Pressure Cycle Leaching of High Density Carbon Capsules

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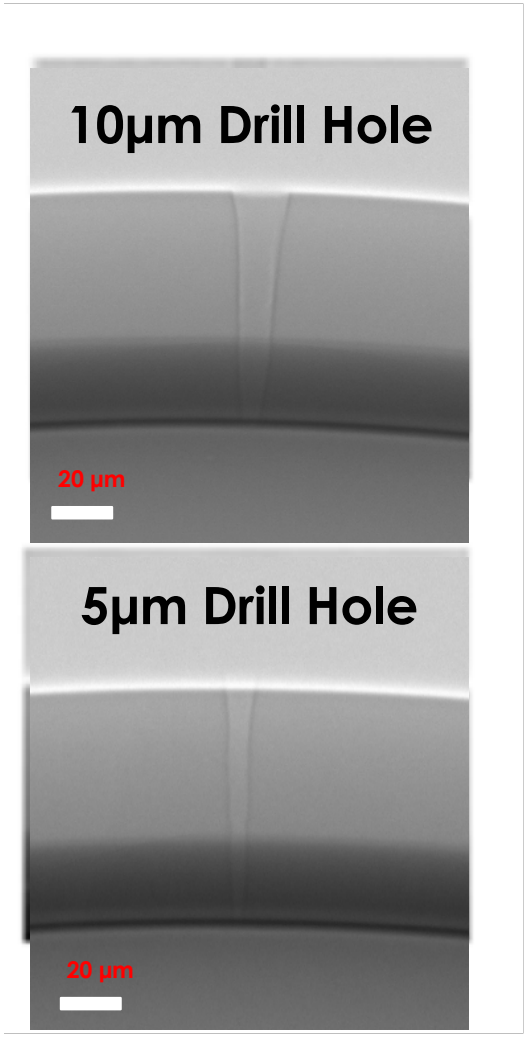
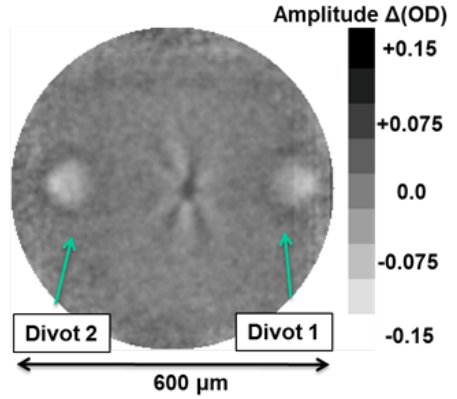
GENERAL ATOMICS

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

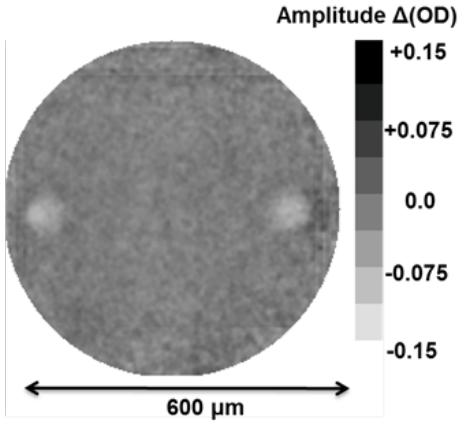


N. Rice, et al. (GA)

Reduced instability growth with smaller tube (HGR)

