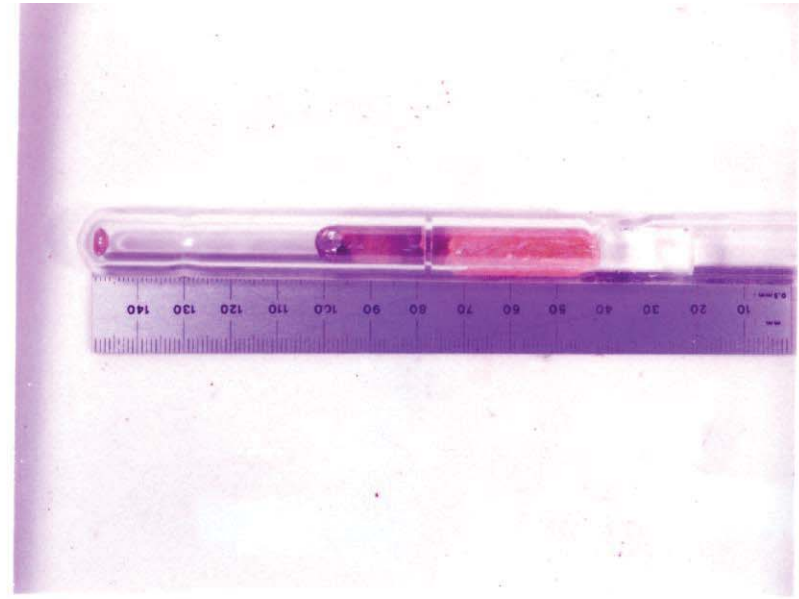




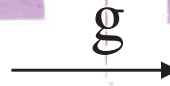
Morphologies of Seeded Vertically Grown ZnSe Crystals



ZnSe(S)-9V



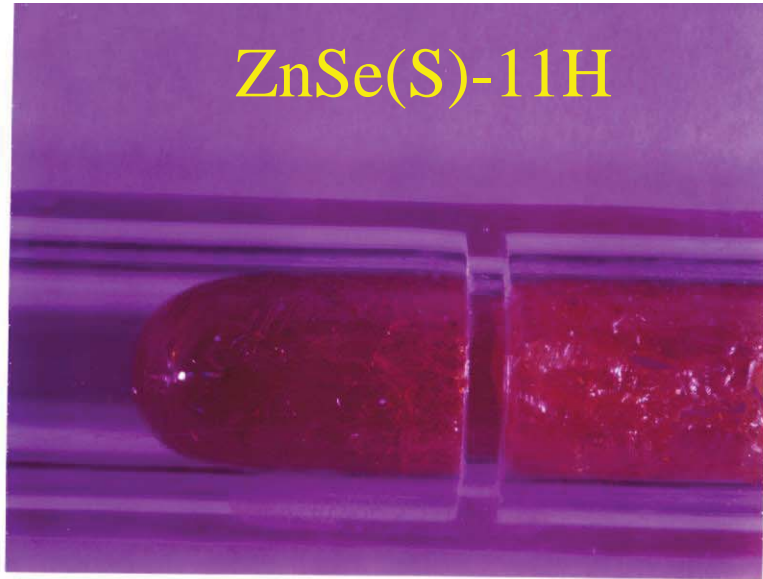
ZnSe(S)-12V



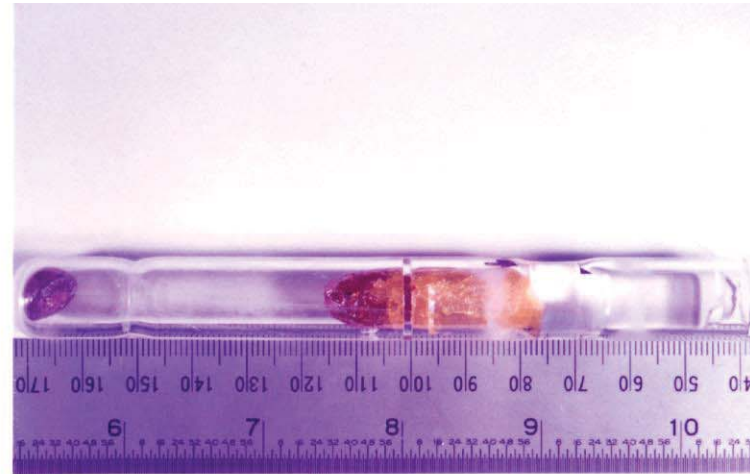


Morphologies of the Seeded Horizontally Grown ZnSe crystals

ZnSe(S)-11H



g
X



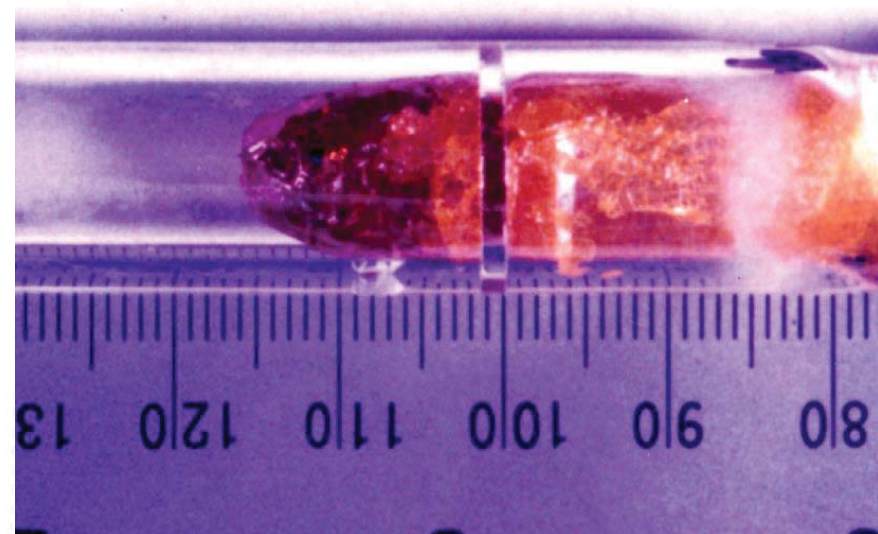
ZnSe(S)-8H

g
↓

ZnSe(S)-13H



g
↑





Gravity Effects on Impurities and Defects Distribution

I. Results from SIMS mappings :

For the horizontally grown self-seeded ZnSe crystal [Si] and [Fe] showed clear segregation toward the bottom on the wafer cut axially along the growth axis.

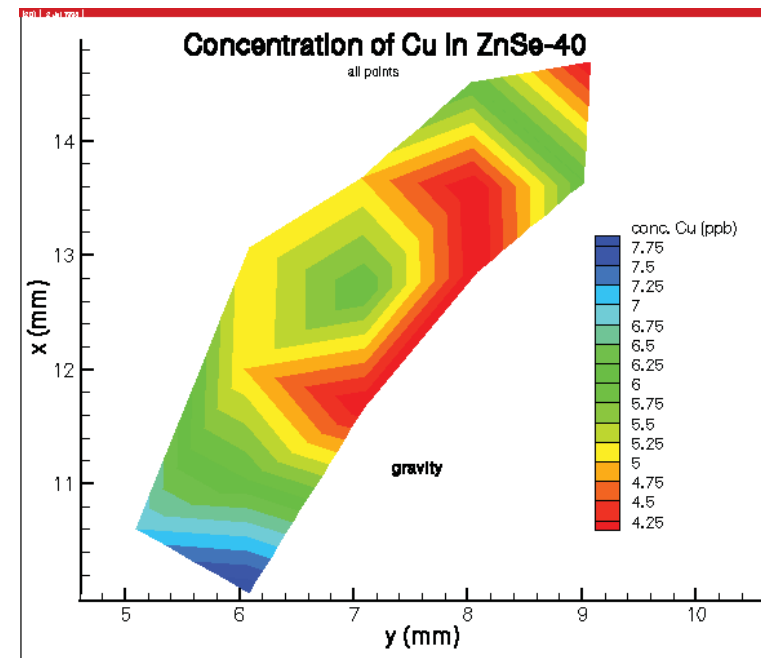
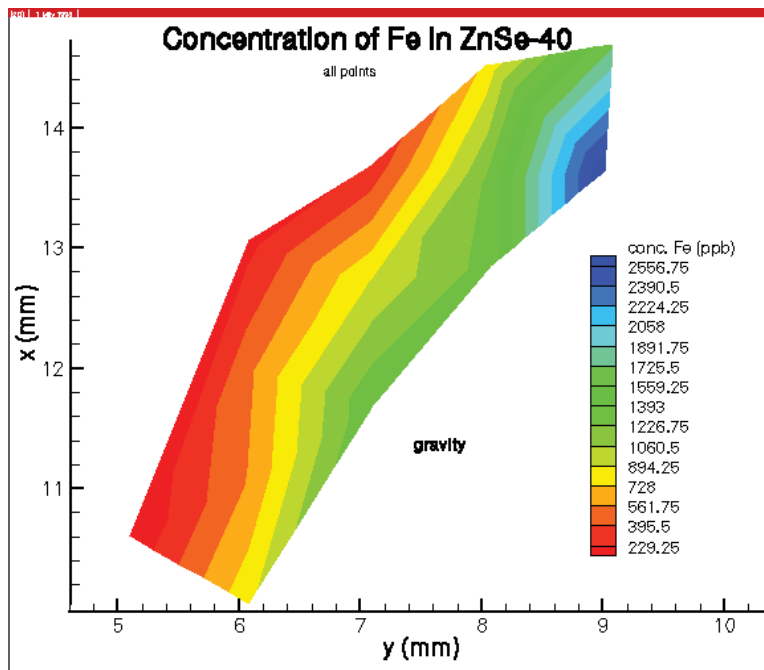
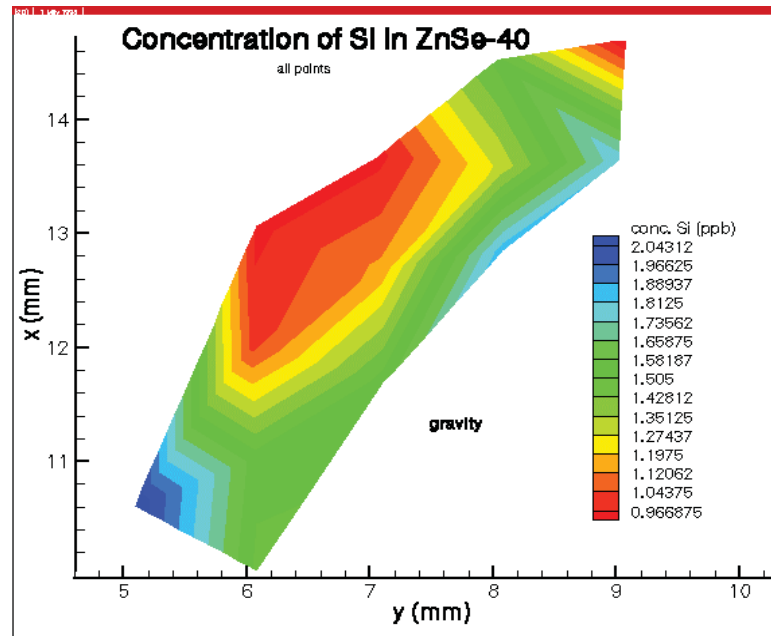
For the vertically grown seeded ZnSe crystal [Si] and [Cu] showed segregation toward the edge of the wafer cut perpendicular to the growth axis .

II. Mappings of PL near band edge intensity ratios indicated:

- (1) All the horizontally-grown crystals showed the following trends in the radial and axial segregation of [Al] and $[V_{Zn}]$:
[Al] segregates radially toward the top and axially toward the first grown region and $[V_{Zn}]$ segregates radially toward the bottom and axially toward the last grown region.
- (2) The as-grown surface of the seeded vertically stabilized grown crystal showed [Al], [Li or/and Na] and $[V_{Zn}]$ segregate radially toward the center.
- (3) The as-grown surface of the self-seeded vertically destabilized grown crystal showed [Al] and $[V_{Zn}]$ segregate radially without an apparent pattern.

