INTERNATIONAL ATOMIC ENERGY AGENCY
UNITED NATIONS EDUCATIONAL, SCIENTIFIC AND CULTURAL ORGANIZATION

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SMR/382- 12

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

"Impurities"

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Zurich, Switzerland

MAIN GOAL OF THE MATERIALS RESEARCH in conjunction with the VAPOR GROWTH OF PERFECT CRYSTALS of α-HgI_2

↓

Discovery and elimination of the sources of traps

Sources of Traps can be

1. mechanical (etch pit counting) } handling! many problems solved already
2. structural (rocking curves) }

3. purity dependent — Viewgraph ssms
4. nonstoichiometry dependent
5. crystal growth dependent

1. Plastic deformation

Critical resolved shear stress (CRSS)

0.5 lb/sq in ≈ 0.003 MPa

Georgeson + Milstein (1985)

$\frac{(V)}{(A)}_{crit} = 5.41$ $\qquad d_{HgI_2} = 6.4\ g/cm^3$

↓

crystal dimensions 2.5-3.0 cm $(30\ cm^3)$

plastic deformation under it's weight.

3. Purity

- SSMS high concentration of hydrocarbons.
- Main source iodine Viewgraph
- Where are the hydrocarbons located in the lattice?

Possible adsorption sites
- van d. Waals layers Viewgraph
- Channels in the lattice Viewgraph

In case of INTERCALATION changes of the lattice constants should become visible?

Viewgraph

No significant changes

Alternative explanation

Inhomogeneous incorporation of impurities, breaking the van d. Waals layers (Mosaic Structure) Viewgraph