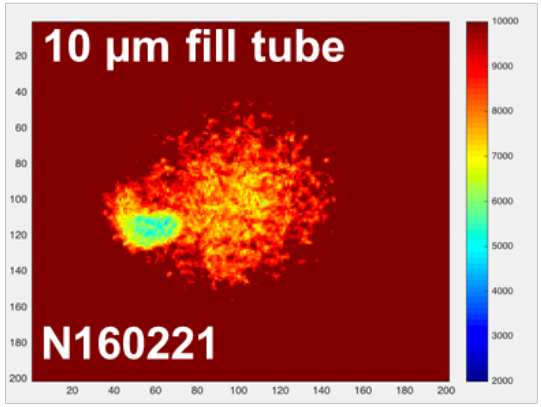


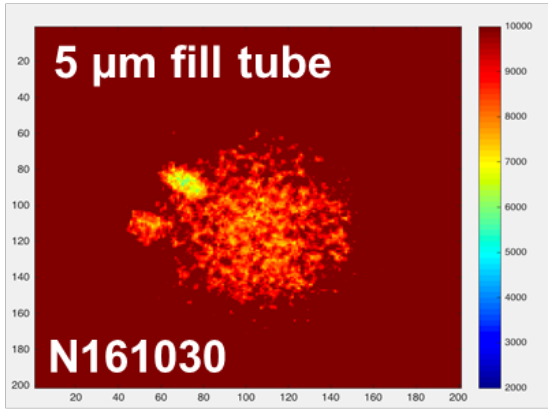
N160413



N170126

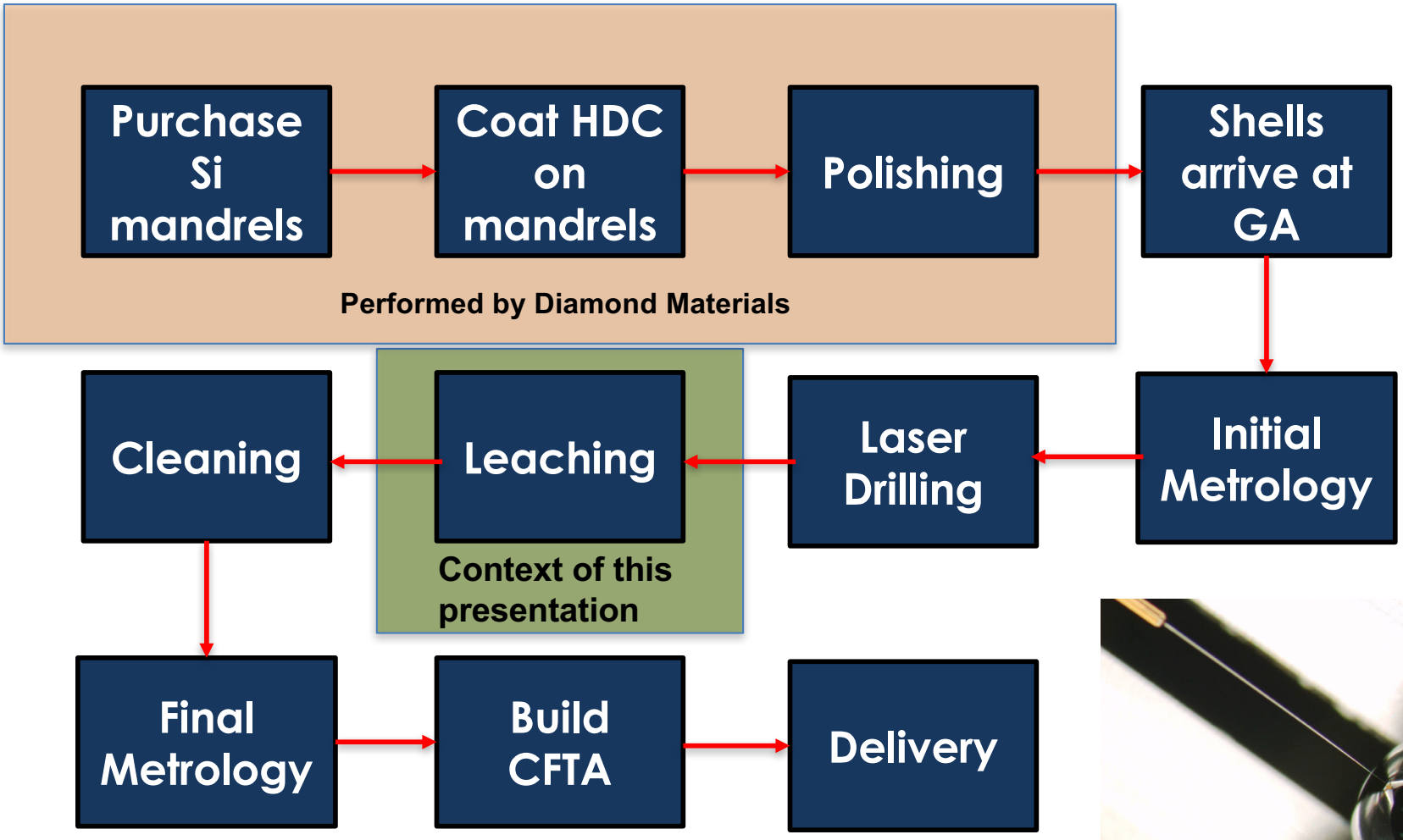
> 5 keV X-ray self emission (before hot spot stagnation)