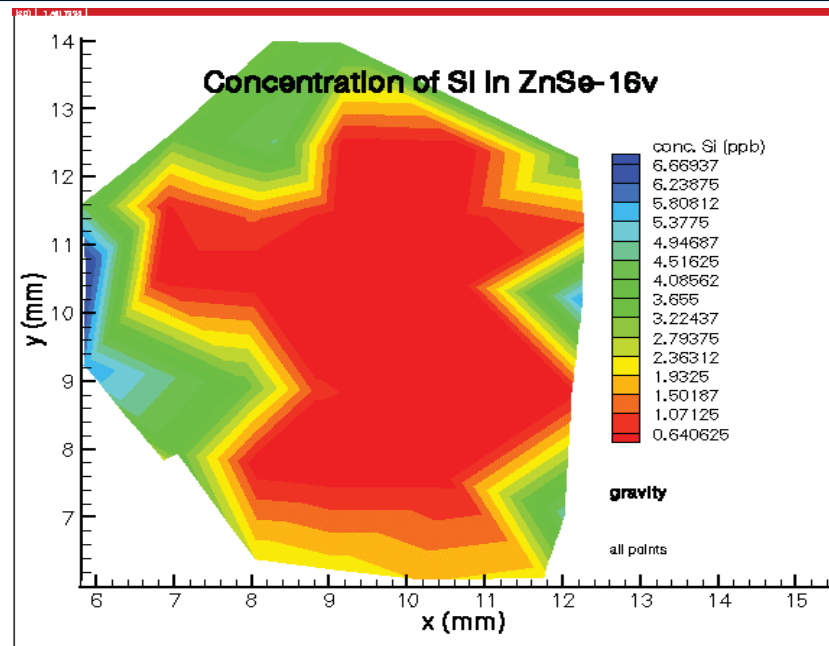


Impurities Distribution by SIMS (horizontally grown)

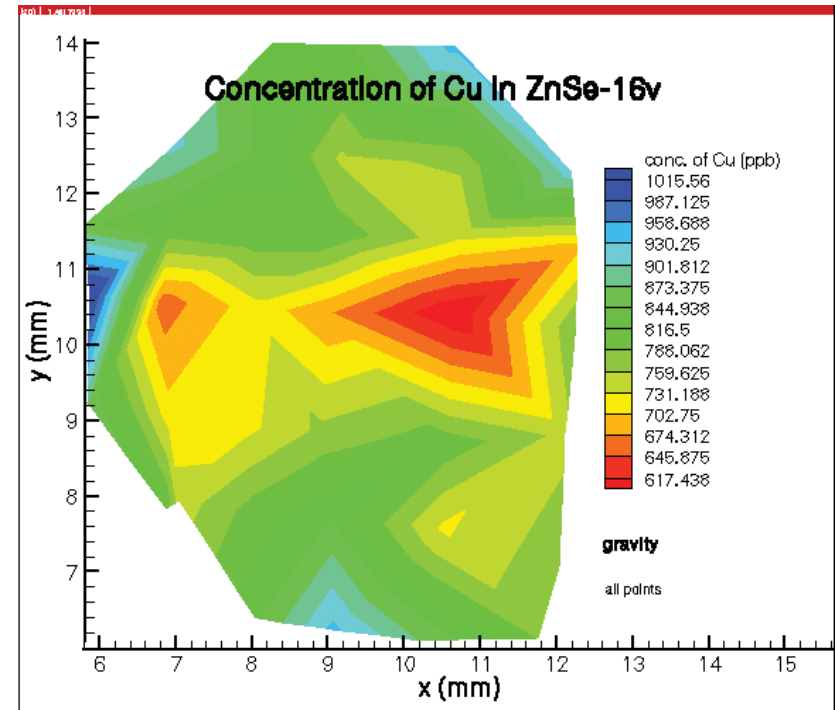
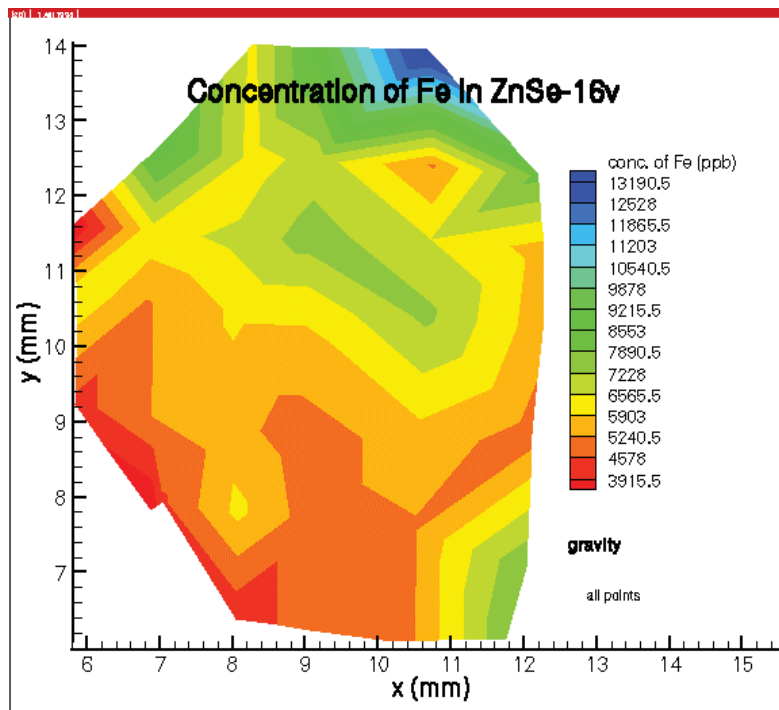




Impurities Distribution by SIMS (vertically grown)



X
gravity and
growth direction





Summary on the Photoluminescence I_2 and I_1^{deep} emissions

I_2 emission:

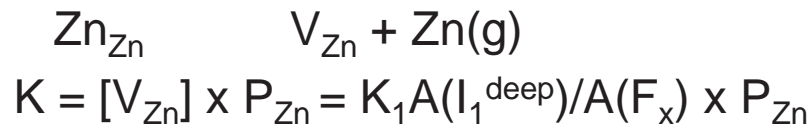
- I_2 , the exciton bound to substitutional donor, emission in our ZnSe samples can be attributed mainly to Al impurity, with $A(I_2)/A(F_x) = 4.88$ corresponding to 1700 ppb, atomic, or $7.46 \times 10^{16} \text{ cm}^{-3}$.
- Isshiki et al. (1991) gave the expression between intensity ratio $(I_2)/(I_{F_x})$ and N_D :

$$\log_{10}(I_2/I_{F_x}) = -22.0775 + 1.46268 \log_{10} N_D(\text{cm}^{-3})$$
- Therefore, $(I_2/I_{F_x}) = 82 [A(I_2)/A(F_x)]$

I_1^{deep} emission:

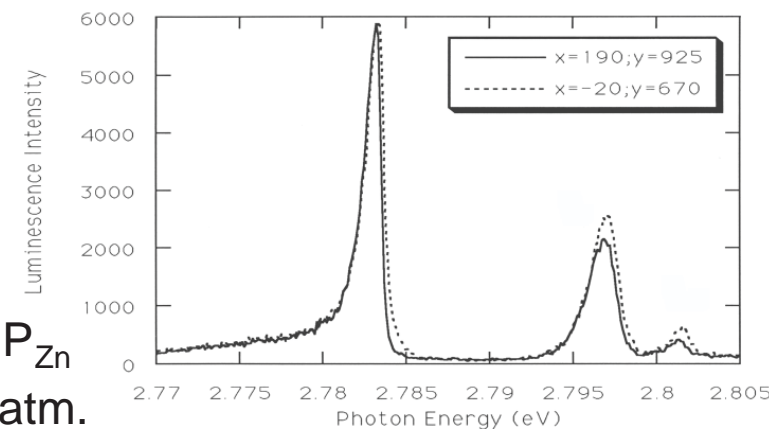
- I_1^{deep} is related to exciton bound to V_{Zn} deep acceptor and $[V_{\text{Zn}}]$ is proportional to $A(I_1^{\text{deep}})/A(F_x)$.

- The reaction during Zn vapor annealing:



- The ZnSe samples were annealed at 1104 °C:

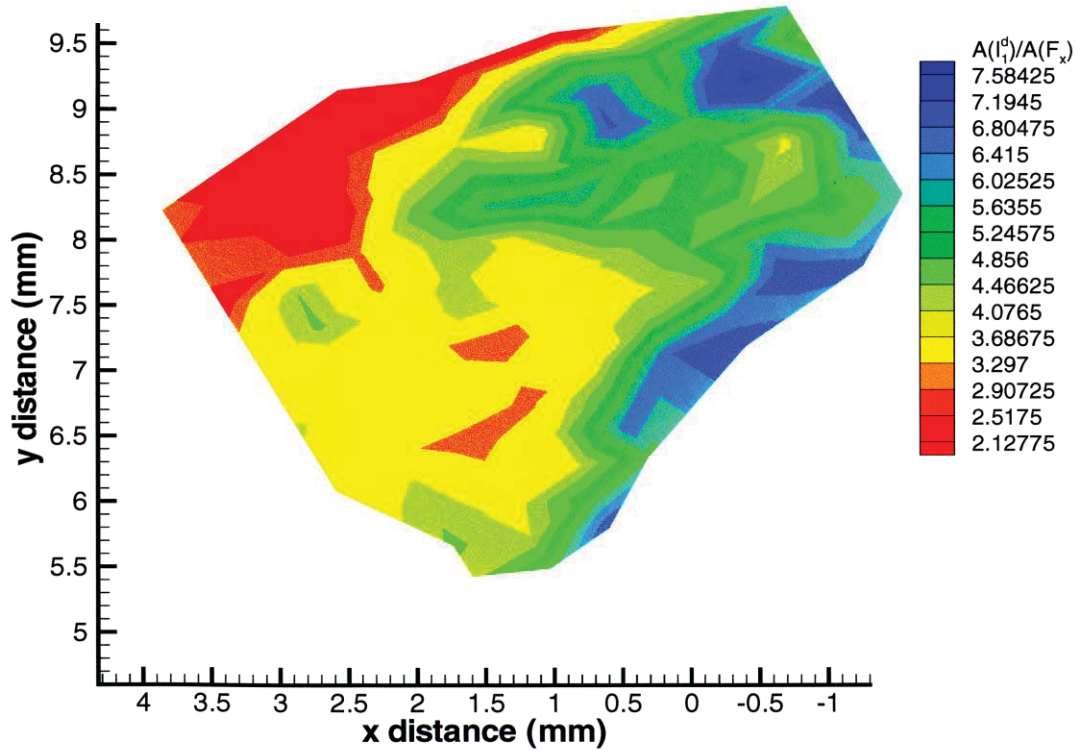
- $A(I_1^{\text{deep}})/A(F_x) = 7.52$ when sample is in equilibrium with $P_{\text{Zn}} = 6.1 \times 10^{-3} \text{ atm}$ ($\alpha = 6.05$) $A(I_1^{\text{deep}})/A(F_x) \times P_{\text{Zn}} = 0.0459 \text{ atm}$.
- $A(I_1^{\text{deep}})/A(F_x) = 5.18$ when sample is in equilibrium with $P_{\text{Zn}} = 9.0 \times 10^{-3} \text{ atm}$ ($\alpha = 19.43$) $A(I_1^{\text{deep}})/A(F_x) \times P_{\text{Zn}} = 0.0466 \text{ atm}$



