A FLUIDIZED-BED REACTOR FOR SILANE PYROLYSIS

UNION CARBIDE CORP.

S. Iya

Summary of Activities

- PDU was modified to install a high-purity liner.
- A suitable liner support system was designed and implemented.
- Seed bed was prepared by screening and acid washing purchased silicon fines.
- A long-duration test run was conducted using polysilicon liner. Product from this run was single crystallized and analyzed for purity.
- A high-throughput test run was conducted using quartz liner.
- A cold model was constructed to investigate coarse particle withdrawal.
SILICON MATERIAL

Run Summary: Long-Duration Test With Polysilicon Liner

- 56 hours run duration followed by voluntary shutdown.
- 280 μm seed grown to 500 μm product.
- Silane feed concentration in the range 10 - 15%.
- Average deposition rate approximately 1 kg/hr.
- Bed temperature 650 - 750°C.
- U/UHF 3.5 - 4.0.
- Complete silane conversion within the bed.
- Several kg product was withdrawn.
- Fine powder 5.3% of silane feed.
- Power consumption 25 KWH/kg.

Long-Duration Run: Mass Balance

<table>
<thead>
<tr>
<th>Component</th>
<th>Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial bed weight</td>
<td>26.7 kg</td>
</tr>
<tr>
<td>Silicon in</td>
<td>63.0 kg</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>89.7 kg</td>
</tr>
<tr>
<td>Bed material withdrawn</td>
<td>83.5 kg</td>
</tr>
<tr>
<td>Powder in filter hoppers</td>
<td>3.8 kg</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>87.3 kg</td>
</tr>
<tr>
<td>Error in mass balance</td>
<td>2.7%</td>
</tr>
</tbody>
</table>

Run Summary: High Throughput Test With Quartz Liner

- 10 hours total run duration.
- Maximum silane feed concentration 48%
- Maximum deposition rate 3.8 kg/hr.
- Fine powder 6.9% of silane feed
- Power consumption 8 KWH/kg.
- Shut down caused by heater failure.
Test Product Characterization:
Long-Duration Run With Poly Liner

**PARTICLE PROPERTIES**
- 500 µm mean particle diameter
- 100 lb/ft³ bulk density
- Smooth, rounded surface
- Free flowing

**PARTICLE MORPHOLOGY**
- Dense deposition layer
- Layered ring-like growth structure
- Growth layer thickness ∼100 µm

**PARTICLE PURITY**
- Fe, Cr, Ni not detected by emission spec
  (Fe < 20 ppm, Cr < 5 ppm; Ni < 5 ppm)
- Samples of seed, intermediate & final product
  were sent to JPL for neutron activation analysis
- Single crystal resistivity 8 ohm-cm, P type
- FTIR measurements showed ppb levels of boron and phosphorus

**Plans**

- Additional purity runs starting with Union Carbide seed material.
- Product purity evaluation.
- Coarse product withdrawal tests.
- Technical and economic assessment.