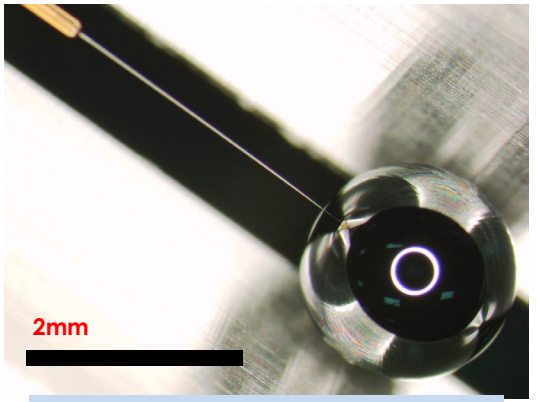




Slide courtesy of Laura Berzak Hopkins & Vladimir Smalyuk

HDC capsules go through multiple fabrication and characterization steps

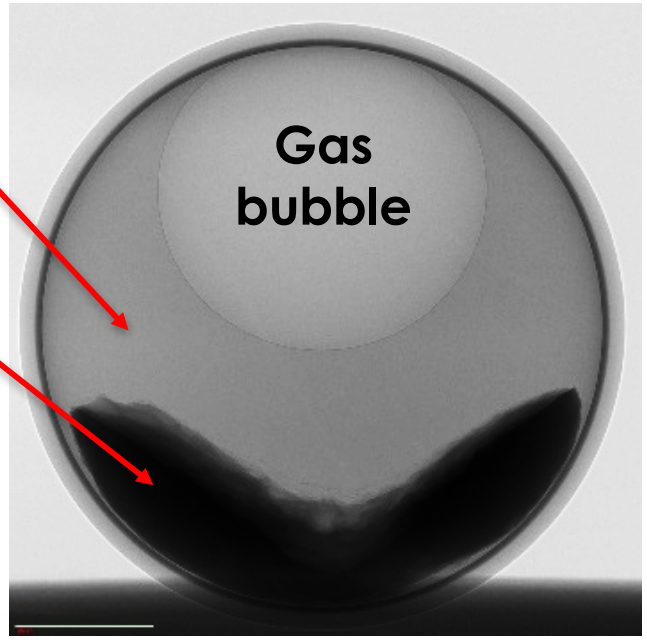


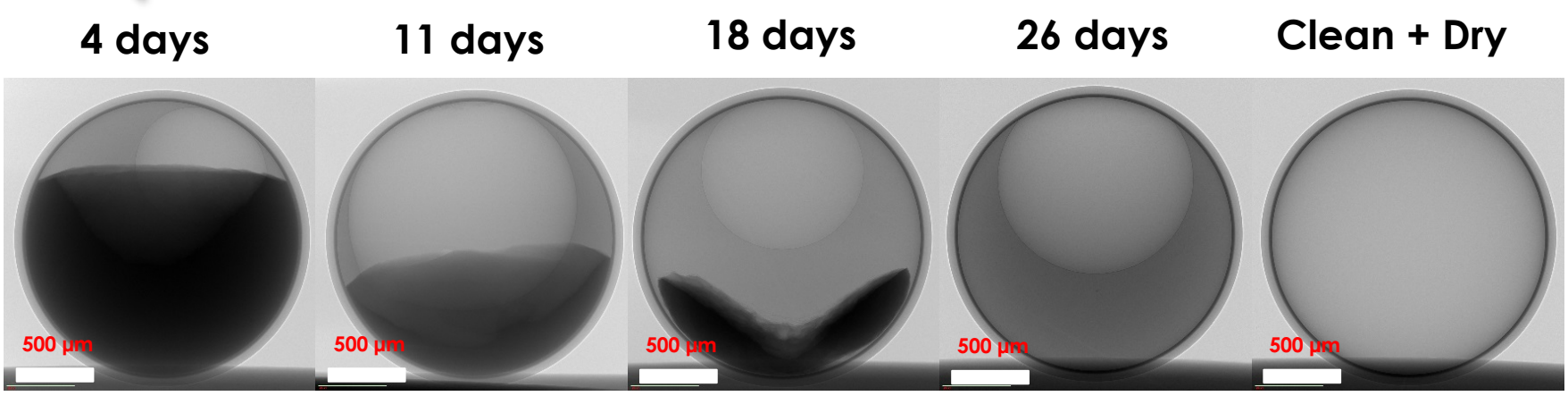


HDC CFTA

Sonication had previously been used to leach HDC capsules

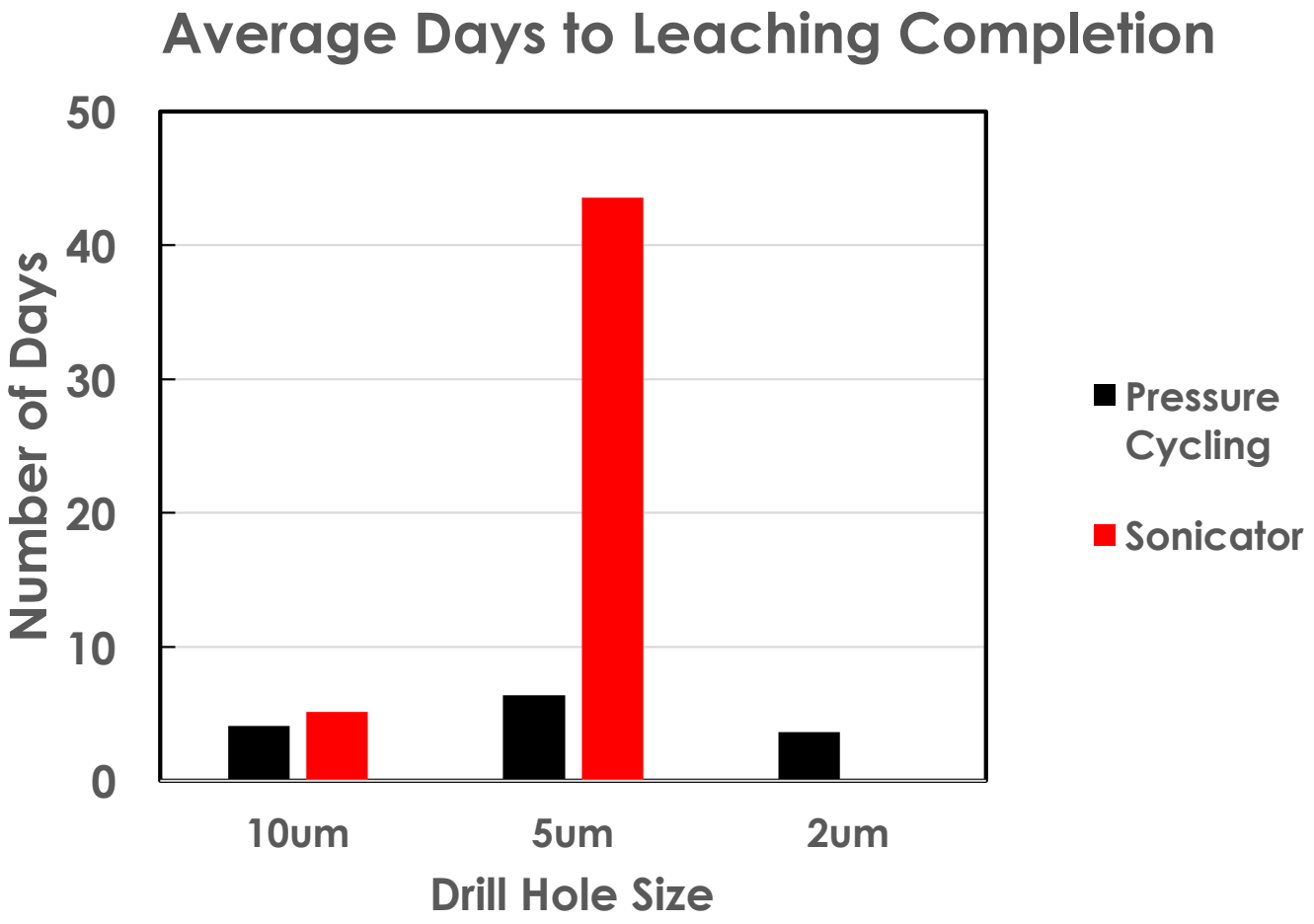
- Keyholes (~1mm): <1 day
- 10µm drill hole: ~3-4 days
- 5µm drill hole: ~1 month (or more!)





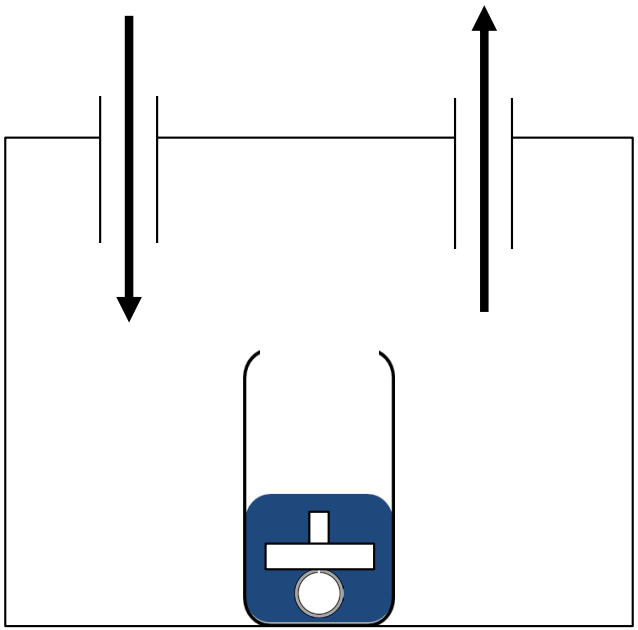
We developed pressure cycling to overcome the drill hole size dependence on leach time

- 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



*Pressure cycling data from 3/1/18 to 2/1/19

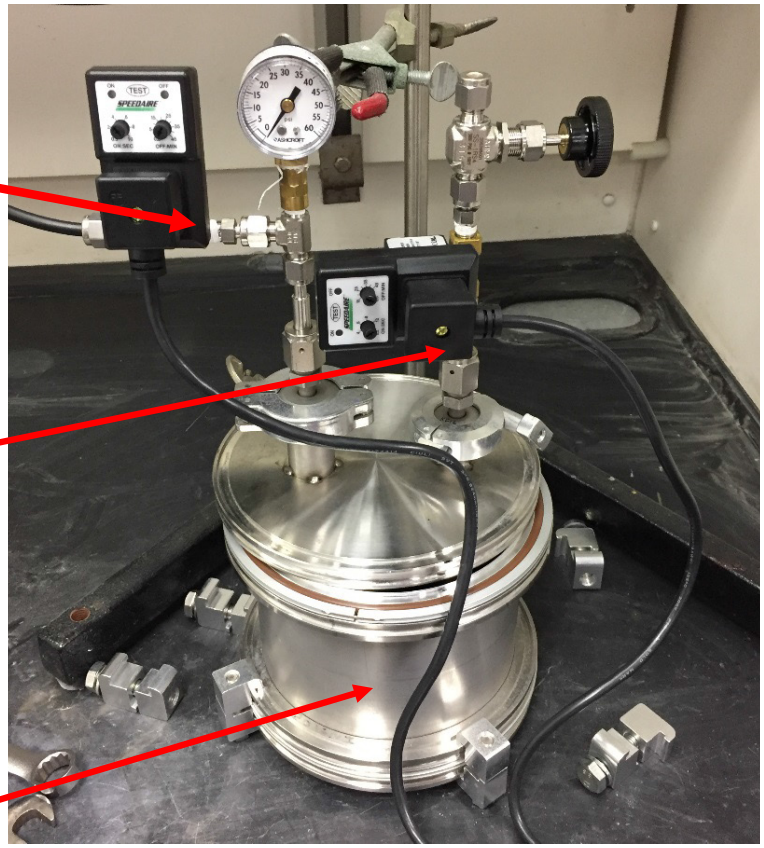
The pressure cycling system is a repurposed GDP coating chamber fitted with timed valves



