Pressure Cycle Leaching of High Density Carbon Capsules

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Successful implementation of the 5 μm fill tube led to achieving yield of over $10^{16}$ neutron yield in HDC capsules.

Reduced instability growth with smaller tube (HGR)

> 5 keV X-ray self emission (before hot spot stagnation)

N. Rice, et al. (GA)

Slide courtesy of Laura Berzak Hopkins & Vladimir Smalyuk
HDC capsules go through multiple fabrication and characterization steps

- Purchase Si mandrels
- Coat HDC on mandrels
- Polishing
- Shells arrive at GA
- Cleaning
- Leaching
- Laser Drilling
- Initial Metrology
- Final Metrology
- Build CFTA
- Delivery

Performed by Diamond Materials

Context of this presentation

HDC CFTA
Sonication had previously been used to leach HDC capsules

- Keyholes (~1mm): <1 day
- 10µm drill hole: ~3-4 days
- 5µm drill hole: ~1 month (or more!)
We developed pressure cycling to overcome the drill hole size dependence on leach time

- With pressure cycling, 89% of shells finish leaching in less than 5 days
- No dependence on drill hole size
- Leaching is no longer the rate-limiting step in HDC throughput

Average Days to Leaching Completion

*Pressure cycling data from 3/1/18 to 2/1/19*
The pressure cycling system is a repurposed GDP coating chamber fitted with timed valves.

- Capsule sits in open vial with etchant in vessel.
- Inlet line connected to nitrogen.
- Vent/outlet to atmosphere in fume hood.
- 50psi pressurization with nitrogen during operation.
The gas generated during Si etching plays an important role in etchant mass transfer

\[ 3Si (s) + 4HNO_3 (aq) + 18HF (aq) \rightarrow 3H_2SiF_6 (aq) + 4NO (g) + 8H_2O (l) \]
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Initial rate is slow, but increases with silicon surface area exposure.
Pressure cycling with initially unleached mandrels is the key to complete leaching

- Only 4 out of 23 partially leached shells completely finished leaching (17%)
- 90% of unleached 5µm shells completely finished leaching

"Unleached" = Pressure cycler only

"Partial" = Started leaching in sonicator and transferred to pressure cycler
Solid residue formed when etchant near drill hole evaporates

- “Crystal” residue seen forming outside of drill hole
  - Liquid leaks out of hole, evaporates, leaves behind precipitate
  - Only observed when taking shells out of leaching to check progress
Residue is composed of fluorine, nitrogen, and silicon

- EDS shows N, F, and Si
  - Potential formation and blocking of hole during leaching
  - Dissolves away in water
  - Removed by high temperature under N₂

“Stuck” shells can be recovered by running through a 700°C oven bake.
Over 200 shells have been leached with pressure cycling

- “Stuck” shells may be recovered through oven process
- No dependence on drill hole size
- Leaching is no longer the rate-limiting step in HDC production process

Days to Leach Completion

<table>
<thead>
<tr>
<th></th>
<th>Number of Shells</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-5 Days</td>
<td>Pressure Cycling</td>
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<tr>
<td>6-15 Days</td>
<td>Pressure Cycling</td>
</tr>
<tr>
<td>16-30 Days</td>
<td>Sonicator</td>
</tr>
<tr>
<td>30+ Days</td>
<td>Sonicator</td>
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