NICOLAU

Work supporting this: Inclusions Viewgraphs

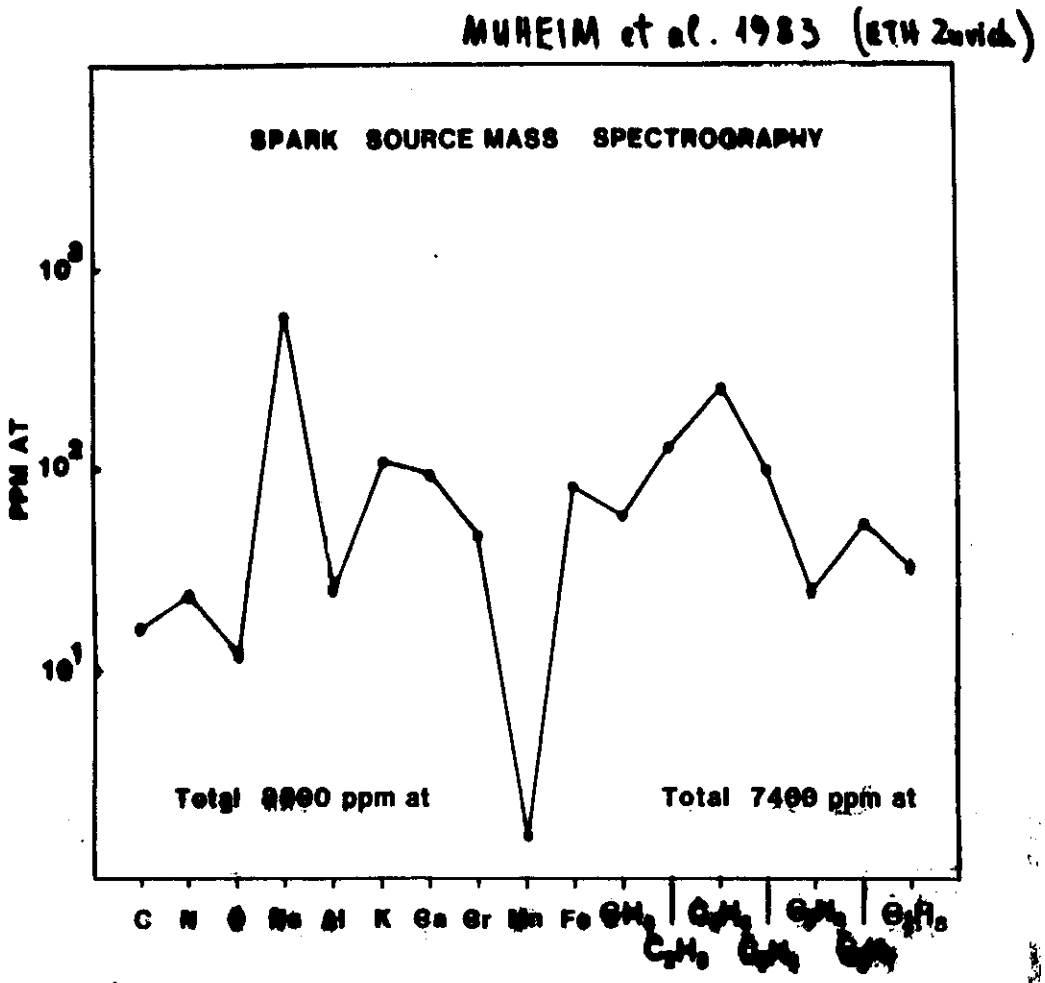
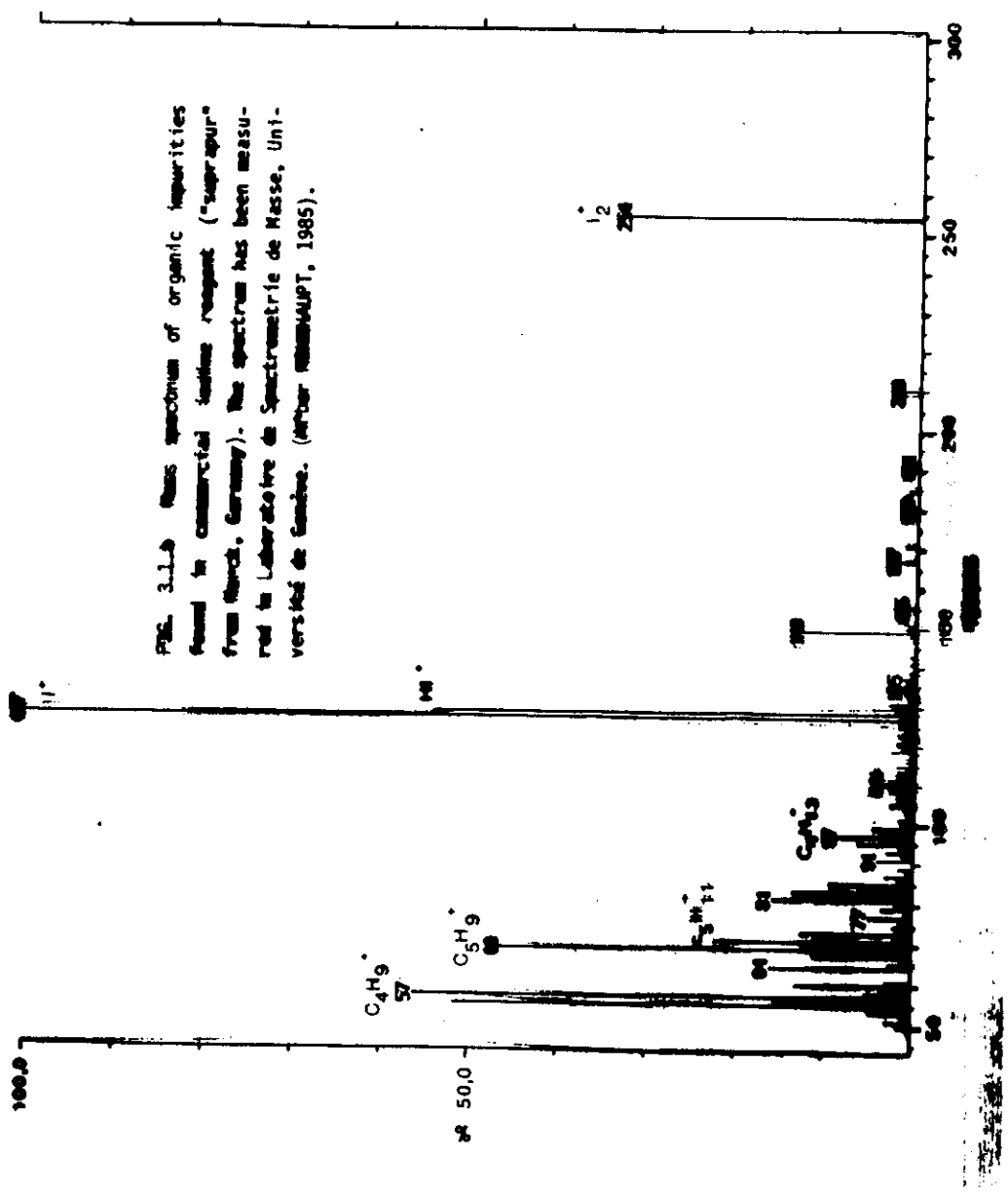


Fig.2 Typical concentration distribution of inorganic and organic impurities in mercuric iodide as measured by SSMS.



