HDC Leaching Cleaning Experiments*

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ABSTRACT

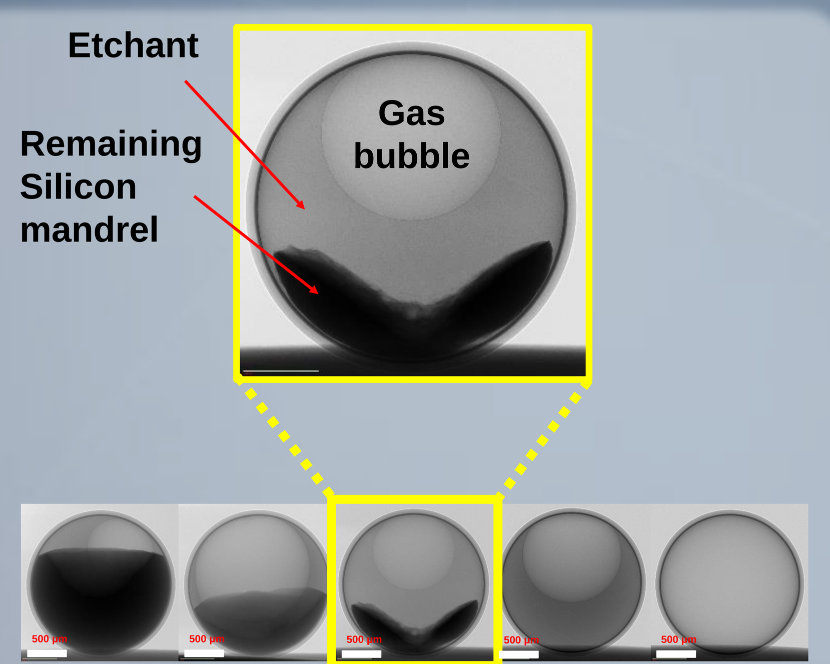
High density carbon (HDC) capsules are the primary ablator material used for inertial confinement fusion (ICF) experiments at the National Ignition Facility (NIF). Recently, HDC capsules have given the highest yields ever achieved at the NIF, including multiple demonstrations of fusion ignition in the laboratory. A key enabler of these experiments has been the development of the capsule fill tube assembly (CFTA) that utilizes a 2µm diameter fill tube. Leaching of the silicon mandrel through the 2µm drill hole is a critical process step in the production of these high yield targets. Improvements have been made to turn what was originally a developmental process into a production process.

Greater understanding of the leaching process has been accomplished through development of a new leaching chamber, reduction in contamination sources, and detailed examination of the effects of drill hole profiles on leaching success. Implementation of a revised low temperature cleaning process has resulted in a yield of effectively 100%. Future work is focused on better understanding of the bubble dynamics inside the capsule during the cleaning process to enhance efficiency. This poster highlights the experiments and improvements that have led to a >90% yield for the HDC leaching and cleaning process.

INTRODUCTION - HDC LEACHING/CLEANING OVERVIEW

HDC capsules are coated on a silicon template bead (mandrel). For use in ICF experiments, the capsule must first be laser drilled with a hole. The silicon mandrel is etched out through the hole using a HNO_3/HF acid mixture.

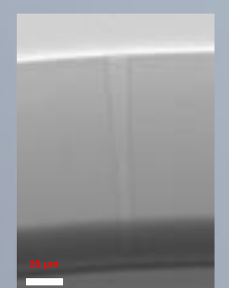
Radiographs (pictured on the right) are used to track the etching progress over time. Previous experiments have shown that etching can be completed within 3 days.