Inlet line connected to nitrogen

Vent/outlet to atmosphere in fume hood

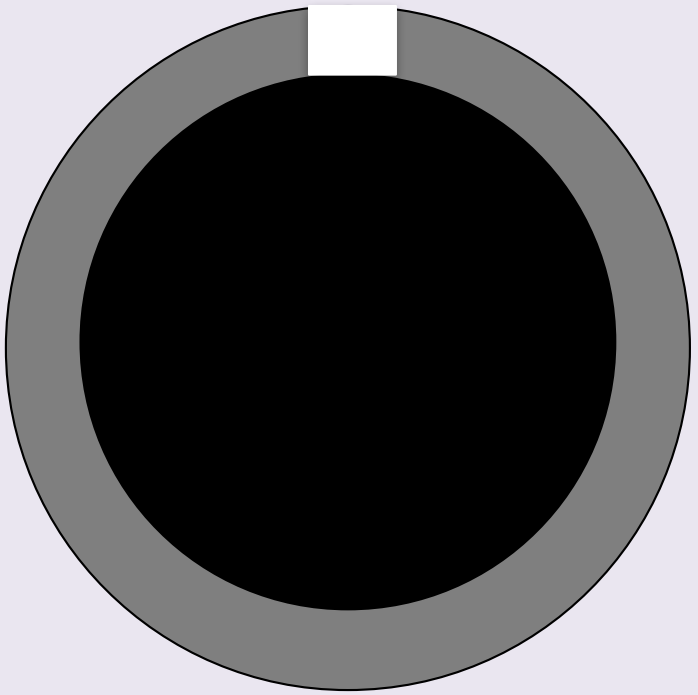
Capsule sits in open vial w/ etchant in vessel



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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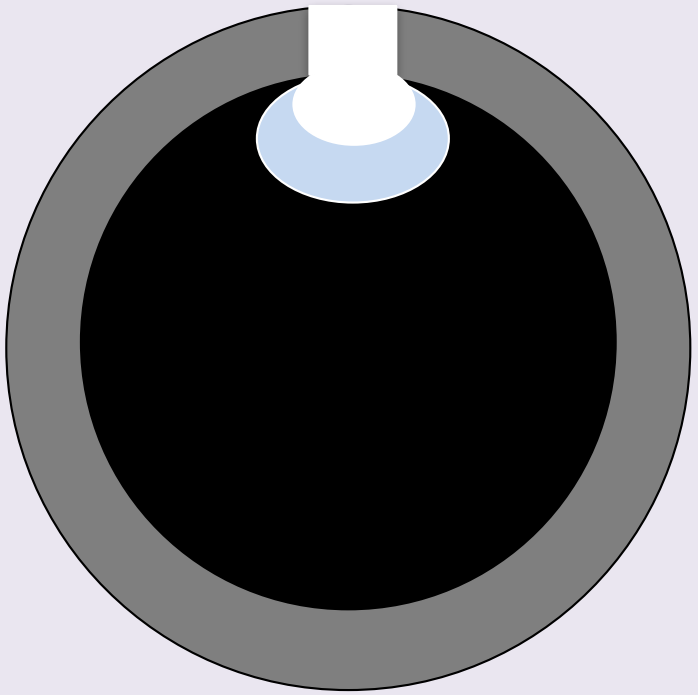

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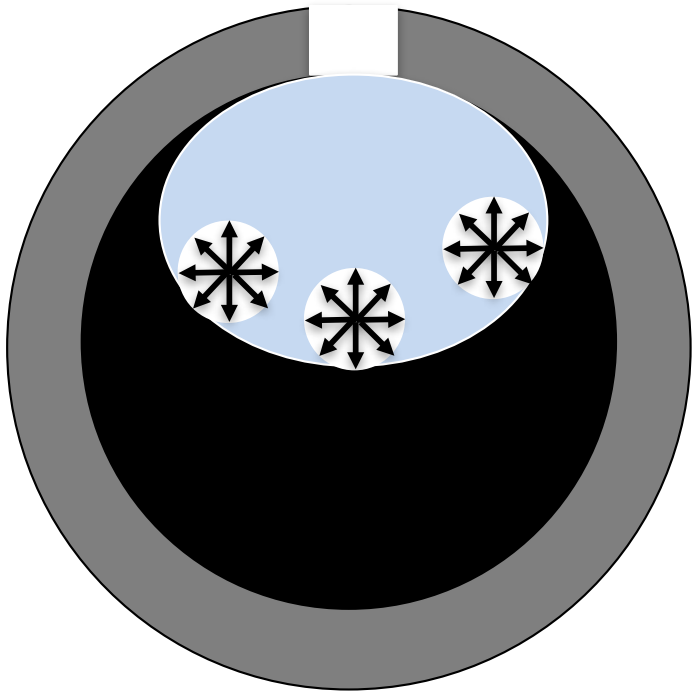