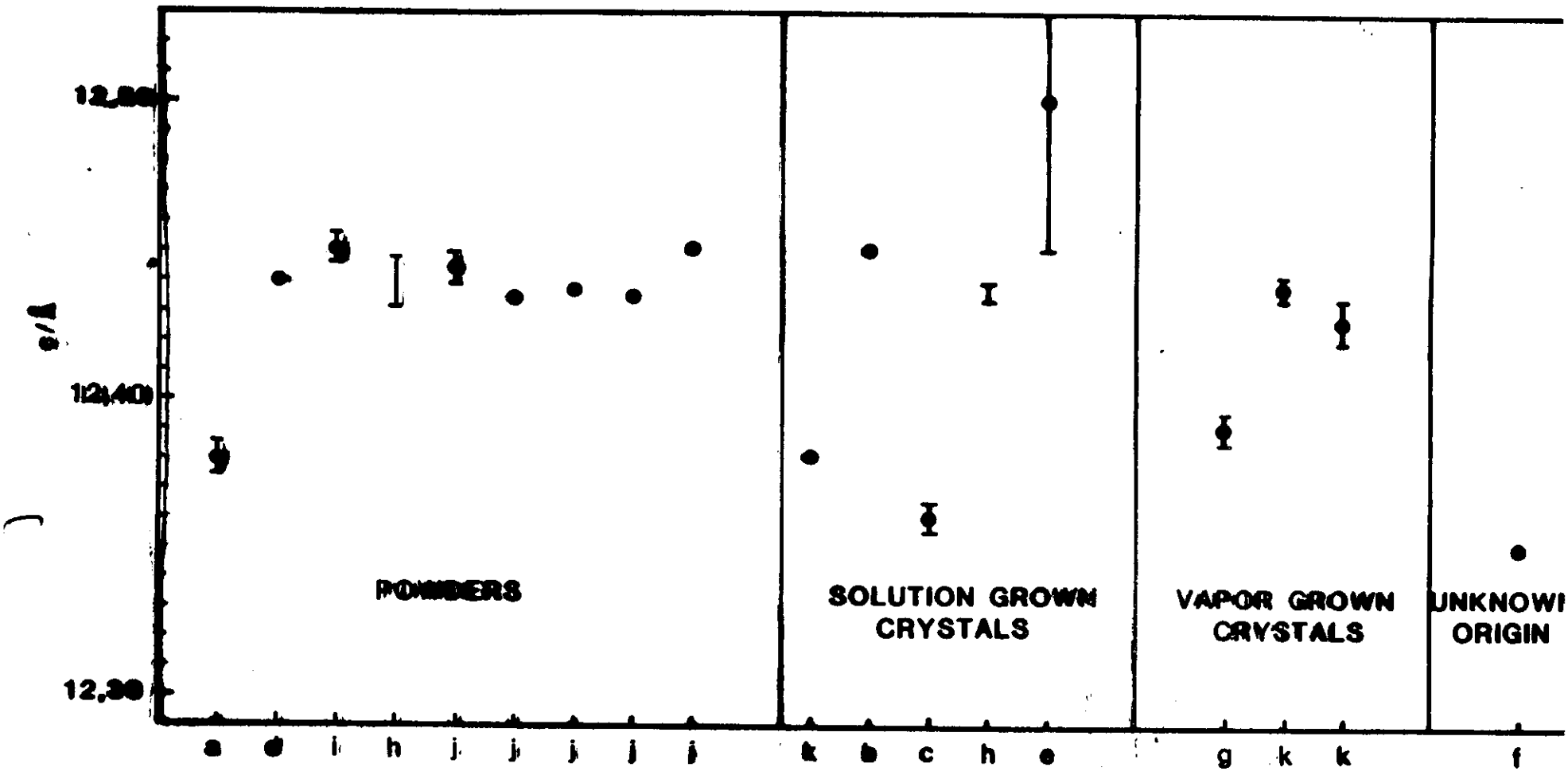


Fig. 4.5 b) Lattice constant c of the red mercuric iodide as measured since [illegible] by various authors. (For references see Tab. 4.2).

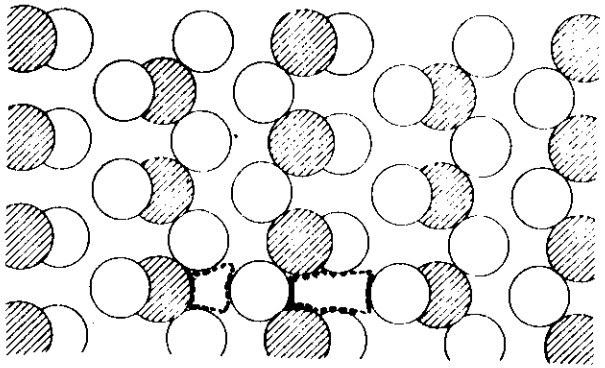


FIG. 4.6 Structure of α-HgI_2 as seen along a-axis (only the first three layers of atoms have been shown). 100% of covalent bonding between Hg and I atoms is assumed. Note two kinds of channels

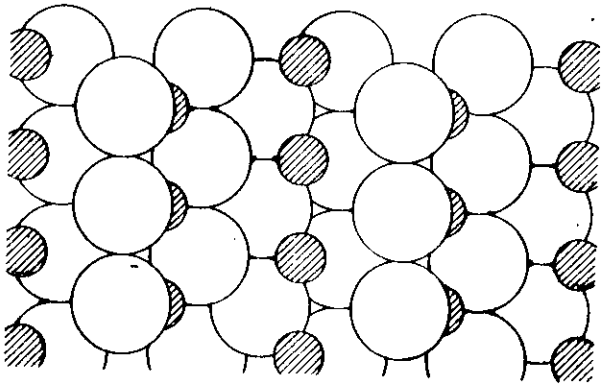


FIG. 4.7 Structure of α-HgI_2 as seen along a-axis (only the first three layers of atoms have been shown). 100% of ionic bonding is assumed.