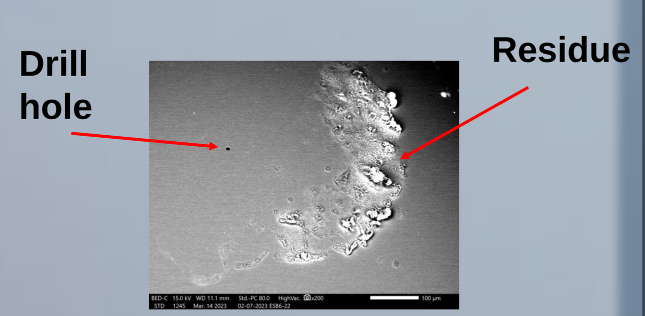
The etching of Si is given by the following equation:

$$3Si\,(s) + 4HNO_3\,(aq) + 18HF\,(aq) \rightarrow 3H_2SiF_6\,(aq) + 4NO\,(g) + 8H_2O\,(l)^{\dagger}$$

Gas generation from this reaction plays a crucial role in the pressure cycling process!



Radiograph of drill hole



Backscatter SEM micrograph of capsule surface after drying

Problem Statement

- Observation of inconsistent 2µm leaching success caused delivery and shot schedules to be put at risk
- Capsule yield loss from drill hole plugging or "stuck" leaching
- Residue stuck on outer surface composed of high-Z contaminants after drying process further reduced cleaning yield

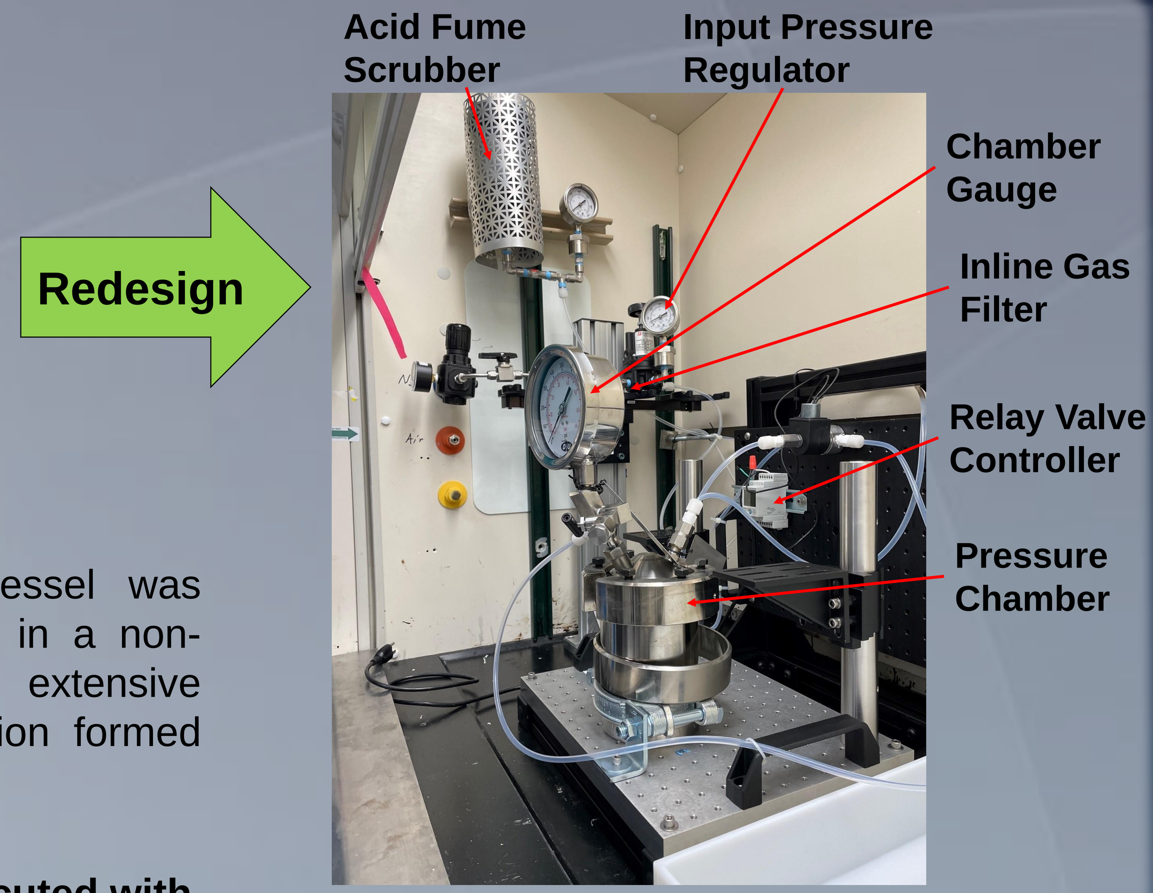
Key Objectives (prior to improvements)