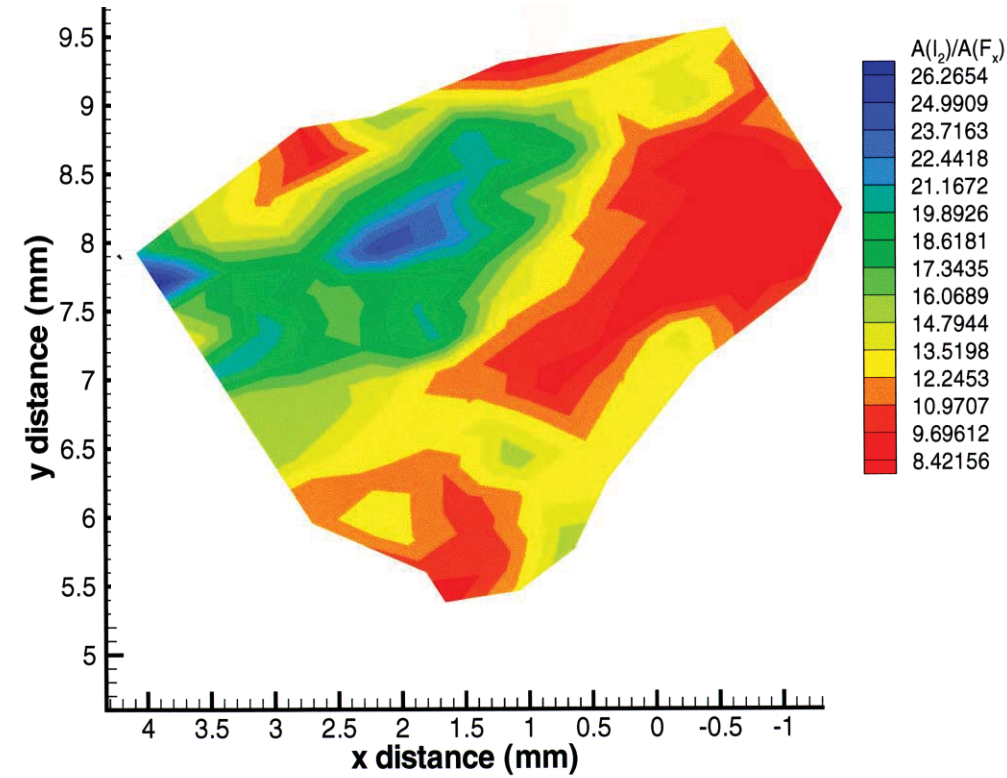


Distribution of $[V_{Zn}]$ and $[Al]$ in ZnSe (horizontally grown)

$[V_{Zn}]$



$[Al]$



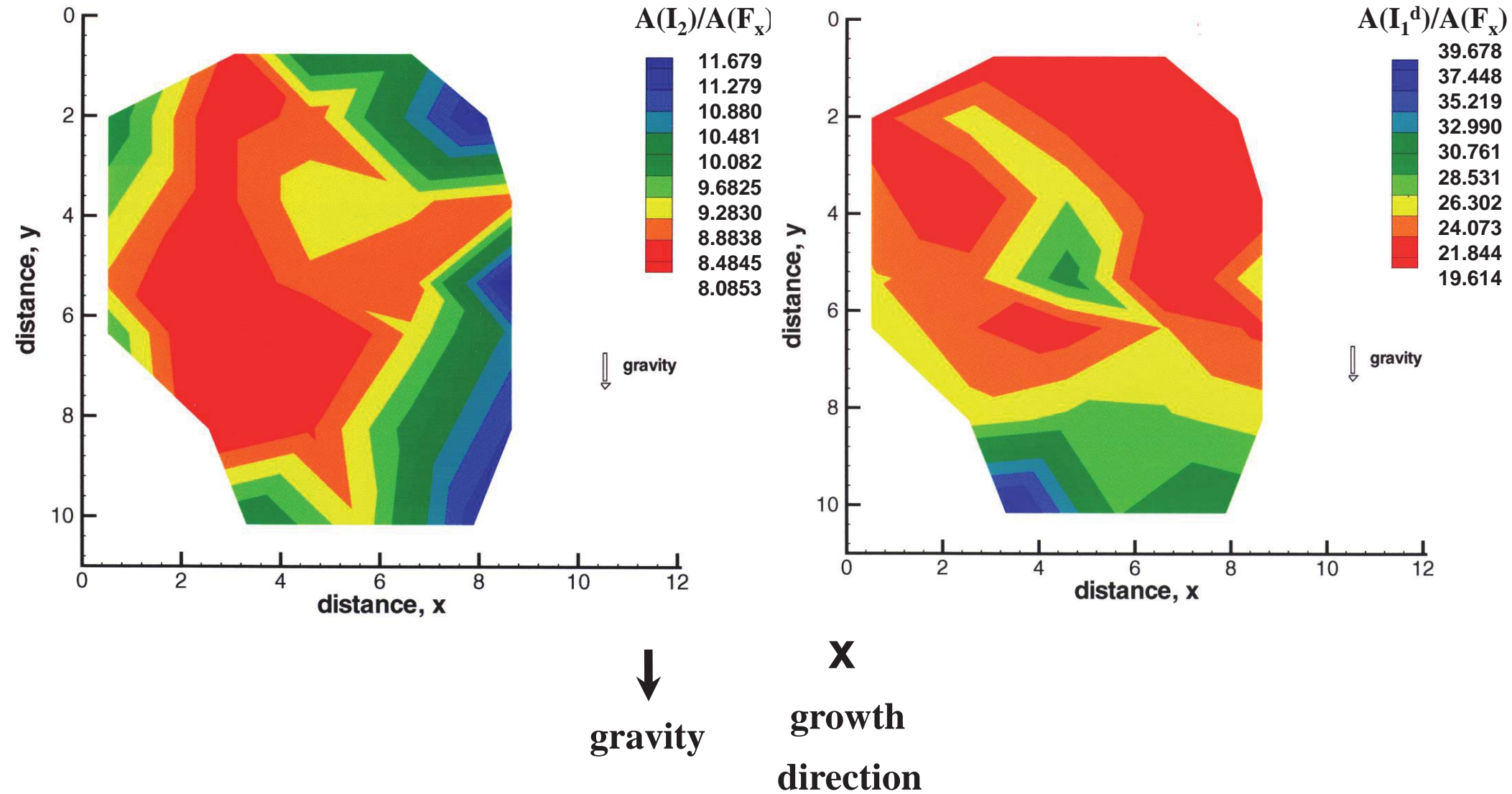
gravity



growth
direction

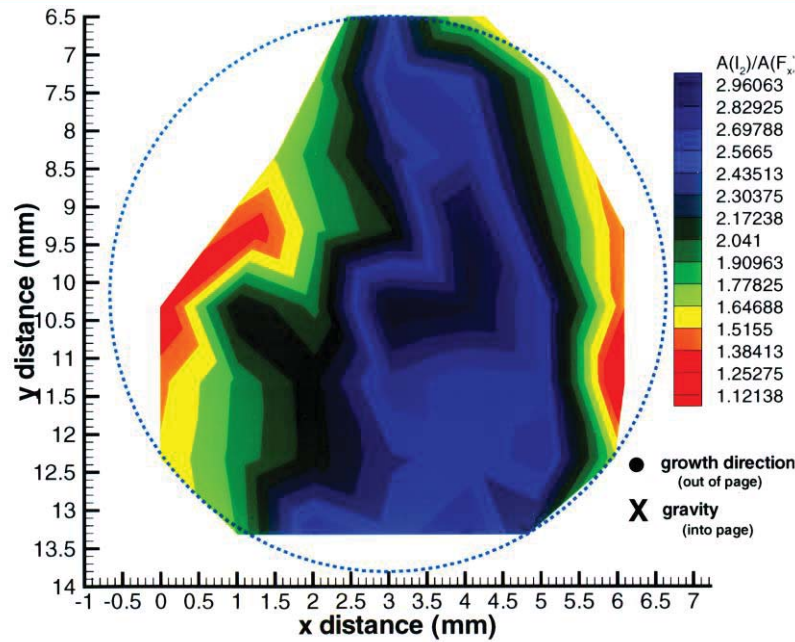


Distribution of $[V_{Zn}]$ and $[Al]$ in ZnSe (horizontally grown)

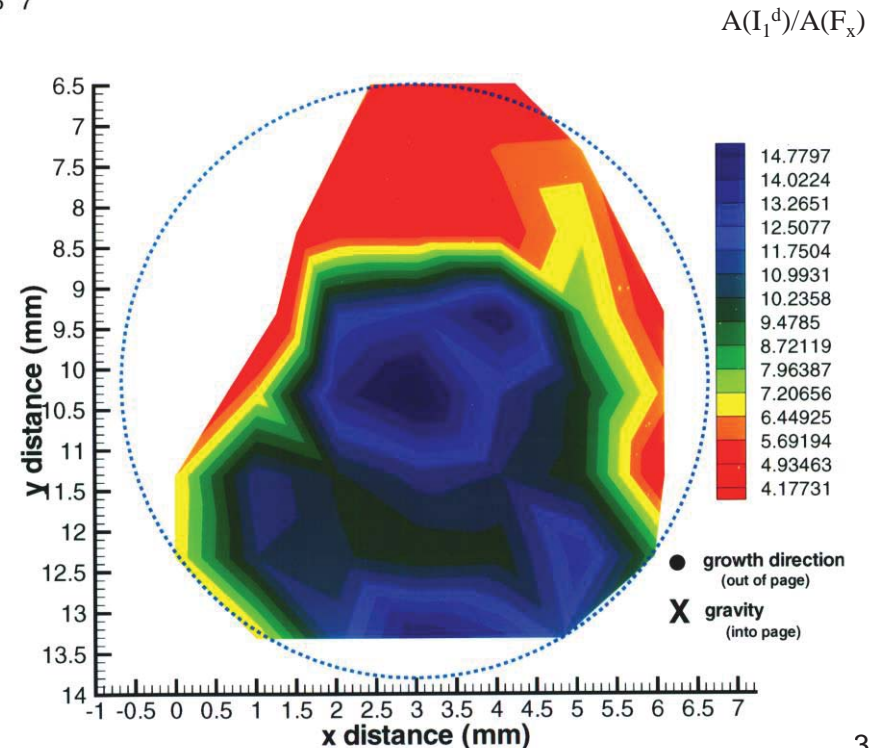
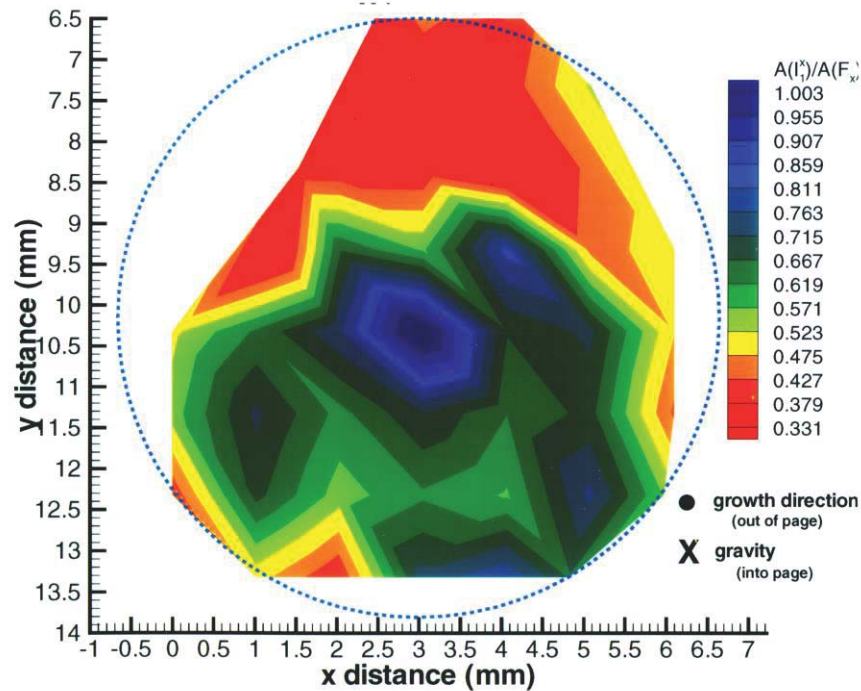




Distribution of $[V_{Zn}]$ and $[Al]$ in ZnSe (vertically grown)