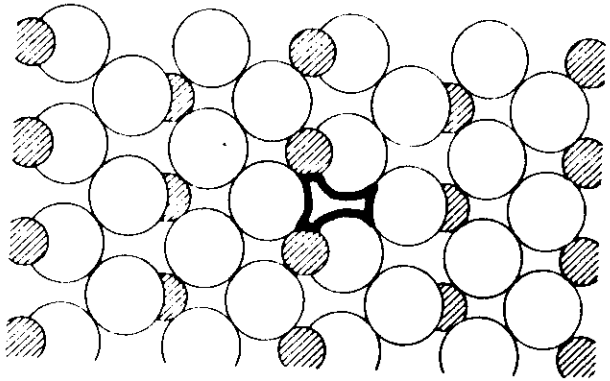


FIG. 4.8 Structure of α-HgI_2 as seen along a-axis (only the first three layers of atoms have been shown). 30% of ionic bonding is assumed.

channels few

Huth USC 1979

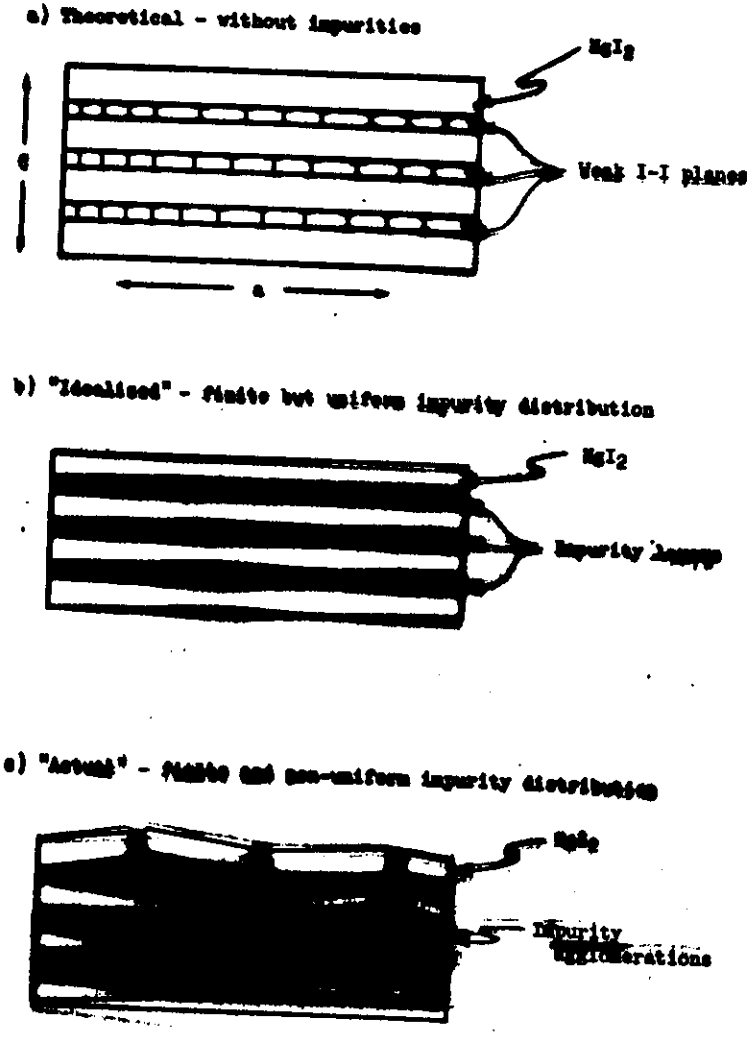


Figure 3.1

NICOLAU (Private Communication)