- For 2µm fill tube capsules
  - 60-70% mandrel removal yield per batch average
  - No batches with <50% mandrel removal yield
- For 5µm fill tube capsules
  - 80-90% mandrel removal yield per batch average
  - No batches with <60% mandrel removal yield

[†]This only shows the balanced **net** equation for the etching process. There is a lot of literature that explores the details of this very complicated reaction that contains many intermediate products/steps

CHAMBER IMPROVEMENTS

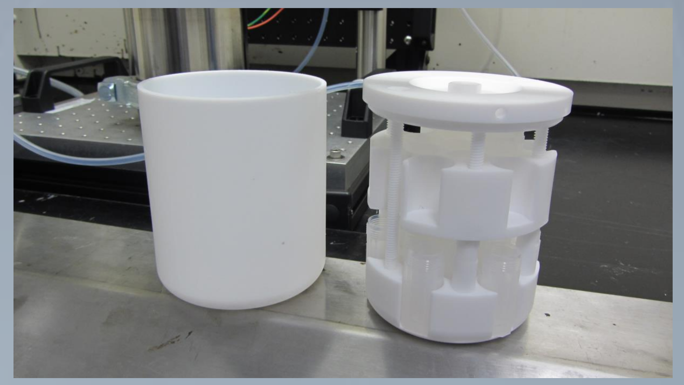
Interior of original pressure leaching vessel



Original pressure leaching vessel was operated inside a fume hood in a non-cleanroom environment. After extensive use, large amounts of corrosion formed within chamber

The system redesign was executed with a few guiding principles:

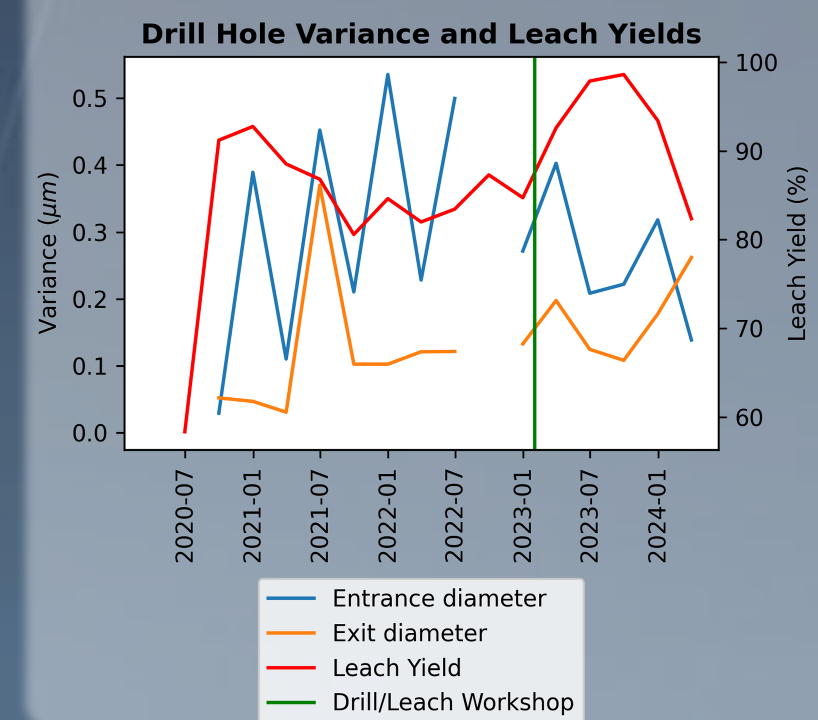
- Minimization of metal parts where possible
- Material selection of acid resistant metals
- Limit pathway for particulates to reach vials containing etchant/capsule
- Capability to reach higher pressures if necessary