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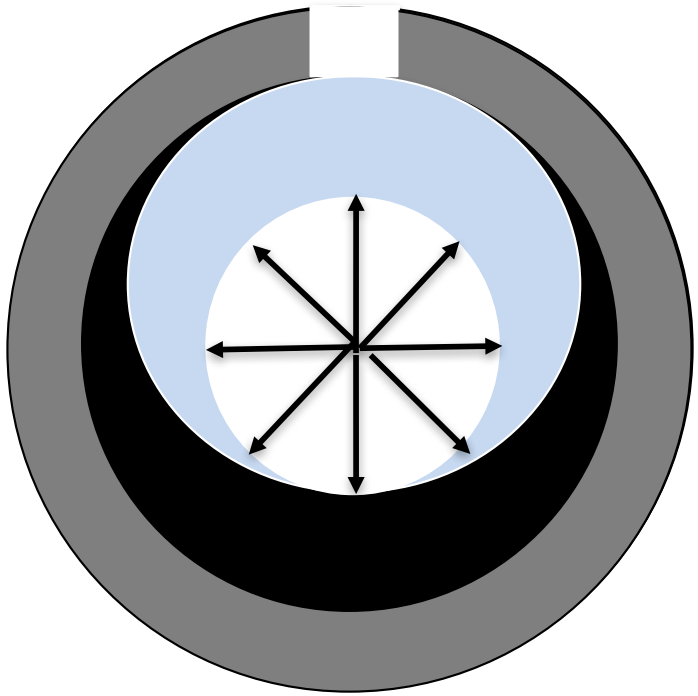

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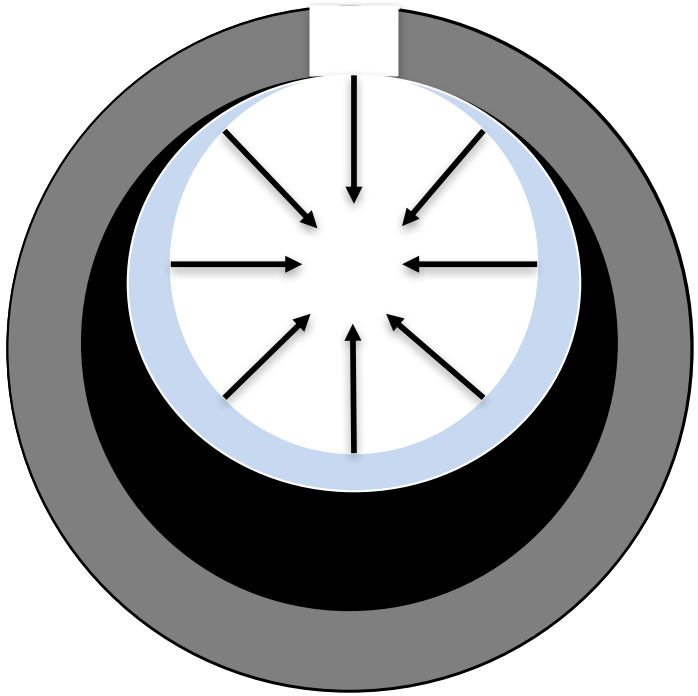

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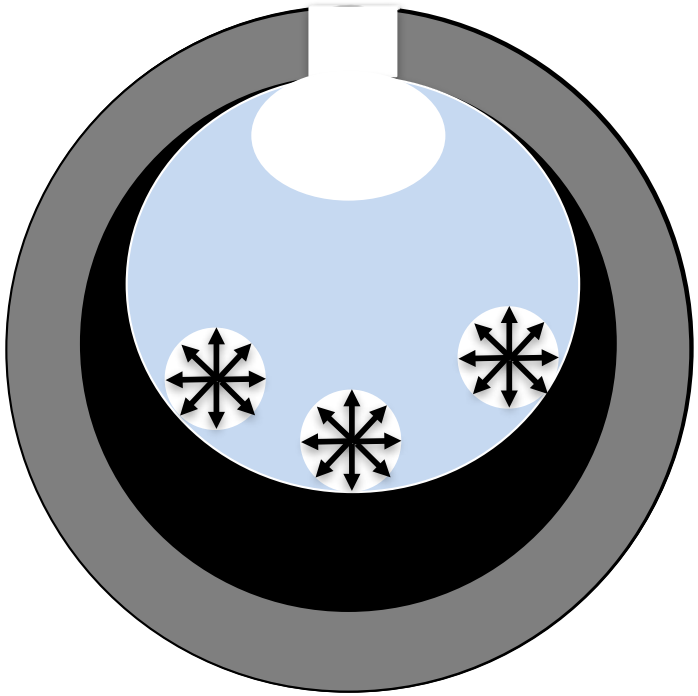