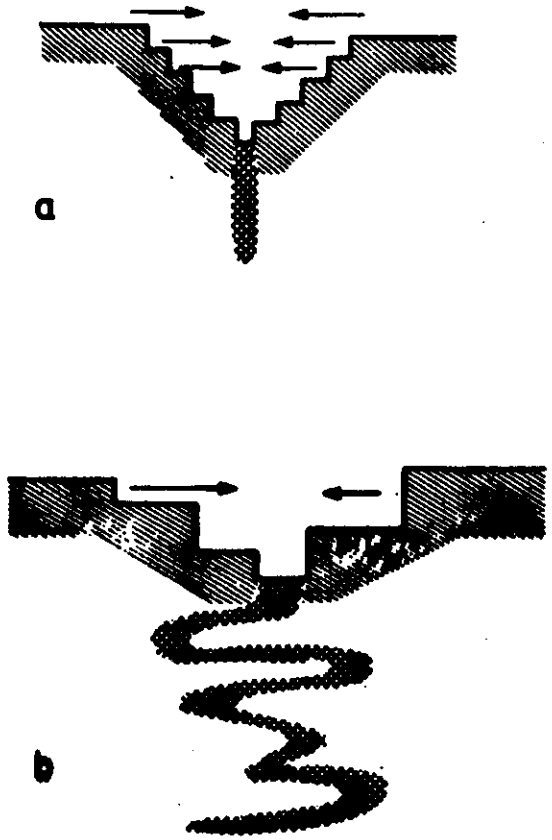


Fig.6.13 Valleys at the growth interface, schematic cross sections. The direction of step and terrace motion is indicated by arrows. At the valley bed, where the steps meet and annihilate, dopants and impurities may be preferentially trapped or rejected. The trace of the valley may be straight as shown in (a), when the steps moving towards each other are regular. The valley trace may "oscillate" in the latteral direction as shown in (b), when the step trains are irregular. After LU and BAUSER, 19[illegible].

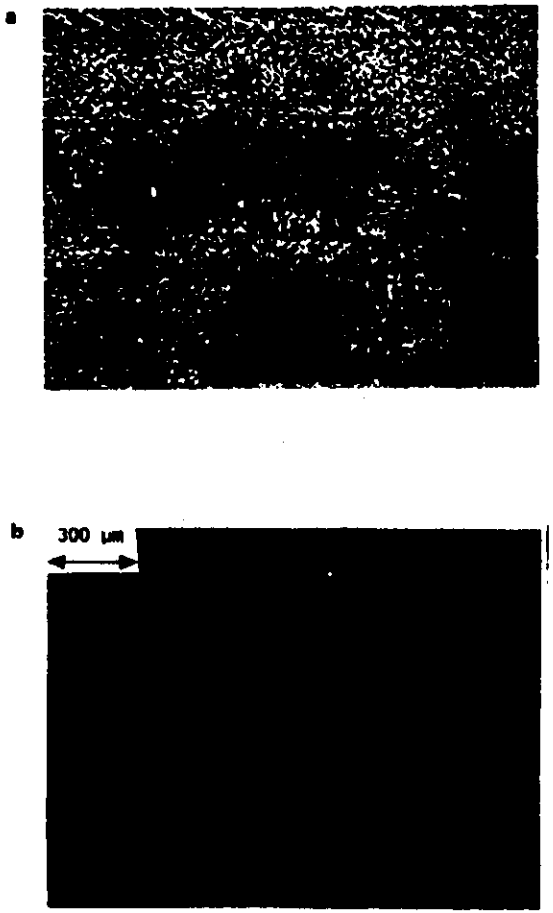


Fig.6.14 a) Straight valley traces on the as grown {110} face of a solution grown (LETI-30) α-HgI_2 crystal.Optical microscopy reflected light, x32 magnification.After NICOLAU,1986.

b) Sinusoildal shaped arrangement of foreign particles trapped in α-HgI_2 crystal grown in a vertical furnace by TOM method.After SCHIEBER et al.,1976.