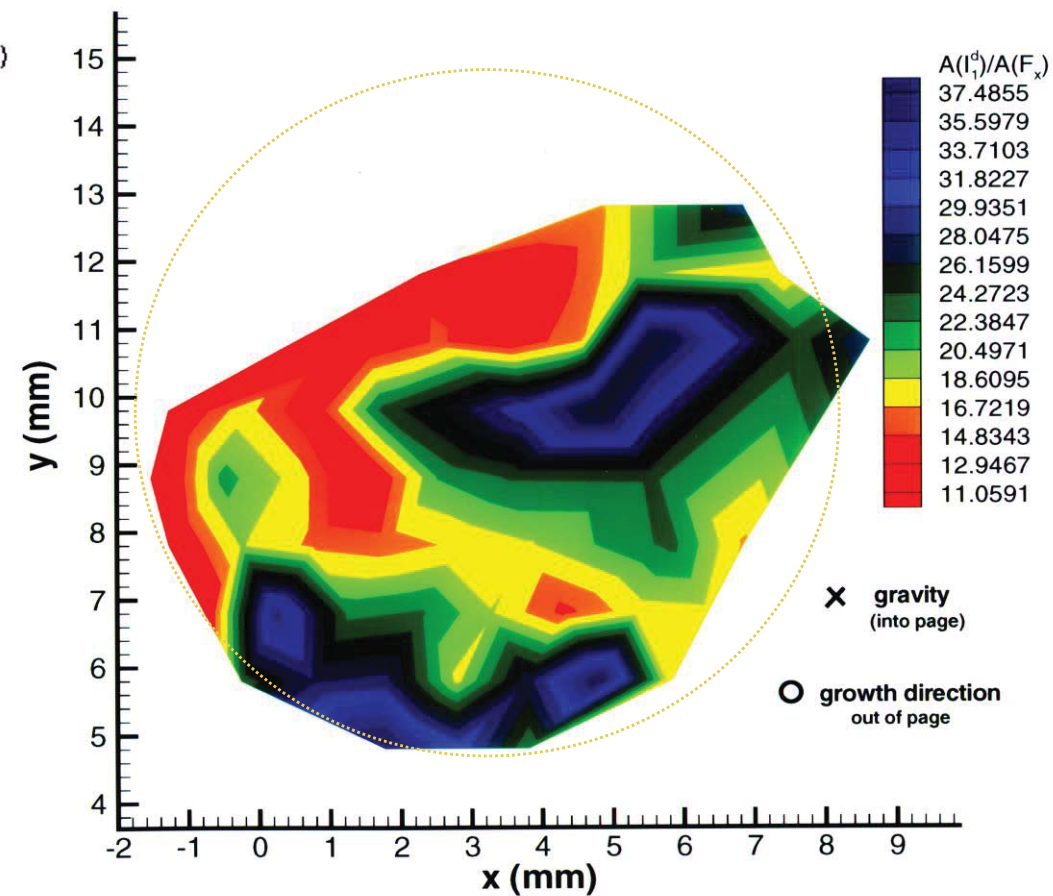
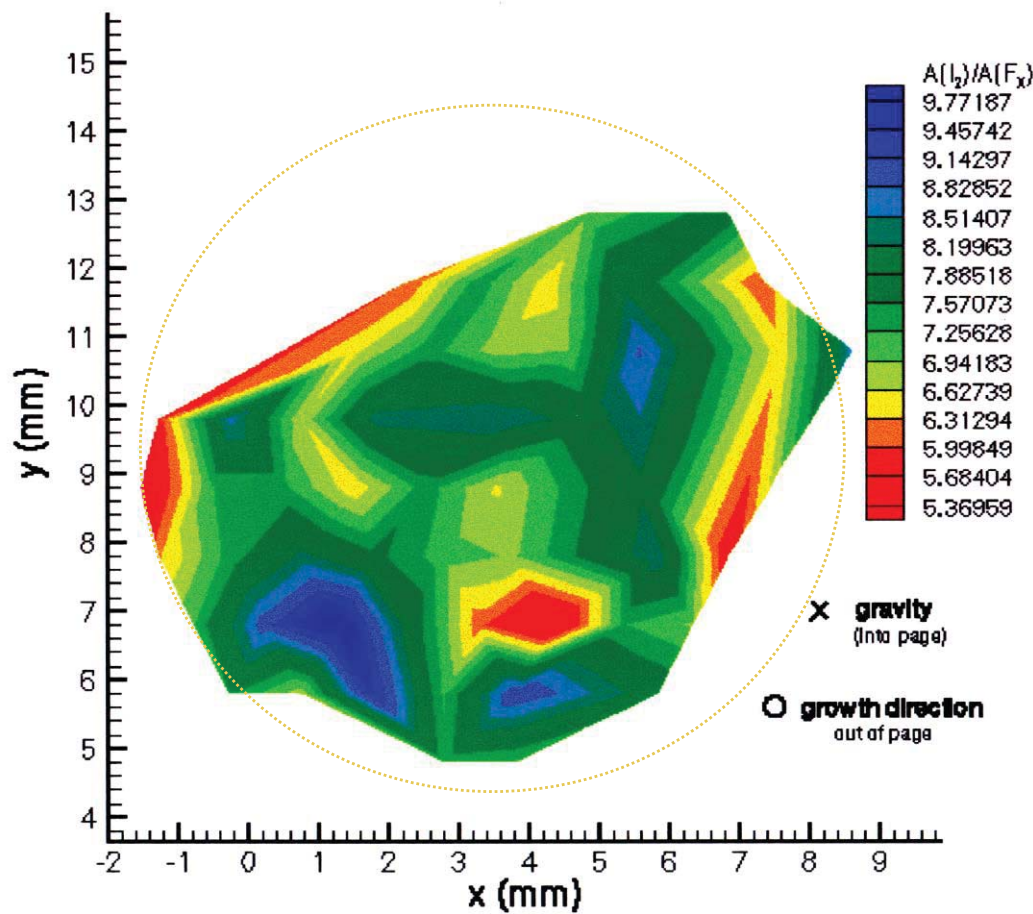
Vertical stabilized configuration





Distribution of $[V_{Zn}]$ and $[Al]$ in ZnSe (vertically grown)

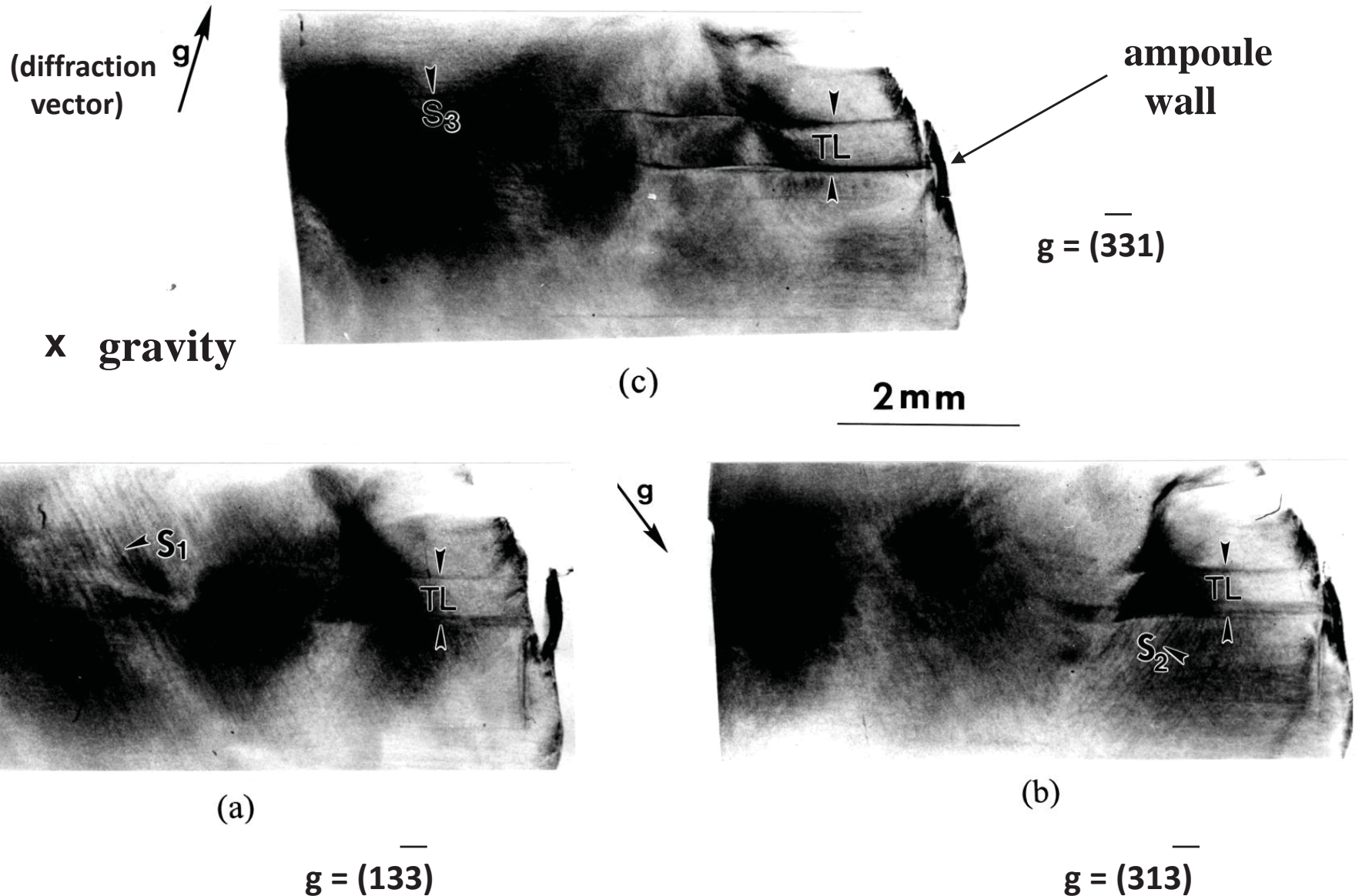
Vertical destabilized configuration





Synchrotron X-ray Transmission Topography of ZnSe wafer

Thin **twin lamellae (TL)** and **three sets of (111) slip bands (S)** originating from the lateral twin boundaries.