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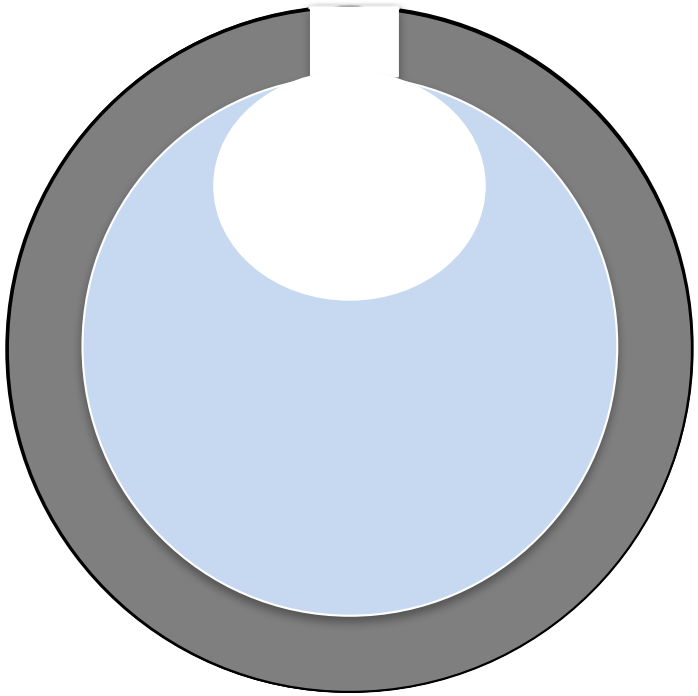
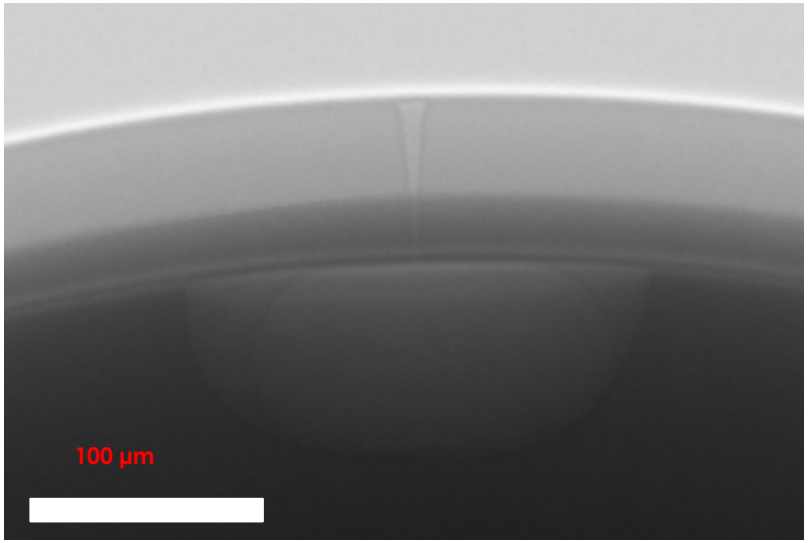
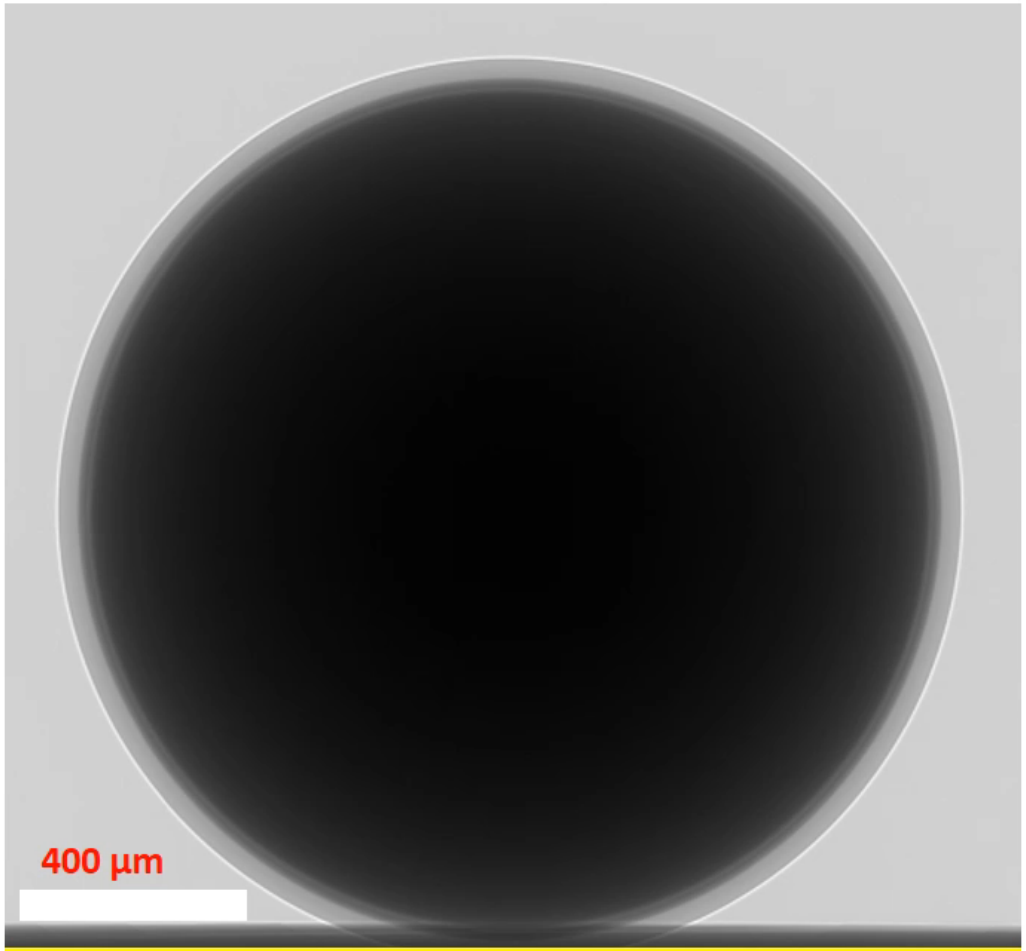


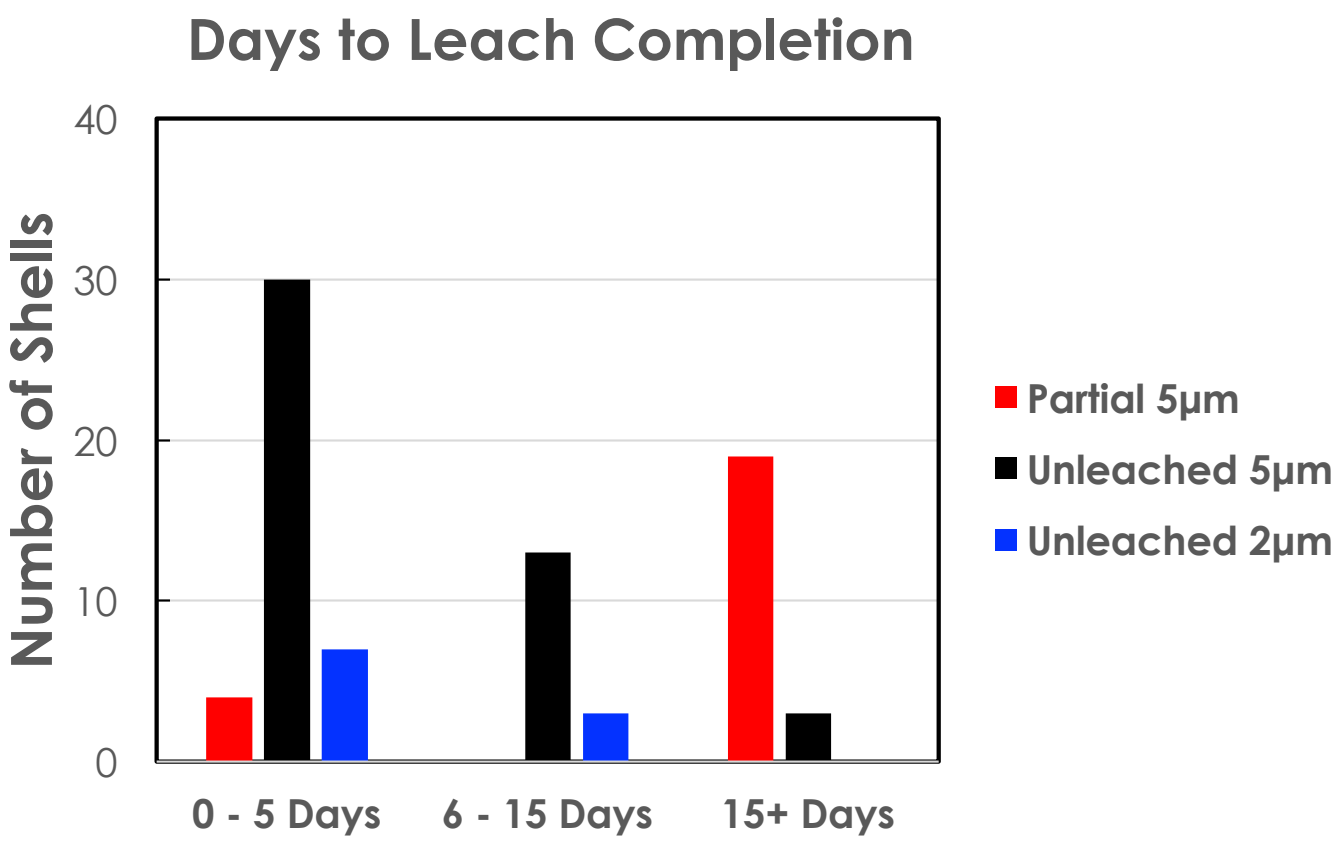
Initial rate is slow, but increases with silicon surface area exposure