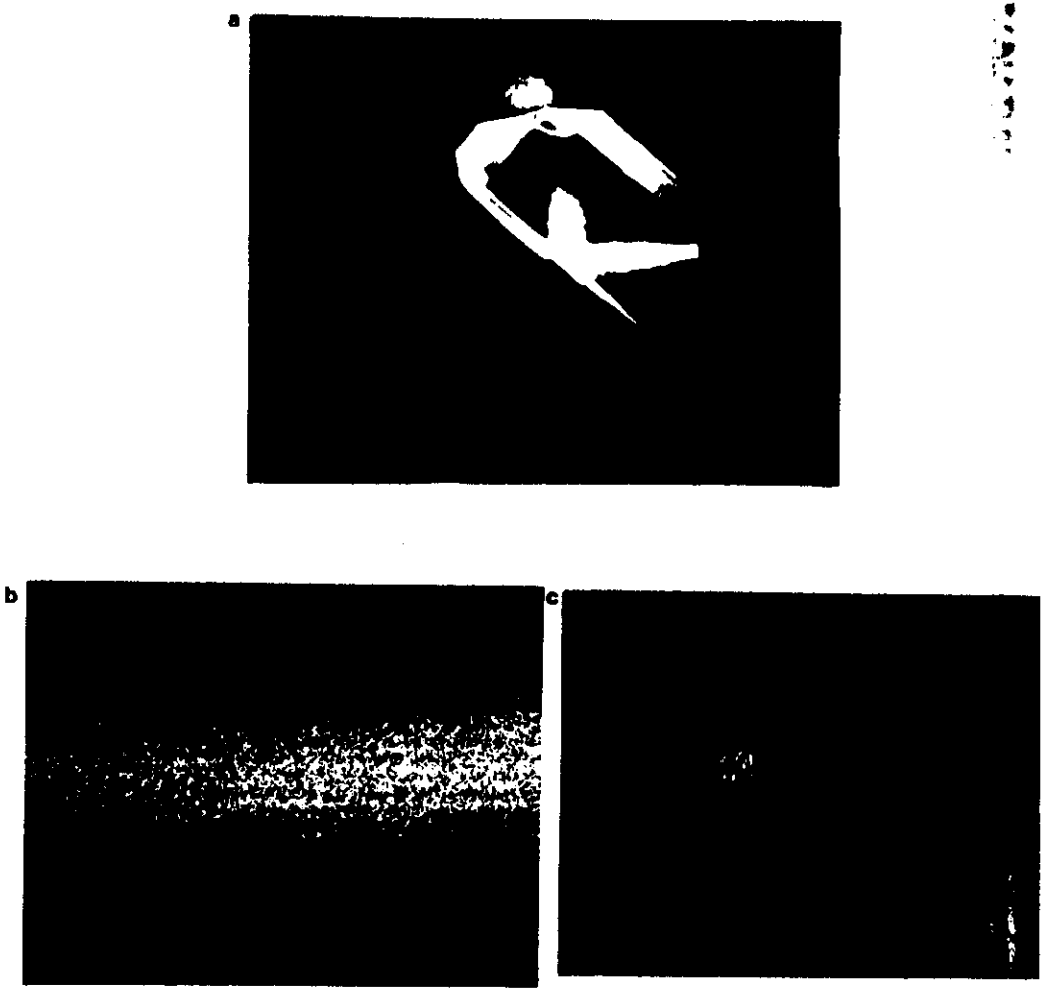
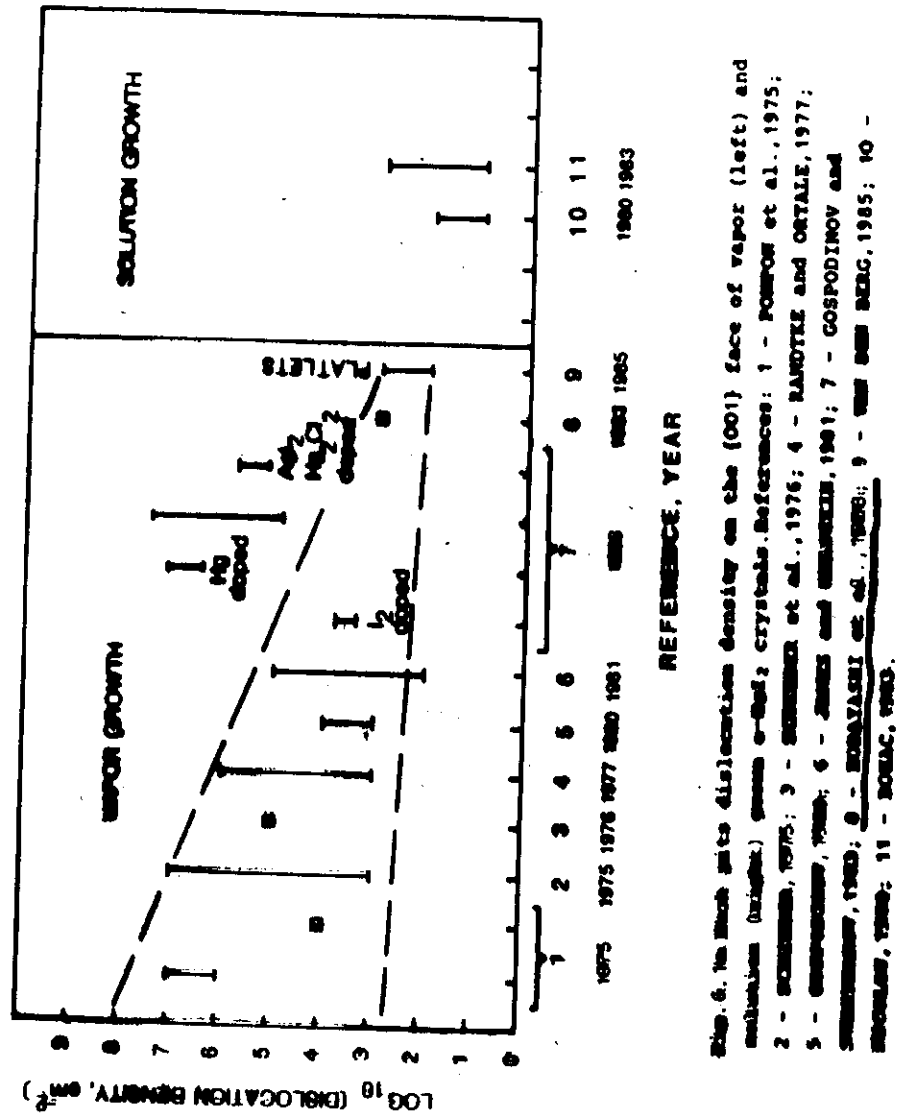


