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UNITED NATIONS EDUCATIONAL, SCIENTIFIC AND CULTURAL ORGANIZATION



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SMR/382- 18

WORKSHOP ON SPACE PHYSICS:
"Materials in Microgravity"
27 February - 17 March 1989

"Development of an Advanced Vapour Growth Facility

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Please note: These are preliminary notes intended for internal distribution only.

Conditions
of vapor growth
depend on
material

} low temperatures ($70-170^\circ\text{C}$)
 $\alpha\text{-HgI}_2$; organic electrooptic materials

} medium temperatures ($T \leq 1050^\circ\text{C}$)
materials: II-VI; III-V (GaAs etc); IV-VI (IR)

Sequential development of the two parts of the facility

1. low temperature range

Advantages → direct observation due to transparent furnace

↓

Exact measurement of the growth rate (CCD-video) as a function of supersaturation, temperature and impurities concentration

Extremely important to understand growth mechanisms and optimize crystal perfection

TECHNICAL REQUIREMENTS OF ■ GROWTH APPARATUS

NASA Apparatus Viewgraph van d. Berg et al

Advantage: Crystal in the middle. Away from the walls.

Theory predicts at the walls
Solutal convection

Excellent electrical properties of the $\alpha\text{-HgI}_2$ crystal grown in space support this argument. Viewgraph van d. Berg et al

Slide

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Optimization of the space facility

NASA Vapor Growth Facility

- First to understand the importance of vapor growth under microgravity.
- Had to follow the hard constraints for the first Spacelab-flights.
- Manual control concept.
- Principal Investigator = Payload Specialist excellent combination.

New Facility

- Advantage, longer flight times (Eureca, Space Station)
 - Large single crystals for applications
Due to mechanical distortion, microgravity beneficial.

With increasing growth time, the average mass uptake increases strongly

CORNERSTONES OF FUTURE VAPOR GROWTH EXPERIMENTS IN SPACE

1. Purification and standardization of the starting material.
 - ↓
Collaboration with EG+G in the IML-Program
2. Measurement of the diffusion coefficient of this material in space.
 - ↓
GAS Experiment
3. Growth facility design LTV (?)
 - a) Volume of $\approx 1\text{t}$ for large crystals
 - b) Expanding cooling surface
 - c) Additional Reservoirs for partial pressure control of excess components and impurities
 - d) Diagnostics

d) Diagnostics (in situ)

- Interface Temperature Measurement

Combined with diffusion coeffic.
under Hg

MONITORING ACTUAL SUPERSATURATION

- Monitoring of growth rate
in various crystallographic
orientations (μ resolution)

↓ Possible collaboration with
other groups in several flights.

Use of know-how from this facility
to develop a high temperature facility
($\approx 1000^\circ C$) for vapor growth of Li_2O
and ethzn II-VI and III-V
compounds
for stabilizations.

(characteristic Elements of the new facility

1. Higher stoichiometry

Reservoirs of the elements
to establish certain Hg/I ratio

2. Optimized Geometry

Start with VAN DEN BERG - NASA
Aspect Ratio

3. Measure + Control Supersaturation

Pyrometric measurement of
the crystal SURFACE TEMPERATURE

4. Decrease Thermal Stresses

Microprocessor controlled
expanding cooling surface
(same rate as lateral crystal growth)
COAXIAL COOLING RINGS
Accuracy $\pm 100^\circ C$

