Detached Melt and Vapor Growth of InI in SUBSA Furnace

A. Ostrogorsky\textsuperscript{1}, V. Riabov\textsuperscript{1}, M. P. Volz\textsuperscript{2}, L. van den Berg\textsuperscript{3}, A. Cröll\textsuperscript{4,5}

\textsuperscript{1}Illinois Institute of Technology, Chicago, IL, USA
\textsuperscript{2}EM 31, NASA Marshall Space Flight Center, Huntsville, USA
\textsuperscript{3}Constellation Technology, Largo, FL, USA
\textsuperscript{4}RSESC, The University of Alabama in Huntsville (UAH), USA
\textsuperscript{5}Crystallography, Albert-Ludwigs University of Freiburg, Germany
OVERVIEW

- Introduction
- SUBSA furnace
- Wetting angle results for InI on different substrates
- Melt growth and vaporr growth ampoule setups
- Summary
INI MATERIAL PROPERTIES

- Monovalent Indium (I) Iodide, InI, apart from its current uses in metal halide lamps and as reagent in organic synthesis, is a promising candidate for γ-ray and X-ray detectors

<table>
<thead>
<tr>
<th>Material</th>
<th>Cd_{0.9}Zn_{0.1}Te (CZT)</th>
<th>Hgl₂</th>
<th>InI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average atomic number, Z</td>
<td>49.1</td>
<td>62</td>
<td>51</td>
</tr>
<tr>
<td>Density, g/cm³</td>
<td>5.78</td>
<td>6.4</td>
<td>5.31</td>
</tr>
<tr>
<td>Band gap, eV</td>
<td>1.55</td>
<td>2.14</td>
<td>2.0</td>
</tr>
<tr>
<td>Melting point, °C</td>
<td>~1100</td>
<td>259</td>
<td>351</td>
</tr>
<tr>
<td>Structure</td>
<td>Zincblende</td>
<td>Tetrahedral-layered</td>
<td>Orthorhombic</td>
</tr>
<tr>
<td>Knoop Hardness, kg/mm²</td>
<td>92</td>
<td>10</td>
<td>27</td>
</tr>
<tr>
<td>Molecule Disassoc. Energy eV Herzberg’s tables [19]</td>
<td>1.2</td>
<td>0.35</td>
<td>3.43</td>
</tr>
<tr>
<td>Electrical Resistivity, Ohm-cm</td>
<td>3 x 10¹⁰</td>
<td>10¹³ to 10¹⁴</td>
<td>5 x 10¹¹</td>
</tr>
</tbody>
</table>
ADVANTAGES OF INI

• The low melting point and congruent sublimation allow both melt growth and vapor growth.
• InI is not toxic and not hygroscopic
PURITY OF THE STARTING MATERIAL

Illinois Institute of Technology and Radiation Monitoring Devices have developed considerable experience in purifying In and I, synthesizing InI from the elements, and purifying InI by zone refining.

100 g ingot, after 70 ZR passes IIT 2012

350 g ingot, was ZR and grown in an open boat, under dynamic gas flow 5% H₂ + 95 %Argon, RMD 2015.
INCLUSIONS

Small inclusions in the grown material are thought to be responsible for reduced electronic properties, compared to theoretical values.

CZOCHRALSKI

Density of precipitates in InI CZ01
Volume: 800x800x100 µm³
Black - last to freeze
Red - first to freeze

BRIDGMAN

Density of precipitates in InI Bridgman
Volume: 800x800x100 µm³
Black - last to freeze
Red - first to freeze
GROWTH IN MICROGRAVITY

- Allows vapor growth under purely diffusive conditions, which has shown to lead to a significantly increased $\mu$-$\tau$ product in the case of HgI$_2$ [1]

- Enhances the chance for detachment in the case of Bridgman growth to reduce stress in the crystal

“SUBSA” FURNACE

- Developed for a series of InSb experiments with a submerged baffle in the ISS MSG (microgravity science glovebox) rack

- One heating zone, low power consumption, $T_{\text{max}} = 950^\circ \text{C}$

- Transparent gradient zone (with ITO radiation shields) to visualize the growth interface

Photo courtesy of Scott Gilley, TecMasters
SUBSA FURNACE IN THE MSG

SUBSA Thermal Chamber

Cohu 3812 video camera

Process Control Module (PCM)
WETTING: SESSILE DROP TESTS

Fused silica

pBN
WETTING: SESSILE DROP RESULTS

- Wetting angle of InI on fused silica: 105±2.6°
  temperature coefficient: -0.64±0.06°/K (!), sample slides off the substrate easily

- Wetting angle of InI on pBN: 128±2.3°
  temperature coefficient: -0.03±0.03°/K, but sample sticks and reacts with the substrate
  (3InI + 2BN ⇌ In₃BN₂ + Bl₃ ?)

- Wetting angle of InI on Al₂O₃ ceramics: 101.6±2.1°
  temperature coefficient: -0.315±0.007°/K [1]

- Wetting angle of InI on carbon: 93.4±0.7°
  temperature coefficient: -0.102±0.006°/K [1]

http://hdl.handle.net/2128/3722
BRIDGMAN GROWTH AMPOULE

SECTION D-D
VAPOR PHASE GROWTH AMPOULE

Modified Markov method setup (semi-closed system)
SUMMARY

- InI has promising properties as a room temperature $\gamma$-ray detector
- The low melting point allows Bridgman growth and CZ growth, although diameter control is difficult with the latter method
- Determination of the wetting angles with different crucible materials showed the highest value ($128^\circ$) for pBN, but InI and pBN react, whereas fused silica ($105^\circ$) does not react. Other materials better suited for detachment have to be tested, e.g. DLC coatings.
- The congruent sublimation allows vapor phase growth in comparison to melt growth
- The melt growth and vapor growth ampoules were launched to the ISS on the Orbital ATK OA-7 Cygnus mission on 18 April, 2017.
- Sample processing on the ISS is expected in fall, 2017 or early 2018.
Funding for this project is provided by the Center for the Advancement of Science in Space (CASIS) and the National Aeronautics and Space Administration

A. Churilov, Radiation Monitoring Devices
G. Nelson, AG Scientific Glass Co.
S. Gilley, TecMasters
J. Quick, Jacobs Engineering
M. Kranz-Probst, U. Freiburg
J. Markert, U. Freiburg