Fig.6.8 Optically clear solution grown (LETI-32) α-HgI_2 crystal (a) illuminated by a laser beam (λ=608-613µm) of 1mm diameter.Much more scattering of the beam is seen in the <110> (b) than in the <001> (c) direction.After NICOLAU,1986.

Fig.6.15 Voids (black dots) of 10-20μm diameter in a vapor grown (EG&G, S 6-24) α-HgI_2 crystal.Sawn and polished (110) plane optical image, x100 magnification,relflected light.After NICOLAU,1986.



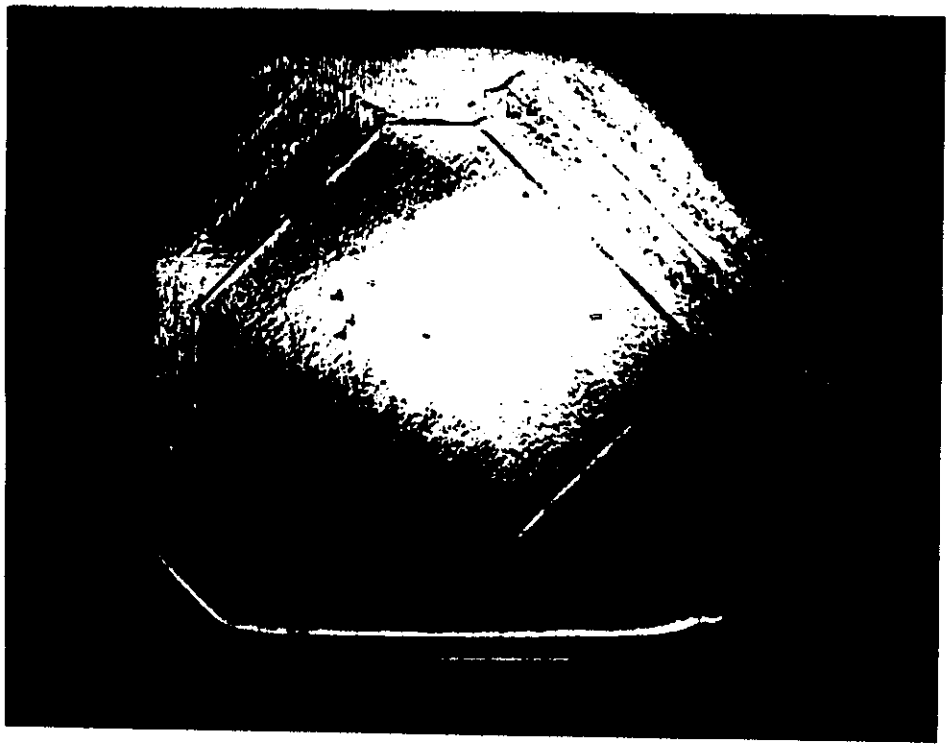


Fig.6.1d As grown crystal surface after polishing and etching.Notice the low concentration of pyramidal etch pits in the central part corresponding to a dislocation density of 10^3 cm^{-2}. Bands with high concentration of flat bottom etch pits appear at the outer parts.After KOBAYASHI et al.,1983.

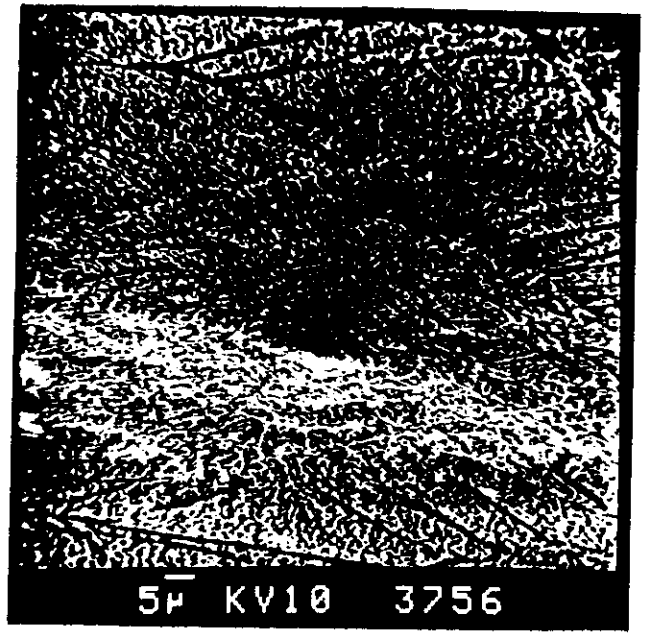


Fig.6.1b SEM micrograph which shows emergence point possibly due to a dislocation.After KOBAYASHI et al.,1983.