- 5 Growth Rate Measurement

2 Video Cameras; Stepwise scanning
Mathematic reconstruction of the position
of various edges; Comp. Assisted TESI

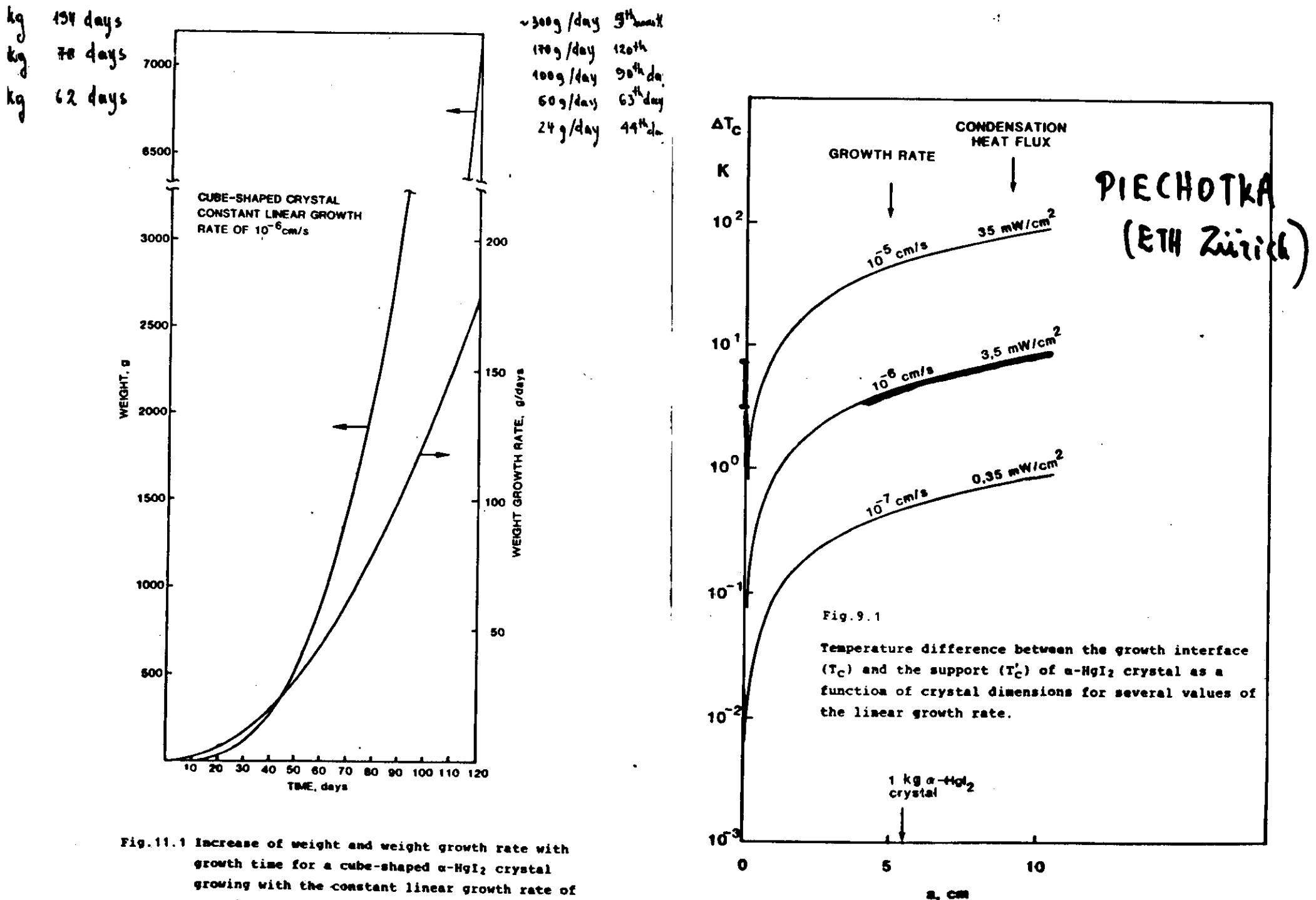


Fig. 11.1 Increase of weight and weight growth rate with growth time for a cube-shaped $\alpha\text{-HgI}_2$ crystal growing with the constant linear growth rate of $1 \cdot 10^{-6} \text{ cm/s}$.