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

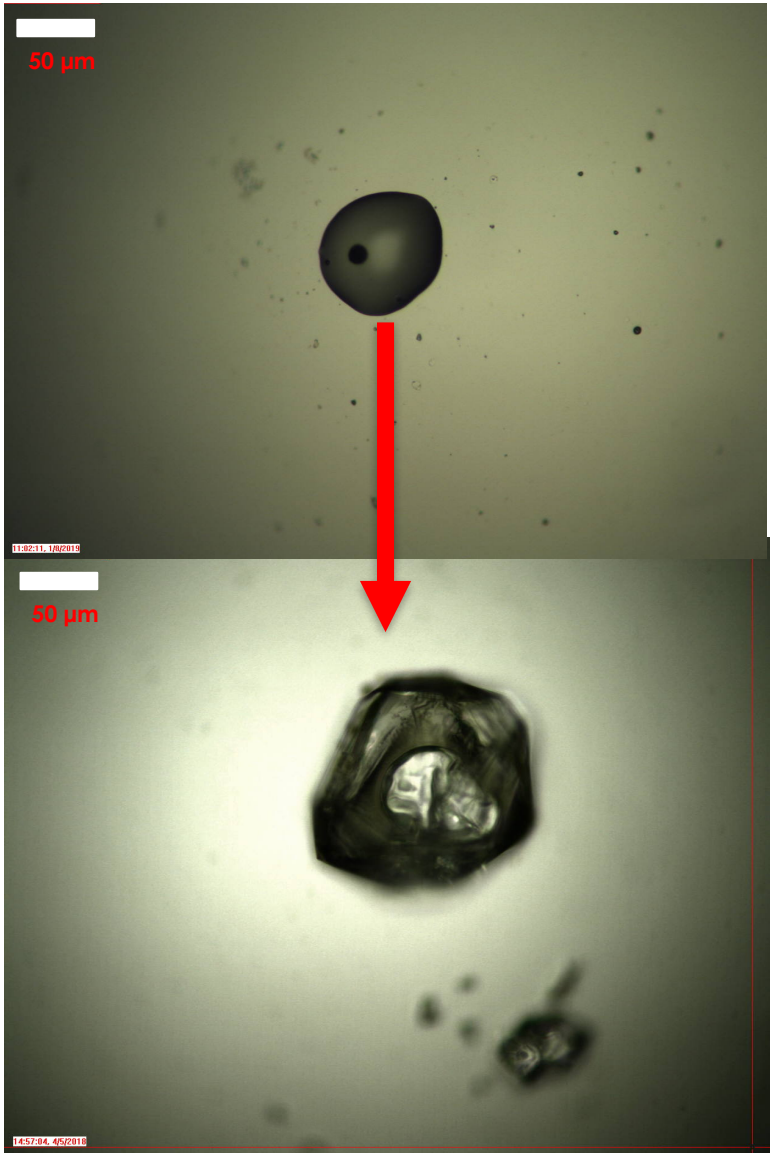


"Unleached" = Pressure cycler only

"Partial" = Started leaching in sonicator and transferred to pressure cycler

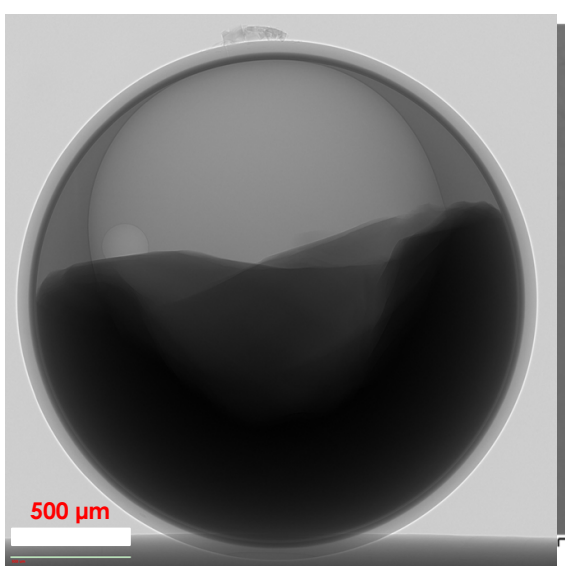
Solid residue formed when etchant near drill hole evaporates

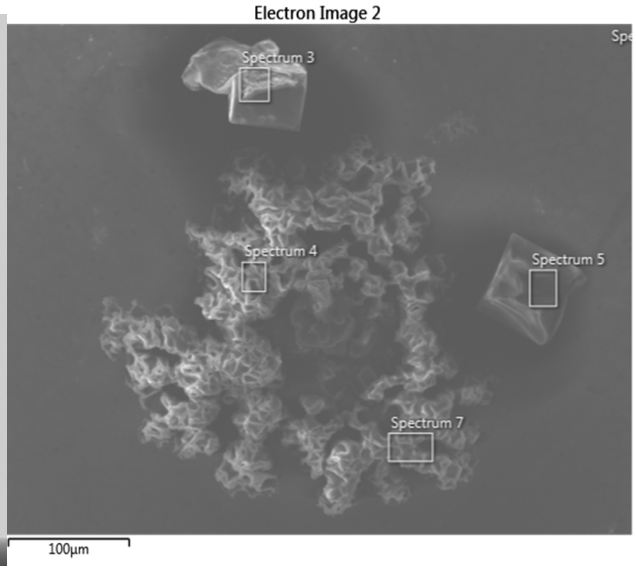
- **"Crystal" residue seen forming outside of drill hole**
  - Liquid leaks out of hole, evaporates, leaves behind precipitate
  - Only observed when taking shells out of leaching to check progress