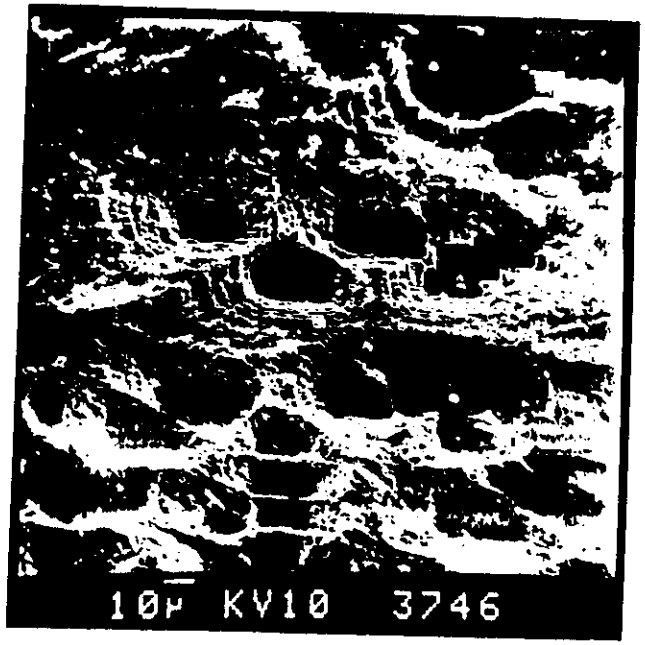


Fig.6.1c SEM micrograph of the flat bottom etch pits found in mercuric iodide crystals by KOBAYASHI et al.,1983.

Straight forward conclusion

A new, nonconventional PURIFICATION METHOD

is conditio sine qua non for further

improvement of the crystal perfection:

→ (Piechotka, Kaldis, 1983) <u>« Lattice Filtering »</u>

Use UHV, oil-free system (mercury pumps, 10^{-6} Torr at best)

Introduce iodine in a closed packed lattice, having no space for hydrocarbon intercalation

$I_2 + Cu \rightarrow CuI$ (fcc) → Purification → vapor growth

(carbon-rich residues rejected; conventional (sublimation zone-melting); (water clear crystals)

↓

decomposition

↓

in situ reaction of I_2 with $Hg \rightarrow HgI_2$

Drawback: very small scale production (100g/3 months!)

→ A much more efficient method (1kg/week) is now tested (Piechotka, Kaldis 1986)

Ampoule for crystal growth

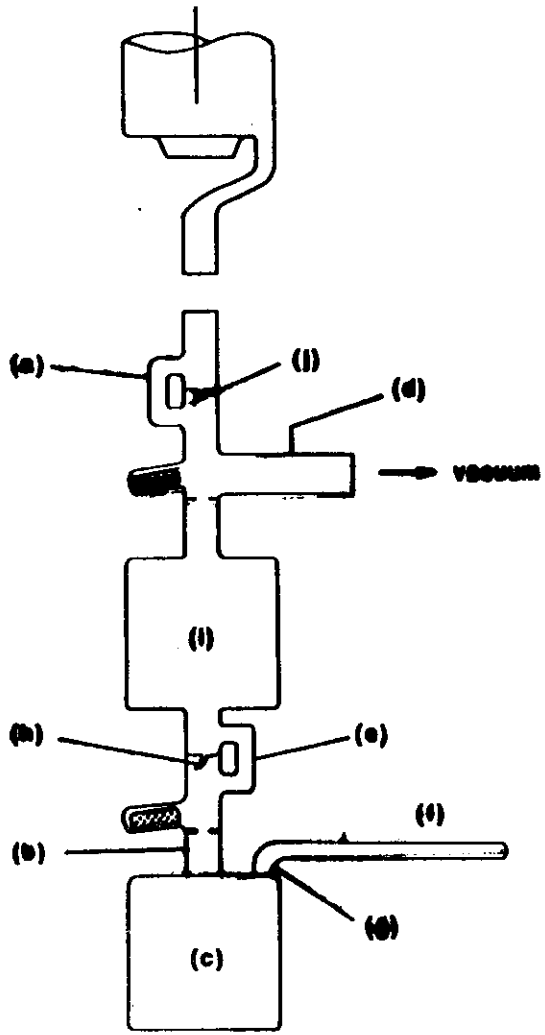


Fig. 26