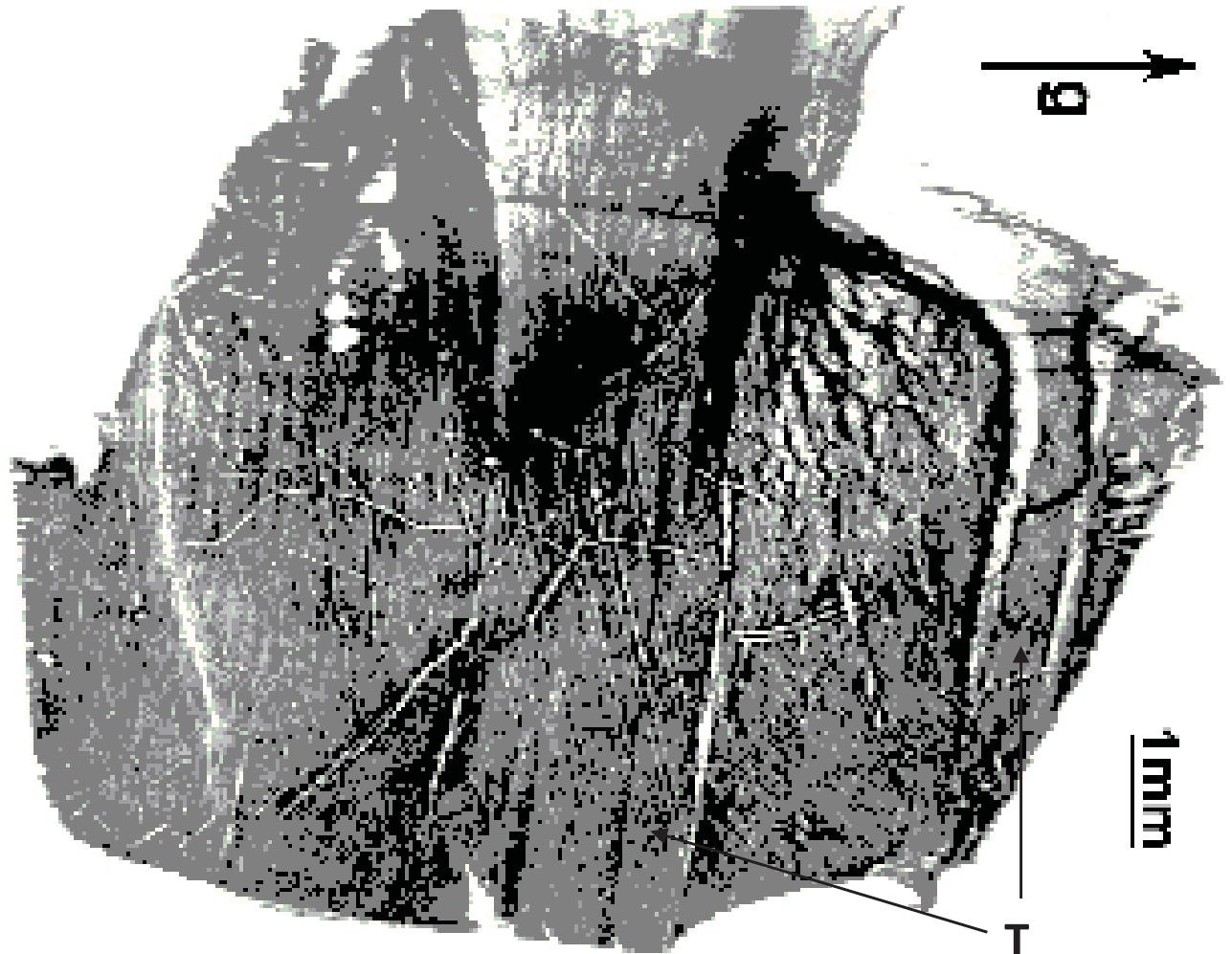


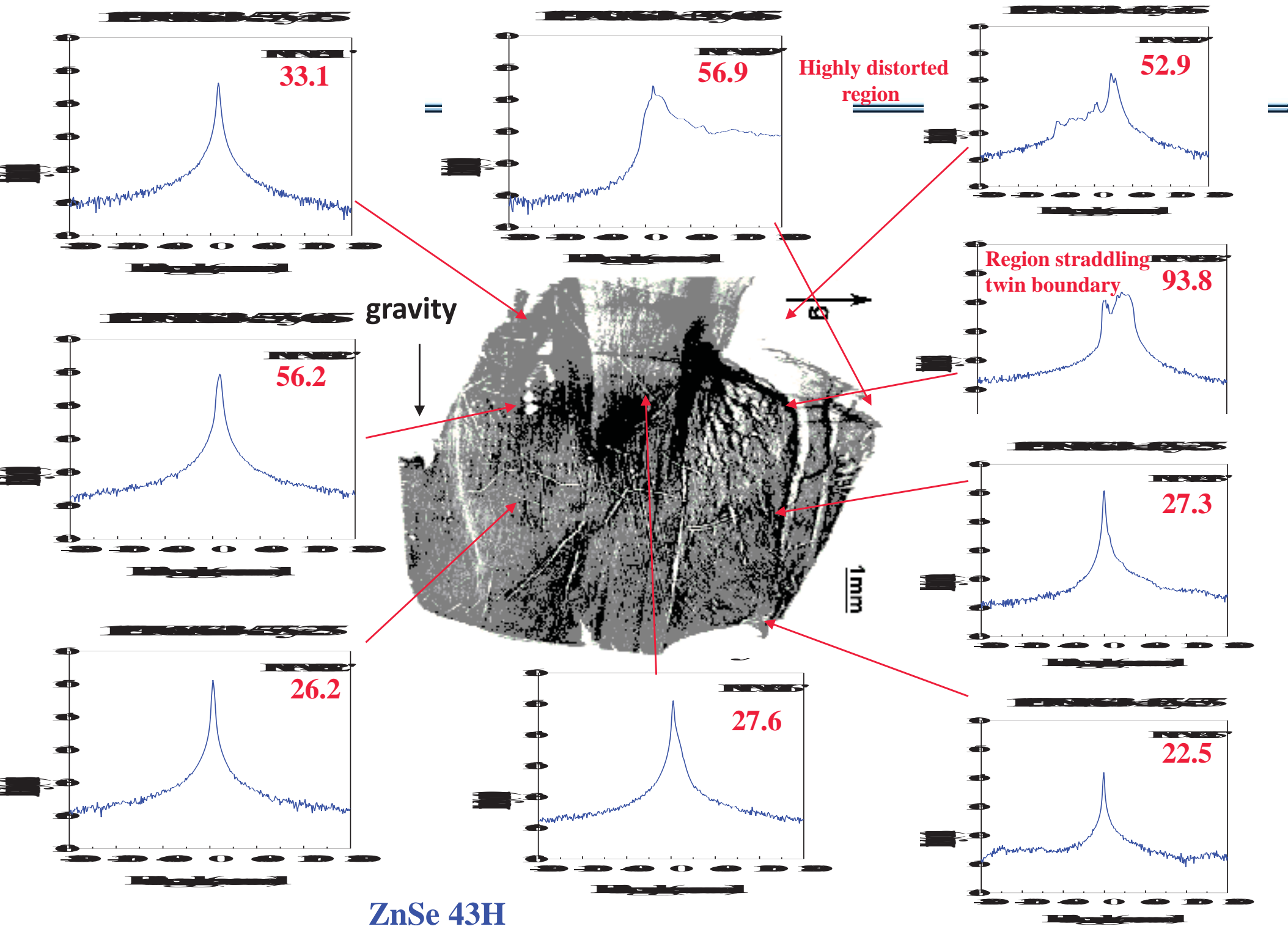
Reflection Topograph of ZnSe-43H as-grown Facet

Reflection SWBXT image on the as-grown facet of ZnSe-43H showed areas of twins. The crystalline quality is generally good except that the upper region, where the crystal started to grow into full diameter and away from the ampoule wall, showed lattice strain.



gravity ↓





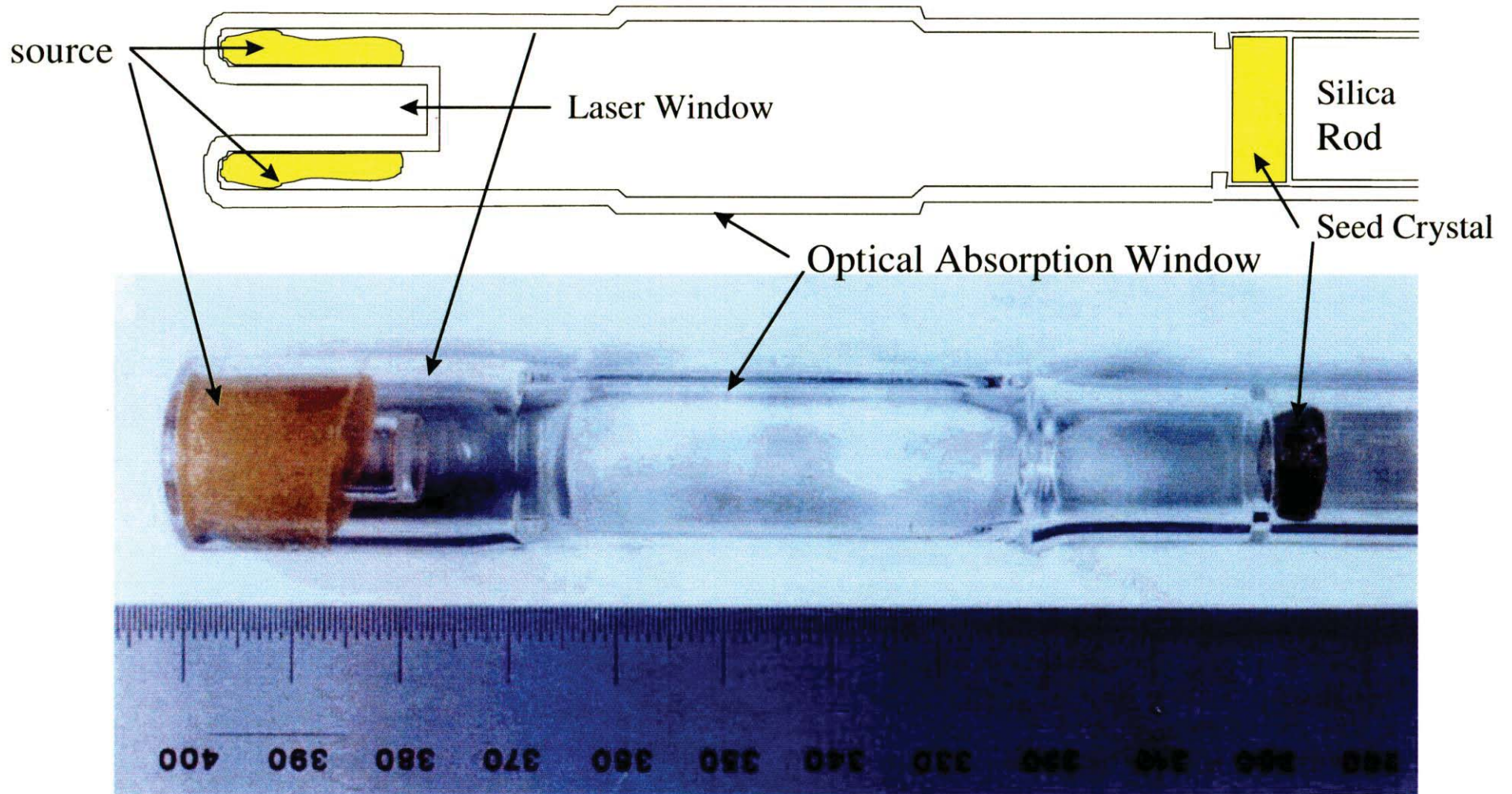


In-situ optical monitoring during crystal growth

- Optical absorption for partial pressure measurements along the length of the growth ampoule to measure **vapor phase transport species distribution**
- Optical interferometry to measure **seed thermal etching, instantaneous growth rate and the evolution of surface topography**
- Visual observation of the growth evolution