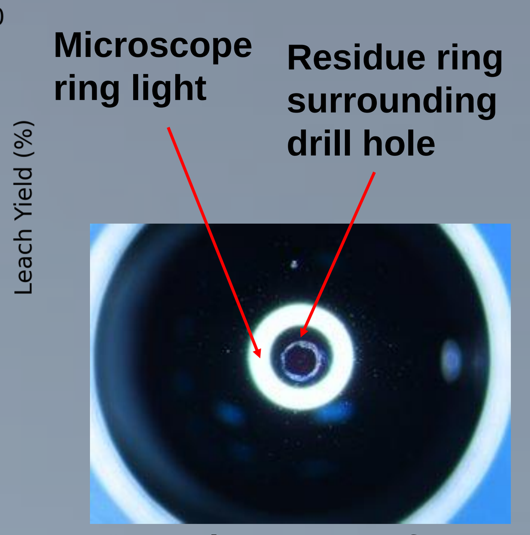


Re-designed vial holder

LASER DRILLING & PROCESS IMPROVEMENTS

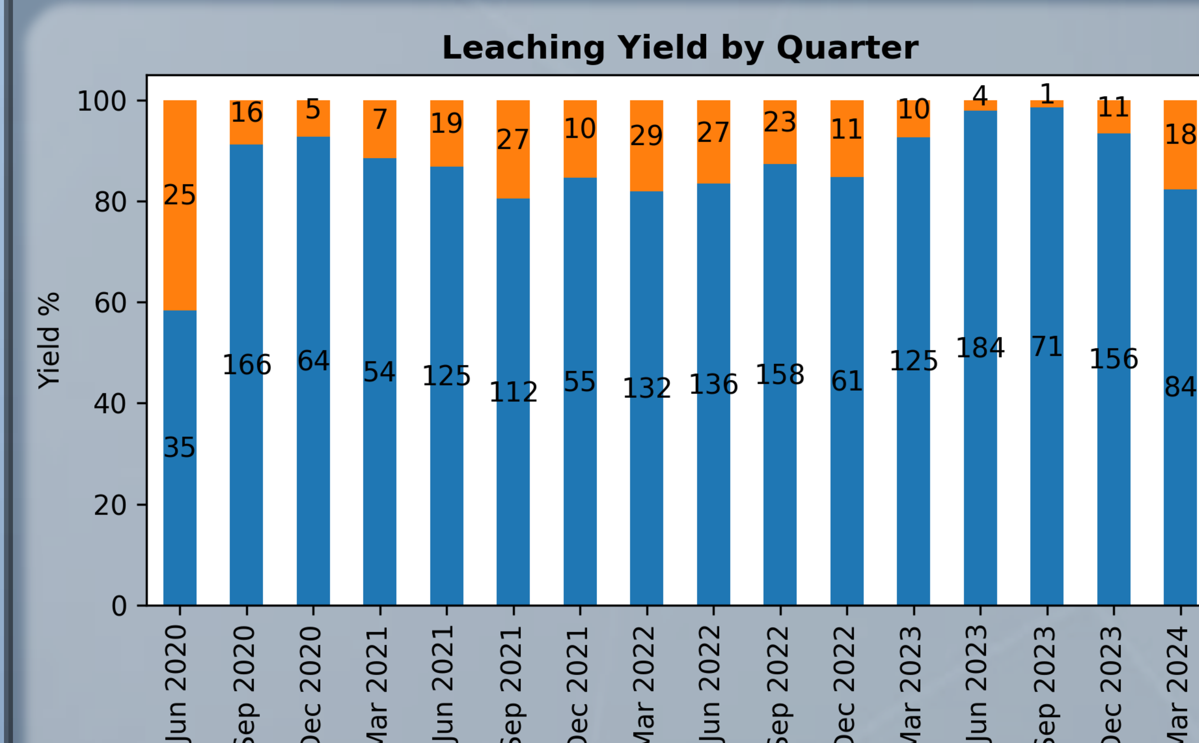
- Implementation of specification for 2µm fill tube holes
  - Tolerance ranges determined by resolution limit of Xradia microscope
  - See N. Alfonso's poster for additional laser drilling details
- Cleaning process control
  - Removal of 700C oven bake (legacy process!)
  - Observation of residues after cleaning step
    - EDS data shows that it is carbon
  - Improvements to cleaning process – 4 overnight cycle exchanges
- ICP analysis of used etchant
  - Led to process improvement in mandrel fabrication – investigated silicon source, now using higher quality material