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 N_2





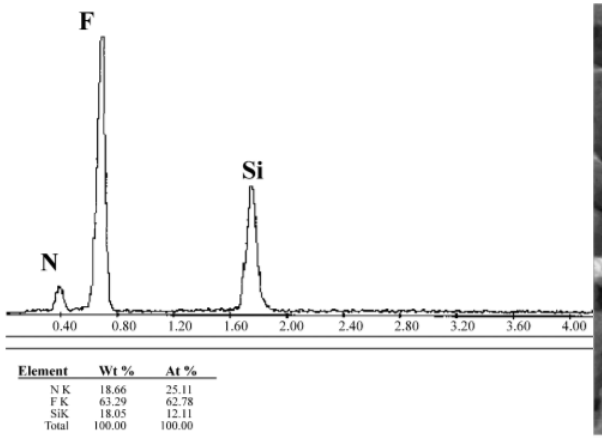
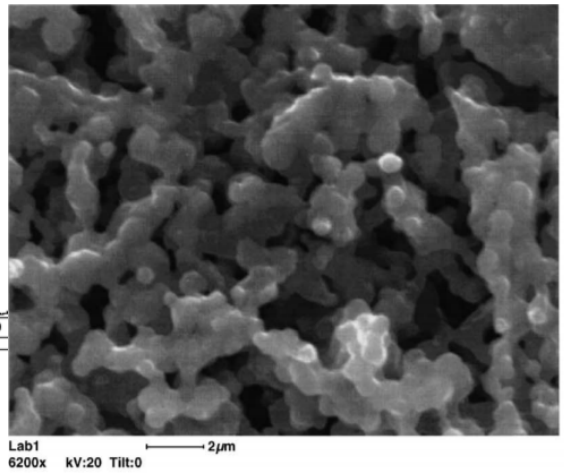
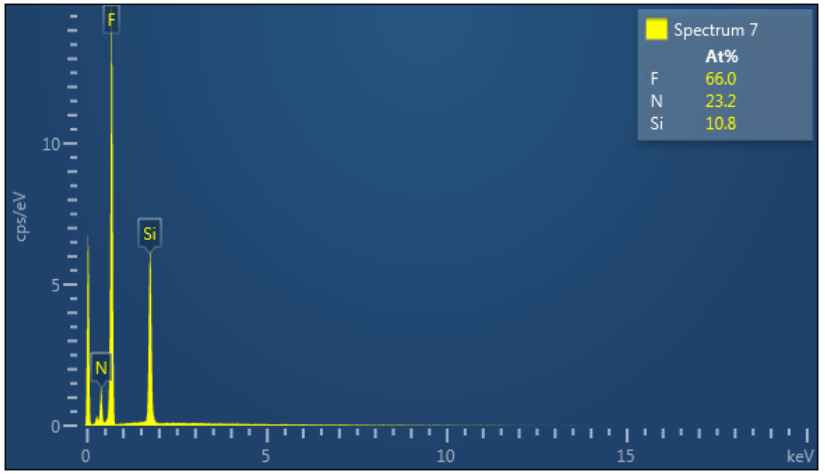


Fig. 3. SEM microanalyses spectrum of the white powder.





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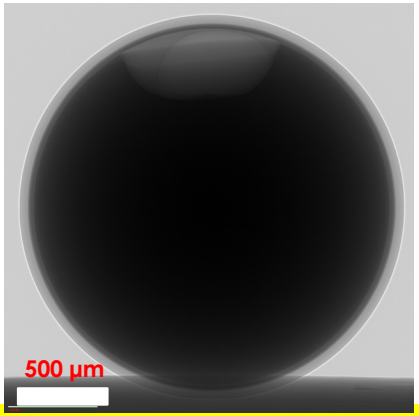
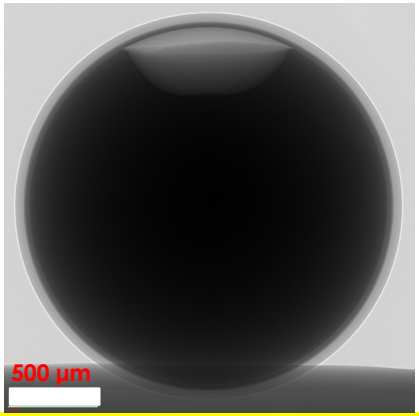
“Stuck” shells can be recovered by running through a 700°C oven bake



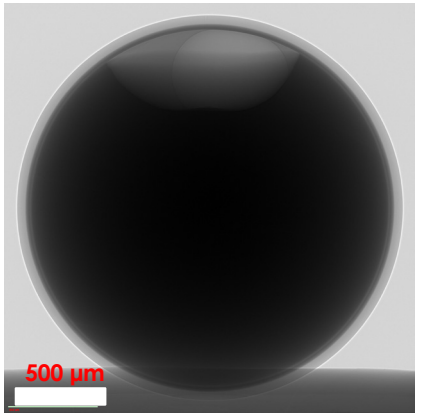
After 7 days (after overnight vacuum)

Oven Bake @ 700°C

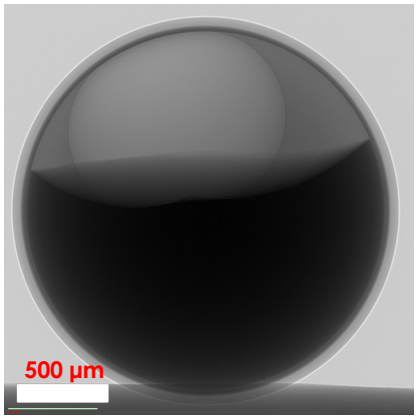


After 7 days

Into pressure cycler

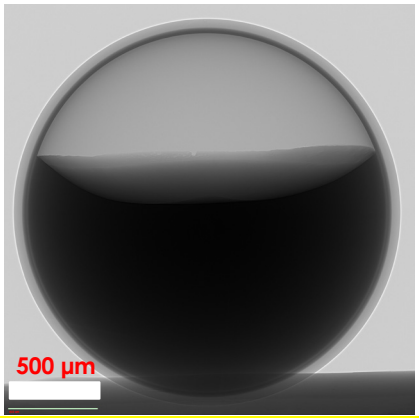


After 8 days



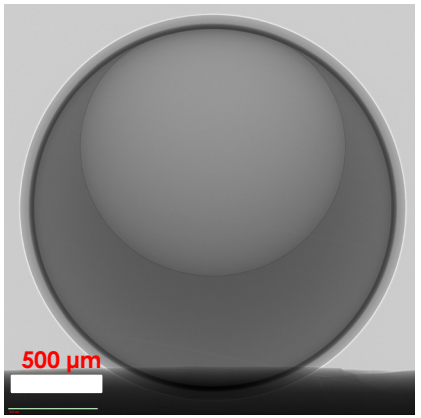
After 11 days

Oven Bake @ 700°C



After 11 days

Into pressure cycler



After 14 days

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