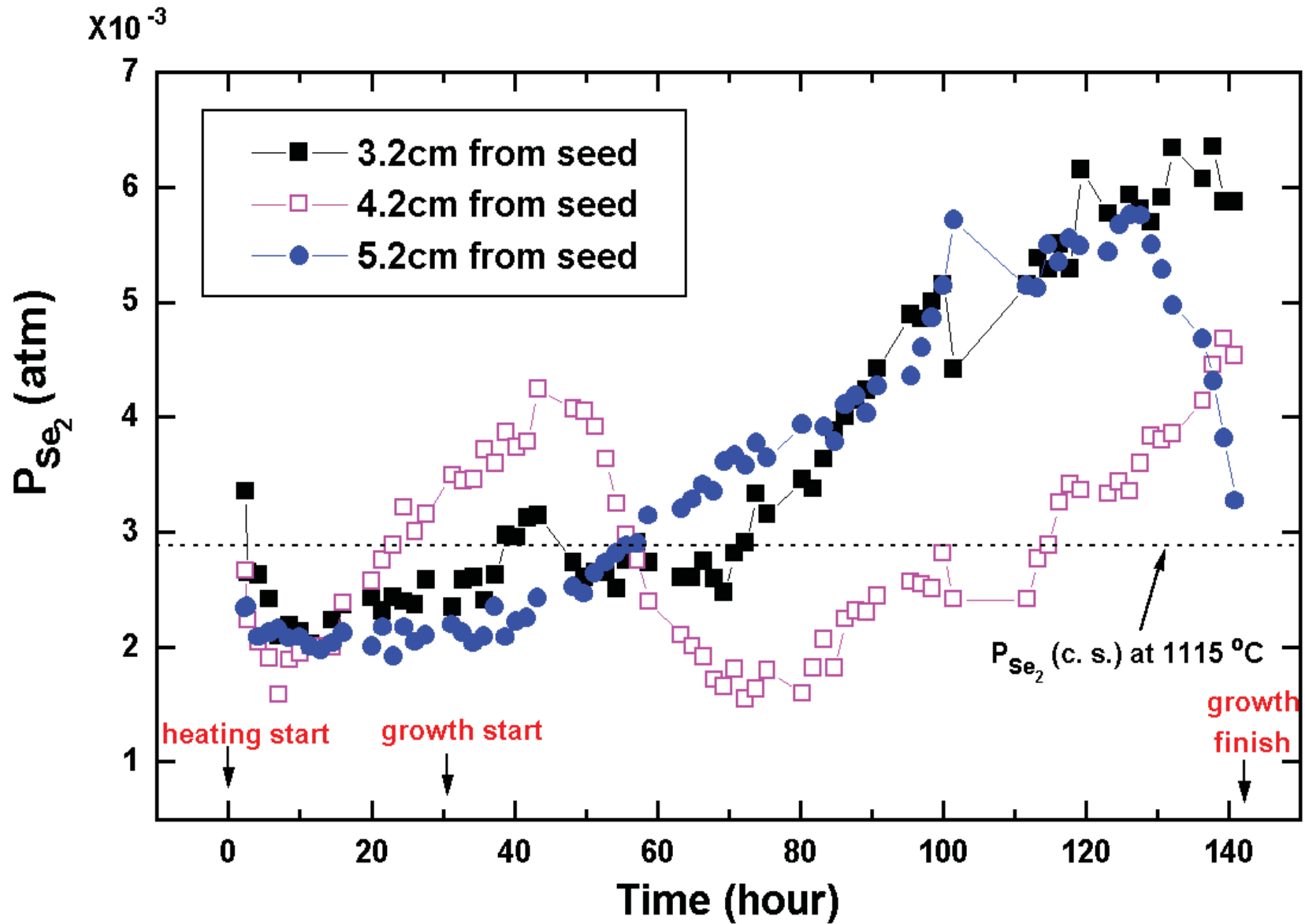


Ampoule Design for in-situ Optical Monitoring





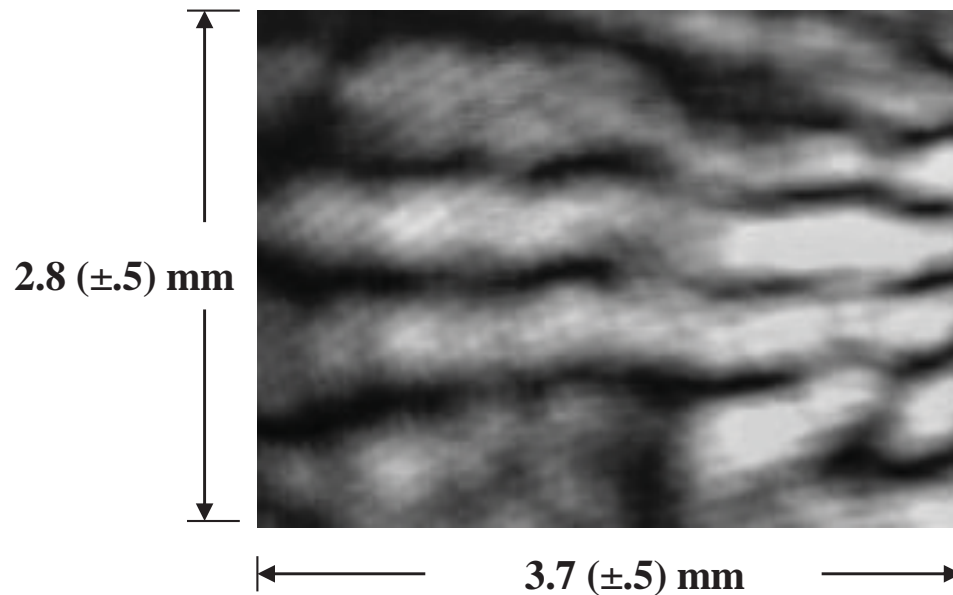
Measured Partial Pressure as a Function of Time



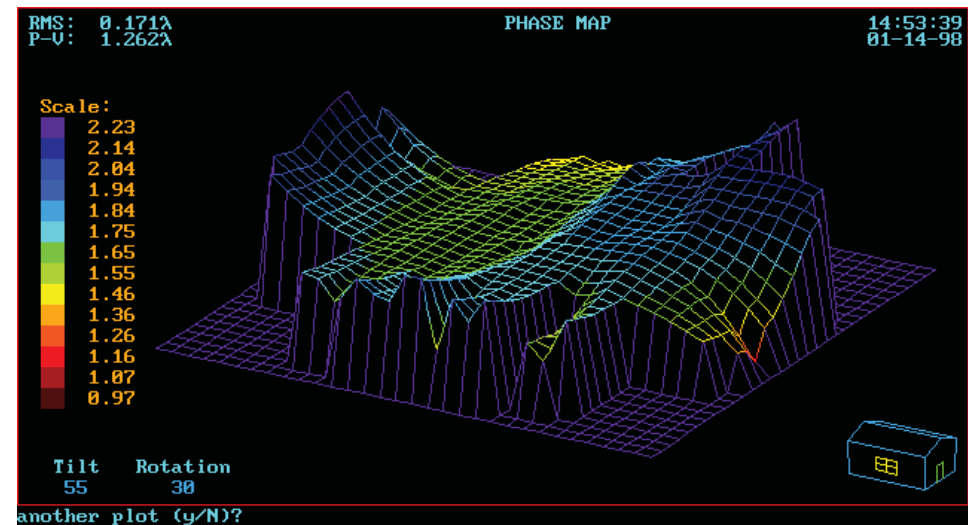


Interferometry Results from Seed Crystal Surface

(Room Temperature)



Fringes from Fabry-Perot Interferometer

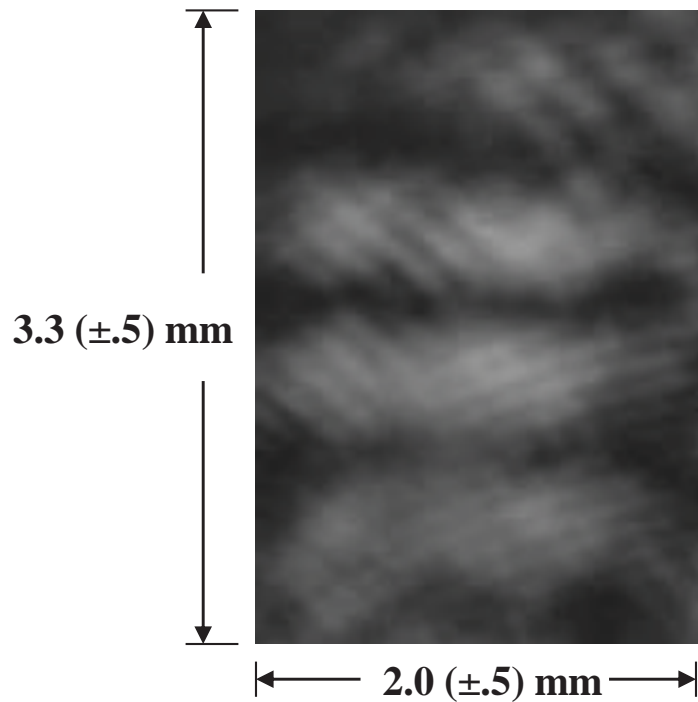


Fast/Vai Phase Map of Crystal Surface, (from Fringes)

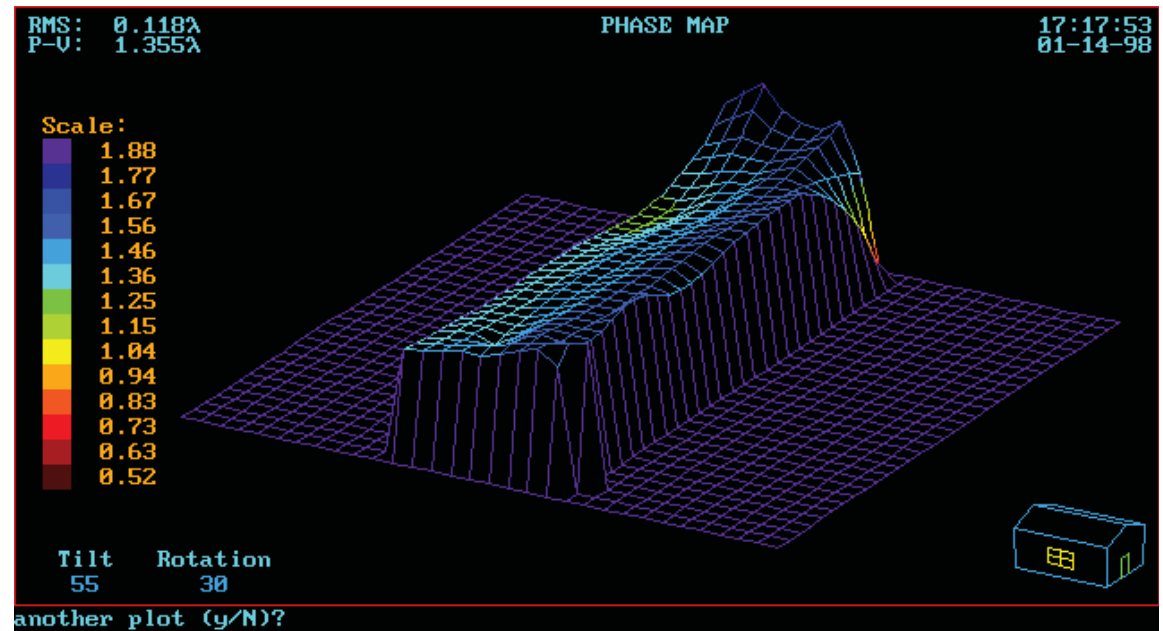


Interferometry Results from Seed Crystal Surface

(at 1120°C)



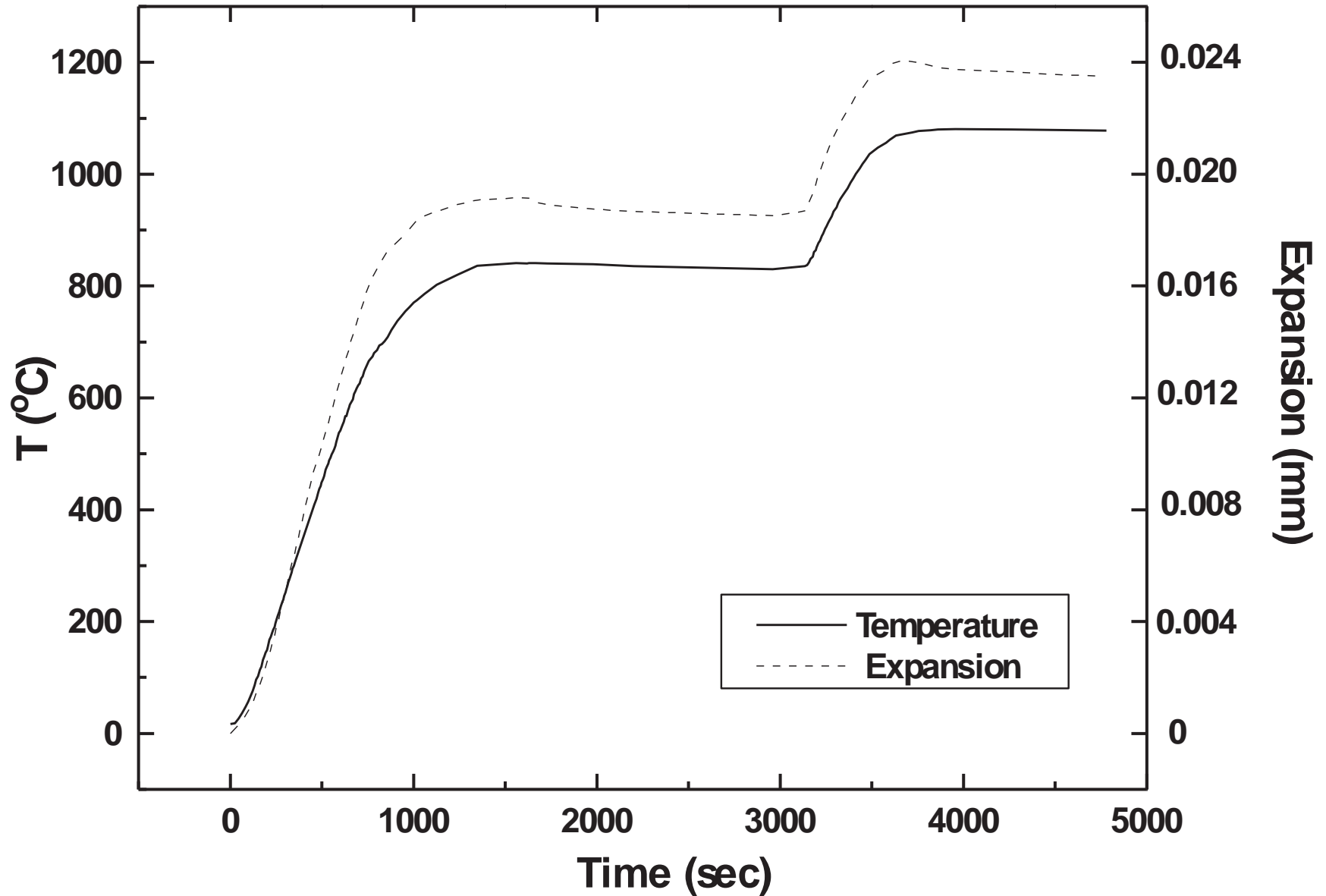
Fringes from Fabry-Perot Interferometer



Fast/Vai Phase Map of Crystal Surface, (from Fringes)