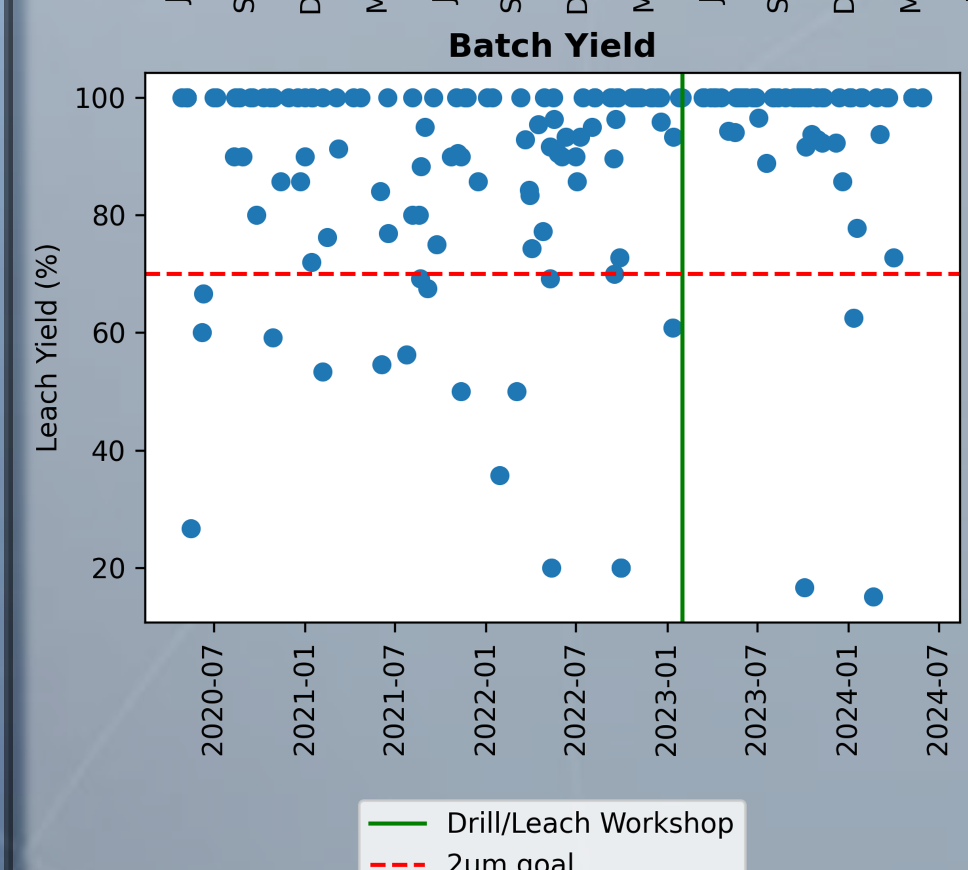


Capsule outer surface after new cleaning process

RESULTS



- Significant improvement in leaching yield rate after implementation of steps from drill/leach workshop in Feb 2023
- Slight drops in yields from early 2024 are the result of specific batch related issues that are currently not well understood



