SILICON MATERIAL AND JPL WEB TEAM

JPL IN-HOUSE FLUIDIZED-BED REACTOR RESEARCH

JET PROPULSION LABORATORY

N. K. Rohatgi

Objectives

- ESTABLISH ANALYTICAL TECHNIQUES TO VERIFY METALLIC IMPURITIES IN SILICON
- OBTAIN CLEAN SILICON SEED PARTICLES
- CONDUCT PURITY EXPERIMENTS
- DRAW SINGLE CRYSTAL Cz INGOT USING SILICON PARTICLES GROWN IN THE FLUIDIZED BED REACTOR.

Silicon Seed Particles

- SILICON PARTICLES OF LESS THAN 2 mm SIZE WERE PURCHASED FROM DYNAMIT NOBEL.
- THE PARTICLES WERE PREPARED BY MECHANICAL BREAKING OF SILICON ROD PRODUCED BY THE SIEMENS PROCESS.
- THE SIZE OF THESE PARTICLES WAS FURTHER REDUCED TO 200 TO 300 μM DIAMETER BY THE FLUID JET MILL.
- PARTICLES WERE ACID CLEANED AND DRIED UNDER NITROGEN BLANKET BEFORE FED INTO THE FLUIDIZED BED REACTOR.
Jet Milling Device for Seed Particle Generation

Silicon Seed Particles from Jet Mill

Nitrogen Flow Rate: 20 SCFM
Silicon Feed Rate: 40 gram/min
Silicon Cleaning Procedure

- Deionized water wash to remove fine silicon particles

- Cleaning in the mixture of two parts of 12N HCl and one part of 16N HNO₃ for 20 minutes.

- Washing with deionized water until effluent water is neutral.

- Etching with 48% HF for 20 minutes.

- Washing with deionized water until effluent water is neutral and has a resistivity of 16 megohms.

- Drying in diffusion furnace at °C under nitrogen blanket.

Fluidized Bed Cleaning of Silicon Seed Particles
Purity Experiment

- Seed particles were prepared via jet mill grinding of less than 2 mm size silicon particles purchased from the Dynamite Nobel.
- Particles were washed and cleaned via fluidized bed cleaning system.

Experimental Conditions

- Avg. initial seed particle size: 250 µm
- Initial bed weight: 11 kg (= 24" bed height)
- U/U_M = 4
- Silane concentration: 30% (in H₂)
- Bed temperature: 650°C
- Duration of run: 4.30 hrs.

Results

- Mass balance

  - Total silicon fed: 7.3 kg
  - Silicon deposited on particles: 6.8 kg (93.1%)
  - Silicon recovered as fines: 0.4 kg (5.5%)

- Production rate: 1.5 kg/hr.

- Particle growth: ≈ 10 µm (radius).
Purity of Silicon (PP$_{\text{na}}$)*

<table>
<thead>
<tr>
<th>ELEMENTS</th>
<th>RAW PARTICLES &quot;AS PURCHASED&quot;</th>
<th>JET MILLED AND ACID CLEANED &quot;SEED FOR FBR&quot;</th>
<th>FBR PRODUCT</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>0.2</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>Fe</td>
<td>20</td>
<td>≤0.6</td>
<td>≤0.6</td>
</tr>
<tr>
<td>Cr</td>
<td>0.05</td>
<td>0.03</td>
<td>≤0.02</td>
</tr>
<tr>
<td>Ni</td>
<td>10</td>
<td>&lt;0.5</td>
<td>&lt;0.5</td>
</tr>
<tr>
<td>Cu</td>
<td>0.06</td>
<td>≤0.02</td>
<td>&lt;0.02</td>
</tr>
<tr>
<td>Zn</td>
<td>&lt;0.02</td>
<td>≤0.04</td>
<td>&lt;0.04</td>
</tr>
<tr>
<td>Co</td>
<td>≤0.1</td>
<td>≤0.1</td>
<td>≤0.1</td>
</tr>
<tr>
<td>Mn</td>
<td>0.5</td>
<td>≤0.02</td>
<td>&lt;0.02</td>
</tr>
<tr>
<td>Na</td>
<td>≤0.1</td>
<td>≤0.1</td>
<td>≤0.1</td>
</tr>
<tr>
<td>Mg</td>
<td>≤1</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Al</td>
<td>2</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>S</td>
<td>≤1</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>K</td>
<td>≤0.07</td>
<td>&lt;0.1</td>
<td>&lt;0.1</td>
</tr>
<tr>
<td>Ca</td>
<td>0.6</td>
<td>0.1</td>
<td>0.1</td>
</tr>
</tbody>
</table>

*SPARK SOURCE MASS SPECTROSCOPY

Work in Progress

- DRAW SINGLE CRYSTAL CZ INGOT USING SILICON PARTICLES GROWN IN THE FLUIDIZED BED REACTOR.
- DETERMINE METALLIC IMPURITIES IN INGOT AND SILICON LEFT IN THE CRUCIBLE.
- FABRICATION OF SOLAR CELLS
- CHARACTERIZATION OF SOLAR CELLS