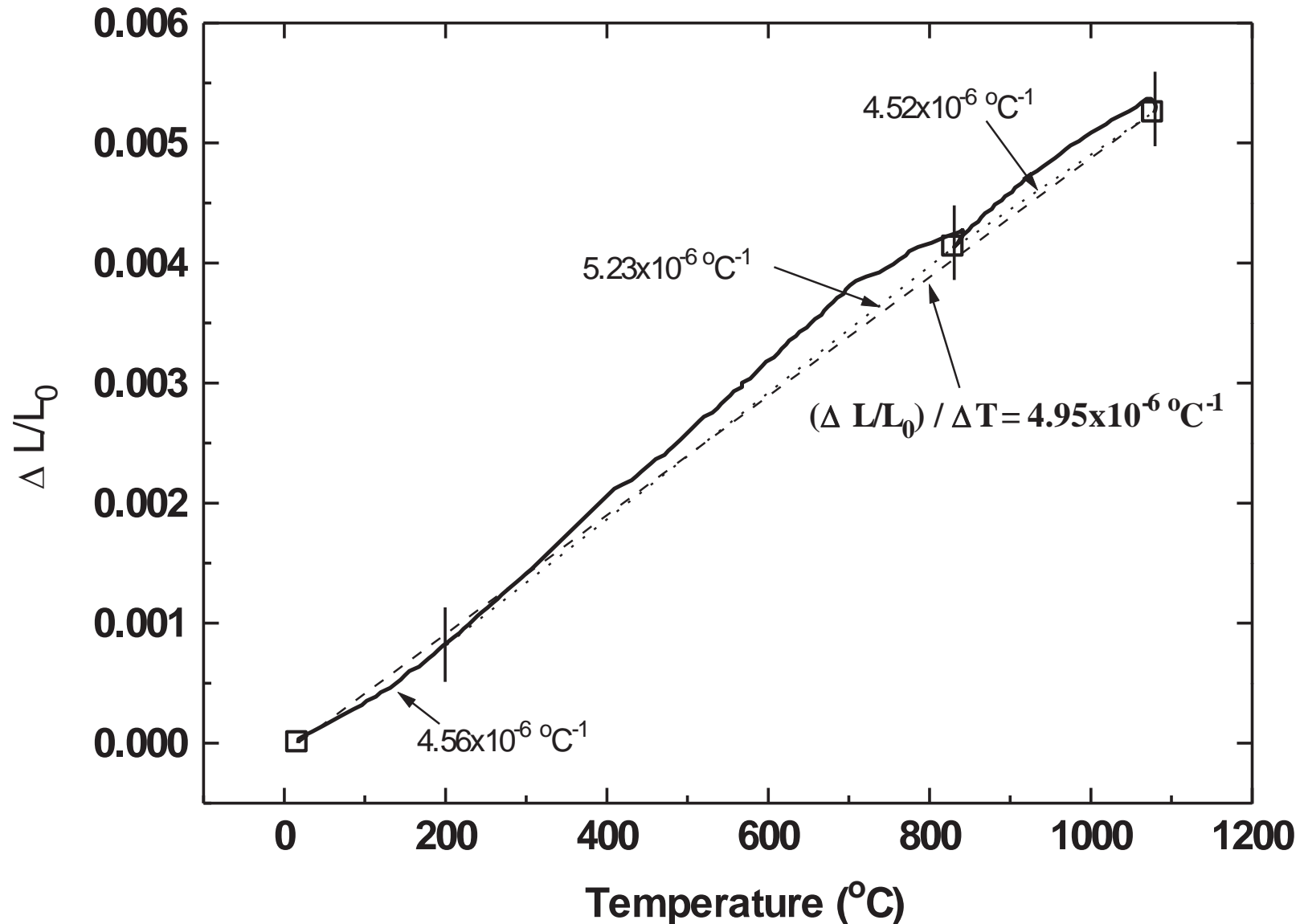
Synchronized plots of sample temperature and the measured thermal expansion





Thermal Expansion Coefficient of ZnSe from Interferometry

Expansion $\Delta L = n(\lambda/2)$ $\lambda = 632.8\text{nm}$ $L_0 = 4.5\text{mm}$





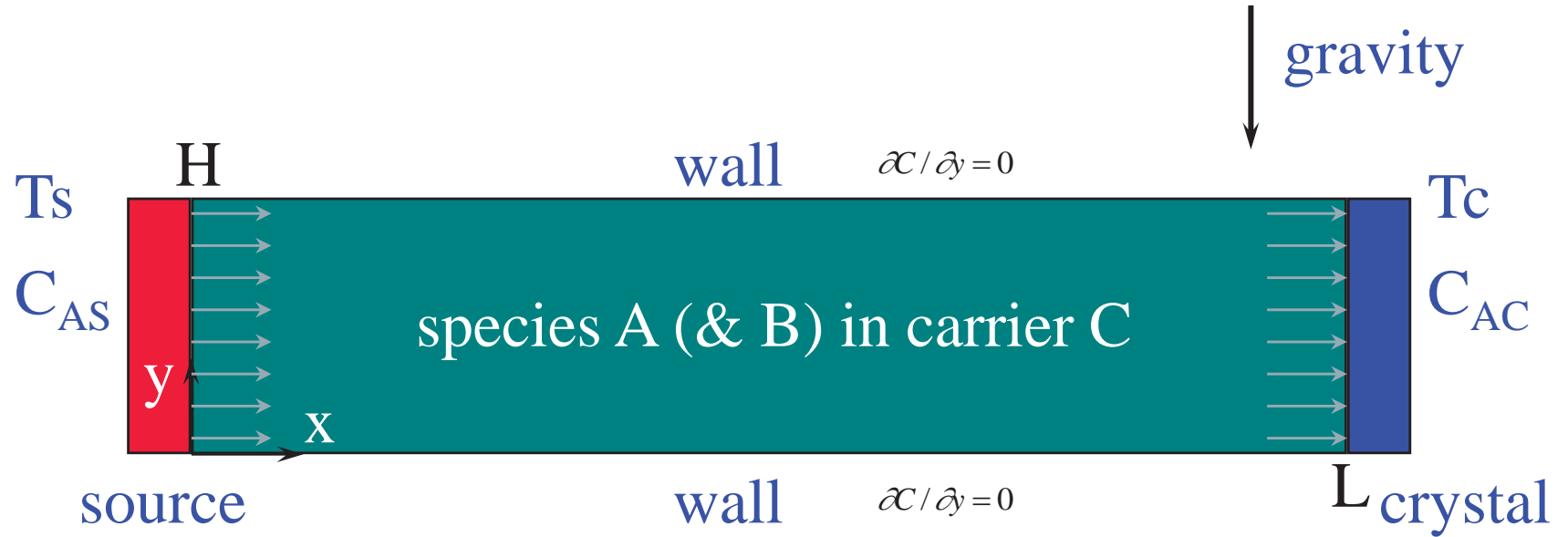
Numerical Modeling of Physical Vapor Transport

- Two dimensional and three dimensional calculations
- Finite element technique - Fidap code
- Thermal and Species induced buoyancy forces
- Compressible or Boussinesq model
- Benchmark -2D (H_2 - I_2 system - PVT growth)
- ZnSe calculations with residual gas
- Benchmark -3D (Natural convection in a cylinder)
- 3-D ZnSe calculations with residual gas
- Ongoing and future work

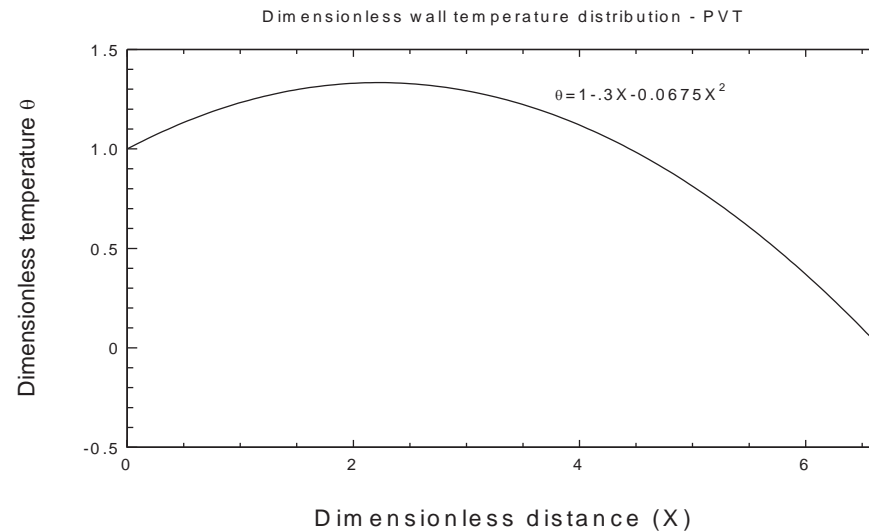
Ramachandran et al. (2000)