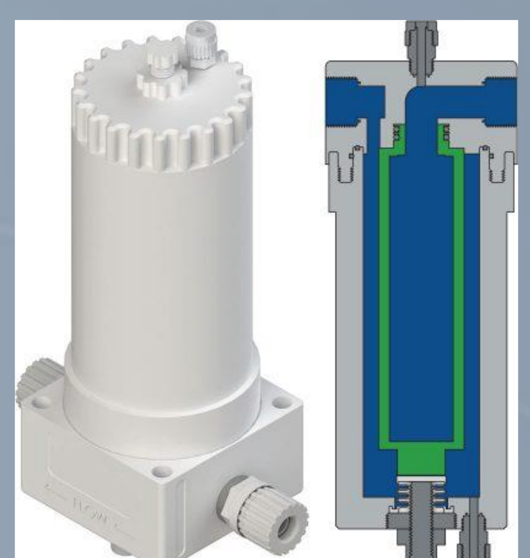
	First Pass Success %	Overall Success %	Total Capsules
Before Leach/Clean Workshop	74.8%	84.4%	1337
After Leach/Clean Workshop	92.1%	94.6%	624
All Data[‡]	80.3%	87.6%	1961

Variance in leaching process can be measured by the difference between "First Pass Success" and "Overall Success" (e.g. how often is our process fully leaching a shell on the first try?)

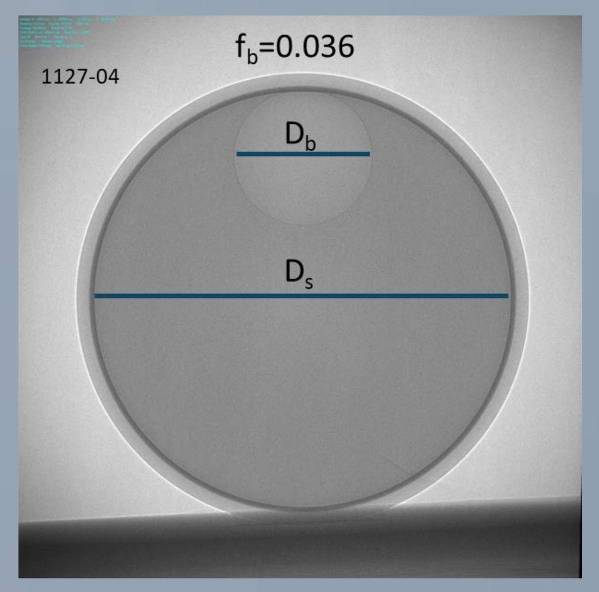
[‡]Data sourced from April 2020 to June 2024

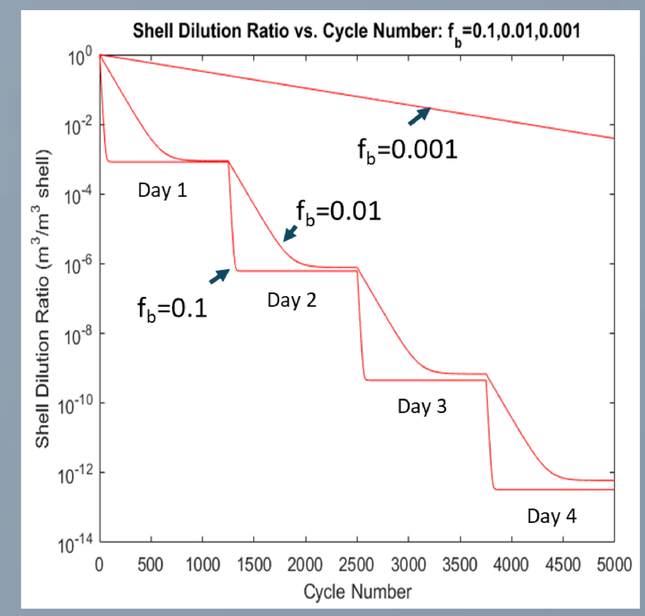
FUTURE WORK

- Explore design and development of completely metal-free leaching chamber
- Simulations suggest that larger bubble fraction will increase effectiveness of cleaning process
  - Bubble fraction can be defined as $f_b = \frac{D_b}{D_s}$ where D_b and D_s are the diameter of the bubble and capsule, respectively
  - Bubble fraction can be manipulated through lowered drying time
- Can we extend our learnings here to 1µm capsules?



Next-gen metal-free leaching chamber





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