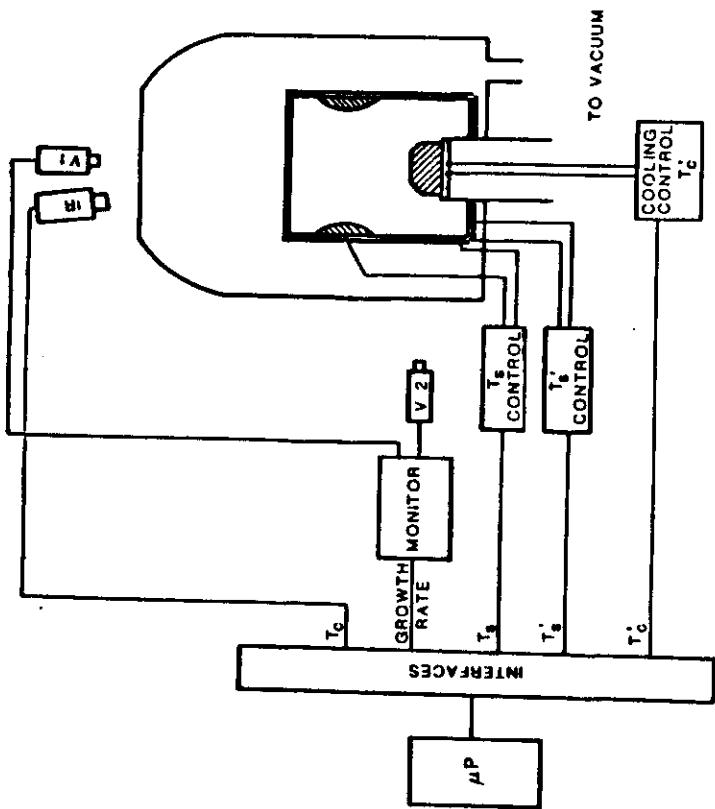


Fig. 9.2 Schematic diagram of the growth chamber and control units for vapor growth at low temperatures (80 - 170°C), suitable for the growth of α -HgI₂ crystals, organic materials etc. For explanations see text.

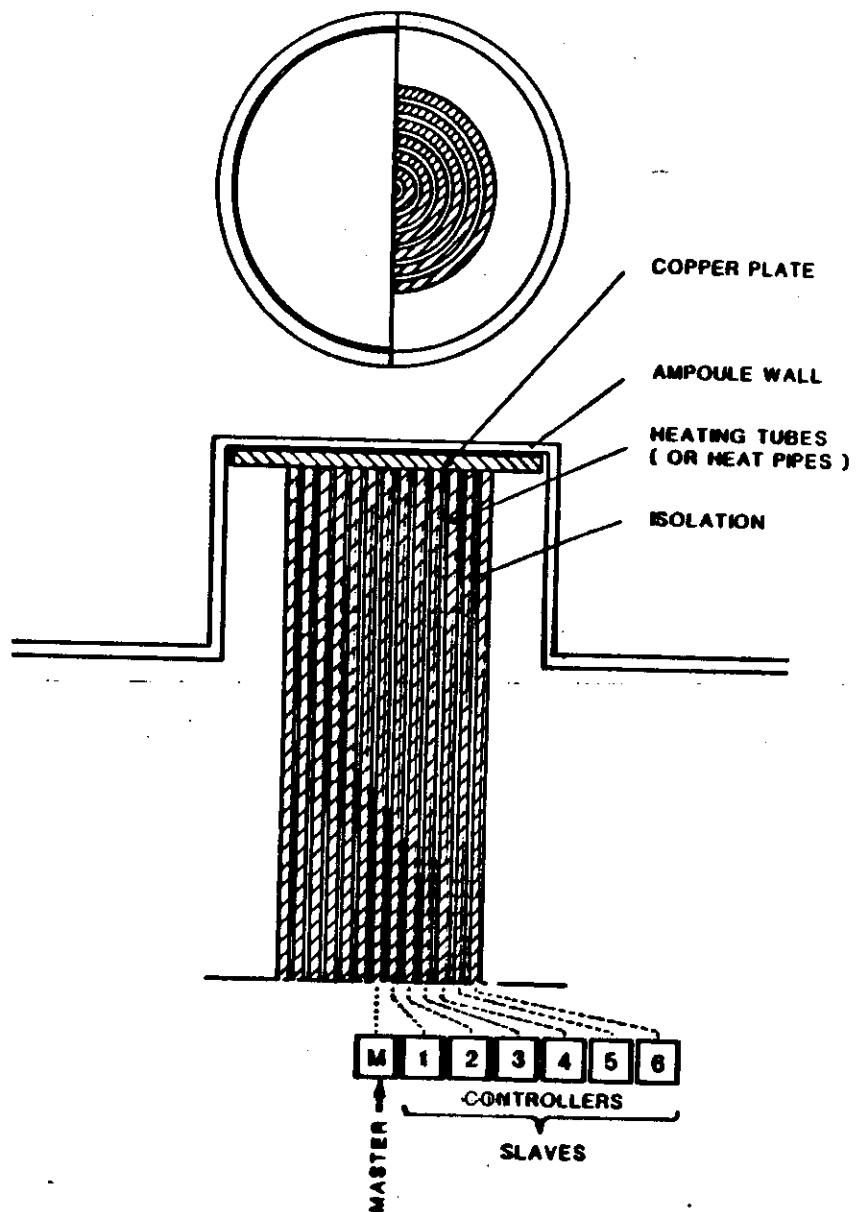


Fig. 9.3 Possible configuration of concentric rings for cooling of the crystal.