The Physical Model



wall
temperature



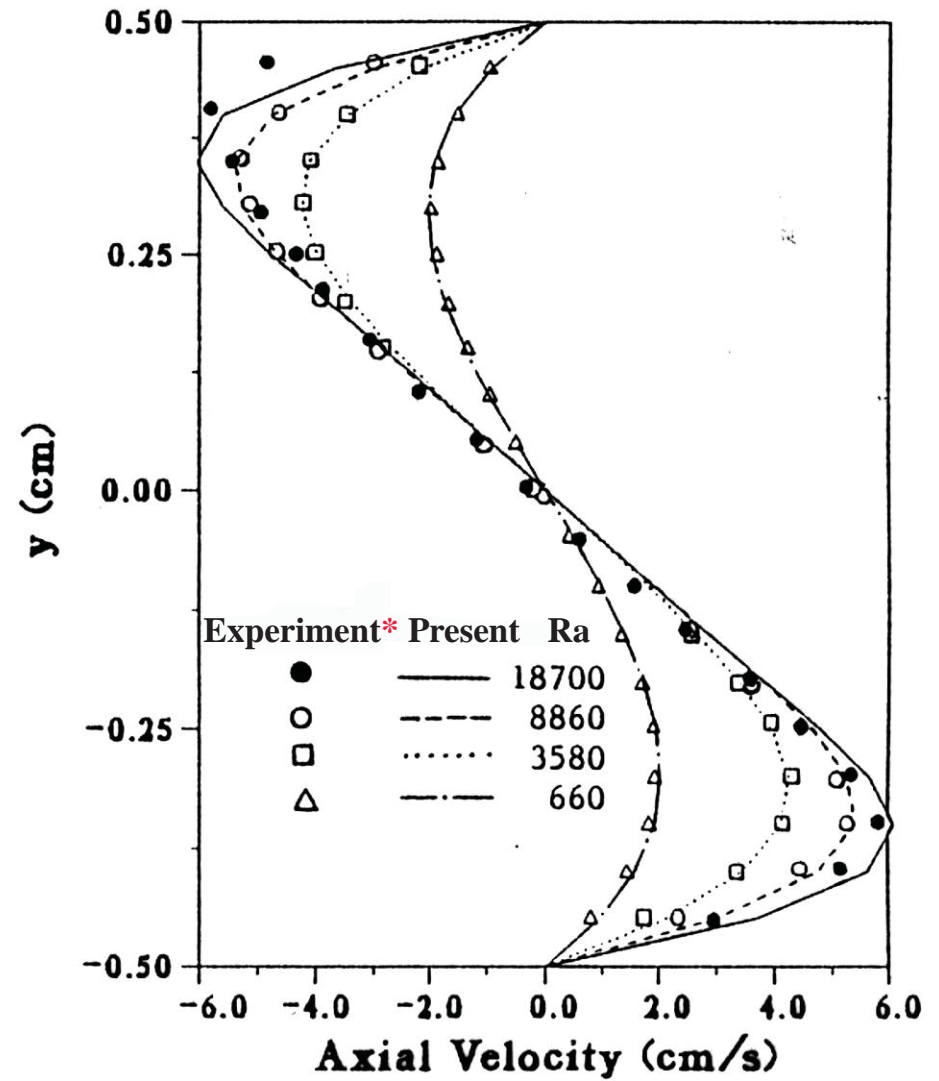
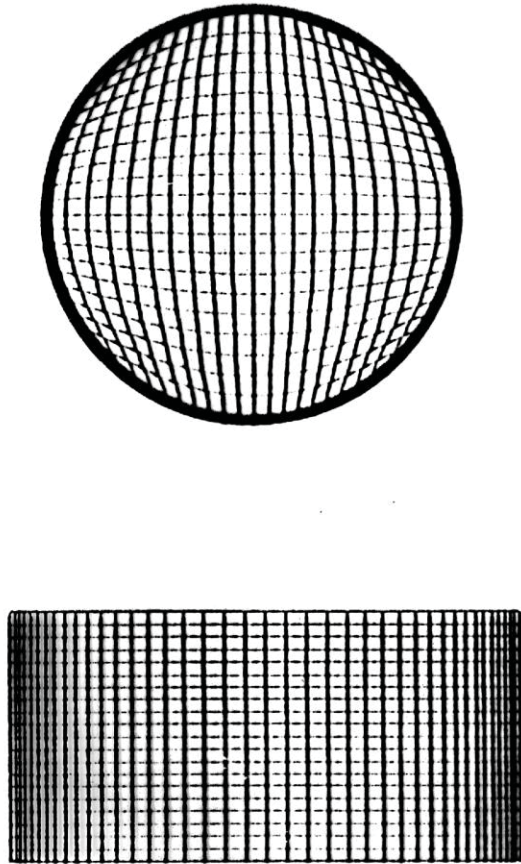


Benchmark → the H₂-I₂ System

- benchmark calculations by Rosenberger et al. (J. Crystal Growth 51 426 1981; 67 241 1984; 118 49 1992)
- source temperature $T_s=370.5$ K
- crystal temperature $T_c=358.1$ K
- ampoule pressure : 100 torr
- I₂ (M=254) is the deposited species and H₂ (M=2.016) is inert
- 2-d Cartesian system
- linear wall temperature
- quasi-compressible and Boussinesq calculations
- Peclet number analysis, $Pe \sim 1$ for diffusive flow
- growth rate results



3-D Computational Grid and Code Validation Results



* Schiroky and Rosenberger (1984)



Parameters of ZnSe System with Residual Gas

$$\alpha = 2.9$$

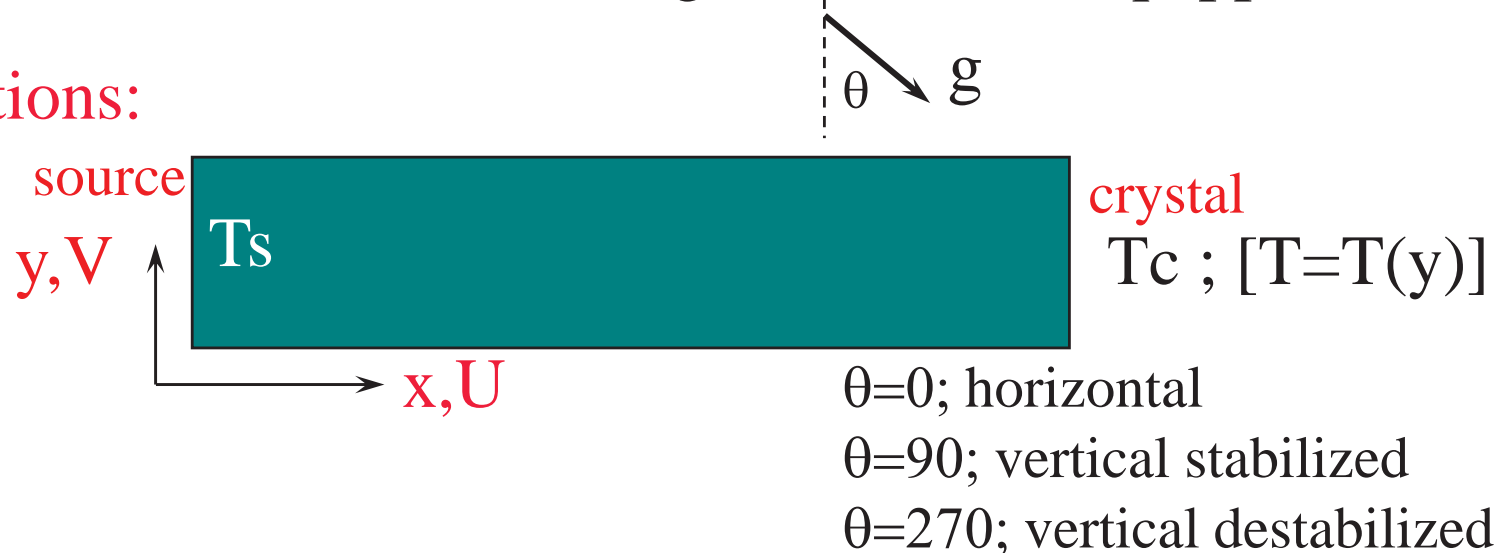
- density, $1.2 \times 10^{-5} \text{ g/cm}^3$
- dynamic viscosity, $4.3 \times 10^{-5} \text{ Pa-s}$
- kinematic viscosity, $36 \text{ cm}^2/\text{s}$
- diffusivity, Zn in N_2 , $64.59 \text{ cm}^2/\text{s}$
- diffusivity, Se_2 in N_2 , $71.46 \text{ cm}^2/\text{s}$
- thermal expansion coefficient, $7.1 \times 10^{-4} \text{ K}^{-1}$
- Prandtl number, 0.439
- Schmidt number, $\text{Sc}_{\text{zn}} = 0.557$
- Schmidt number, $\text{Sc}_{\text{se}} = 0.503$



Velocity Difference Plots - procedure

- calculations for pure diffusion limited conditions, 0g
- calculations for other g conditions, a conditions, etc.
- calculate differences in axial (u) and transverse (v) velocities at all identical grid locations between previous steps; e.g. $U(0g) - U(1g)$ and $V(0g) - V(1g)$
- contour the velocity differences and plot
- determine g-sensitivity
- Note: all calculations were using the Boussinesq approx.

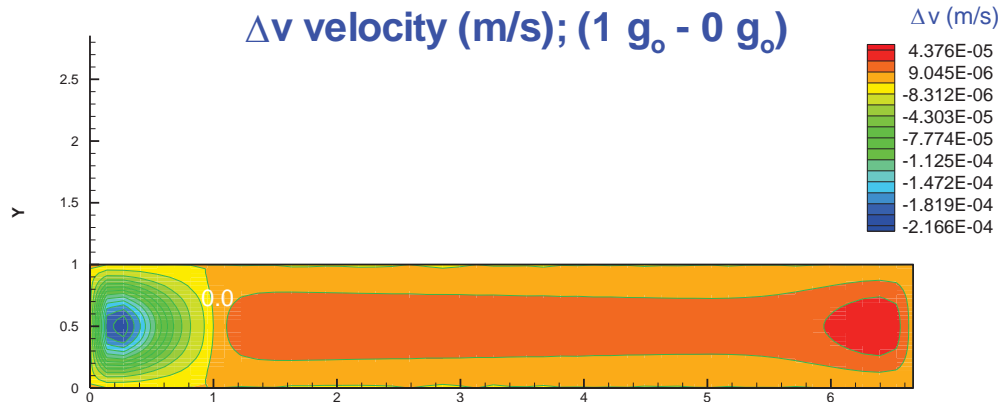
conventions:





Gravity Effect on Velocity Difference (horizontal case)

- g-effects with constant crystal temperature , T_c .
 $\Delta U \sim 0.3 \text{ mm/s}$; $\Delta V \sim 50 \text{ } \mu\text{m/s}$
- g-effects with crystal temperature variation, $T_c = T_c(y)$.
 $\Delta U \sim 0.3 \text{ mm/s}$; $\Delta V \sim 43.75 \text{ } \mu\text{m/s}$



- g-sensitivity (horizontal case) based on max. buoyancy driven flow normal to growth direction is 10% of crystal growth rate (3mm/day or $0.035 \text{ } \mu\text{m/s}$)

transverse acceleration requirement: $\sim 1 \times 10^{-4} g_o$



Gravity Effect on Velocity Difference (vertical case)

- g-effects with crystal temperature variation, $T_c = T_c(y)$.

Stabilized orientation: $\Delta U \sim 23.1 \mu\text{m/s}$; $\Delta V \sim 9.4 \mu\text{m/s}$

- g-effects with crystal temperature variation, $T_c = T_c(y)$.

Destabilized orientation: $\Delta U \sim 18.1 \mu\text{m/s}$; $\Delta V \sim 9.4 \mu\text{m/s}$

As far as transverse velocity difference is concerned both vertically stabilized and destabilized orientations have similar effects

- g-sensitivity (vertical case) based on max. buoyancy driven flow normal to growth direction is 10% of crystal growth rate (3mm/day or $0.035 \mu\text{m/s}$)

longitudinal acceleration requirement: $\sim 2.7 \times 10^{-3} g_0$



ZnSe with Residual Gas - 3D Calculation Results

- significant flow observed along the ampoule axis (z-direction) indicative of more deposition in the central area than near the walls
- velocity contours in the cross planes(x-y) show appreciable variation only near the end walls (source and crystal)
- species (Zn and Se_2) show fairly uniform distributions in the cross planes
- predicted crystal growth rate from 2-D and 3-D calculations are in fair agreement



Summary of Theoretical Calculation

- 2D and 3D calculations performed for ZnSe system
- Residual gas effects considered
- Calculations show that shear flow velocities of **10 to 50 microns/s** are induced by buoyancy effects (290 to 1400 times growth rate)
- g-level requirements established based on time scale analysis
required transverse g level: $< 1.2 \times 10^{-4} g_0$
required longitudinal g level $< 8.5 \times 10^{-3} g_0$
- It is noted that the Boussinesq model used in the calculations tend to underpredict velocities



Flight Experiments on International Space Station

- The flight experiments will be conducted in the Low Gradient Furnace (LGF) in the Microgravity Science Research Rack (MSRR) on International Space Station (ISS)
- Nine different growth runs will be performed for ZnSe, Cr-doped ZnSe, ZnSeTe, ZnSeS and ZnCdSe materials with different growth parameters.
- The flight experiments are scheduled to commence in late 2015.





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- "Growth and Characterization of CdS Crystals". J. Crystal Growth, 101 221-225 (1990).**
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- "Photoluminescence of Vapor and Solution Grown ZnTe Single Crystals". J. Crystal Growth, 138 219-224 (1994).**
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- “Characterization of Semi-insulating CdTe Crystals Grown by Horizontal Seeded Physical Vapor Transport”, J. Crystal growth 191 377-386 (1998).**
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- “Point Defect Distributions in ZnSe Crystals: Effects of Gravity Vector Orientation during Physical Vapor Transport”, J. Crystal Growth 204 41-51 (1999).**
- “Vapor Growth and Characterization of Cr-doped ZnSe Crystals”, J. Crystal Growth 207 35-42 (1999).**
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- “Thermal expansion coefficient of ZnSe crystal between 17 and 1080 °C by interferometry”, Materials Letters, Vol. 63, 1475-1477 (2009).**