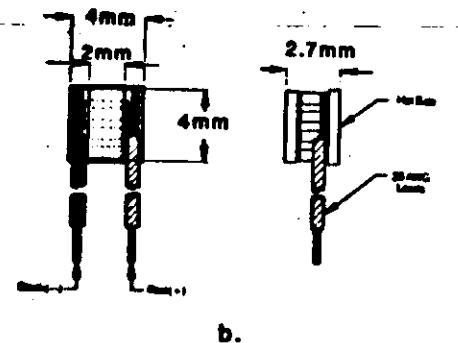
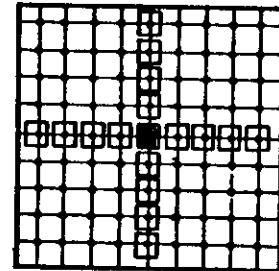
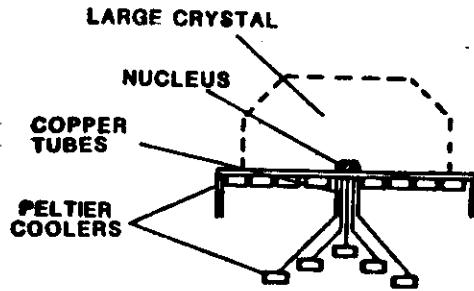
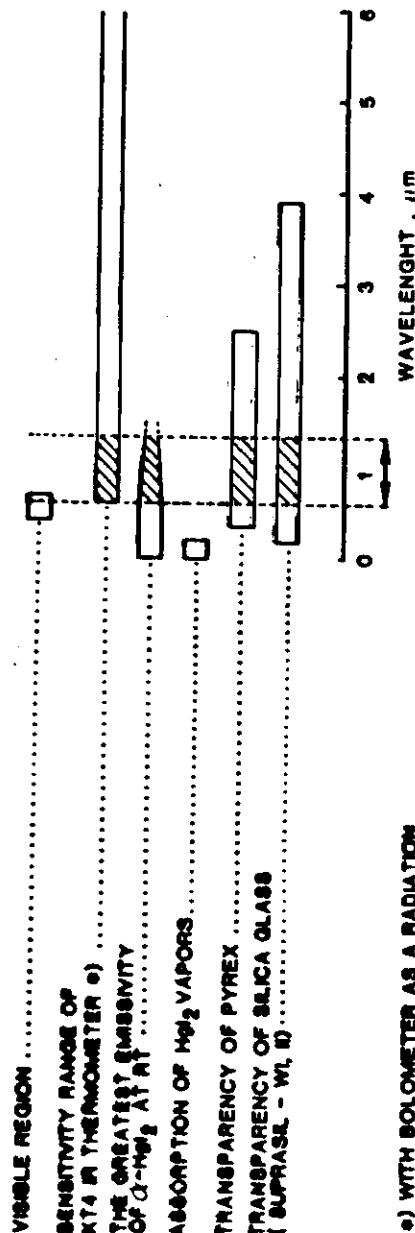


Fig.9.4 a) Possible configuration of the cooling array. It consists of 81 Peltier thermoelectric coolers connected in series forming five separately controlled concentric rings.
b) Elemental unit of the array. (After MIDLAND ROSS CORP., 1985).



e) WITH BOLOMETER AS A RADIATION DETECTOR SENSITIVITY INDEPENDENT ON WAVELENGTH WITHIN 0.8 — 40/μm RANGE.

Fig.9.3 Spectral characteristics of the crystal, vapor and walls of the growth chamber for vapor growth of α -HgI₂ crystals (T=300 K).

The new growth facility
should fulfill following experimental
possibilities to INCREASE CRYSTAL PERFECTION

1. (Independent of design) →

USE MATERIAL WITH HIGHER PURITY
+ STOICHIOMETRY

2. USE OPTIMIZED REACTOR GEOMETRY

3. DRIVE GROWTH WITH EXACTLY MEASURED
AND DIRECTLY CONTROLLED SUPERSATURATION
(Measure surface temperature to overcome the
influence of the thermal resistivity of large crystals)

+ DECREASE THERMAL STRESSES IN THE CRYSTAL
(microprocessor controlled, expanding, cooling
surface)

5 HIGH RESOLUTION GROWTH RATE MEASUREMENT
(for diagnostics of - growth mechanism
- formation of defects)