Atomic Layer Deposition

Team Members
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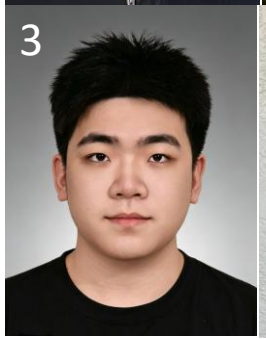
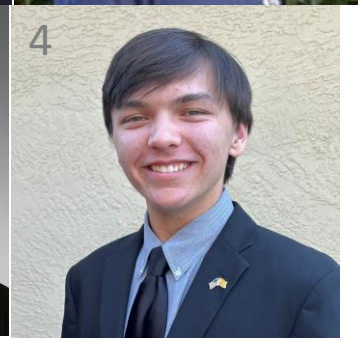
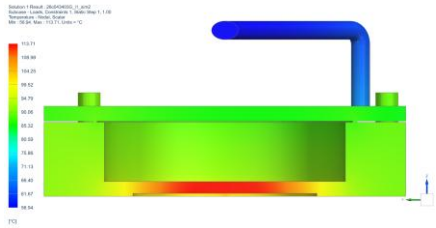
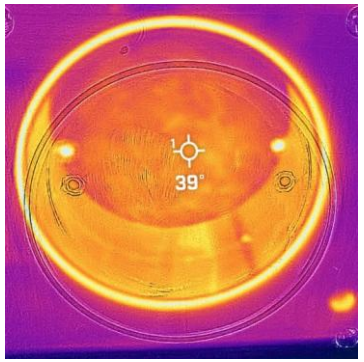
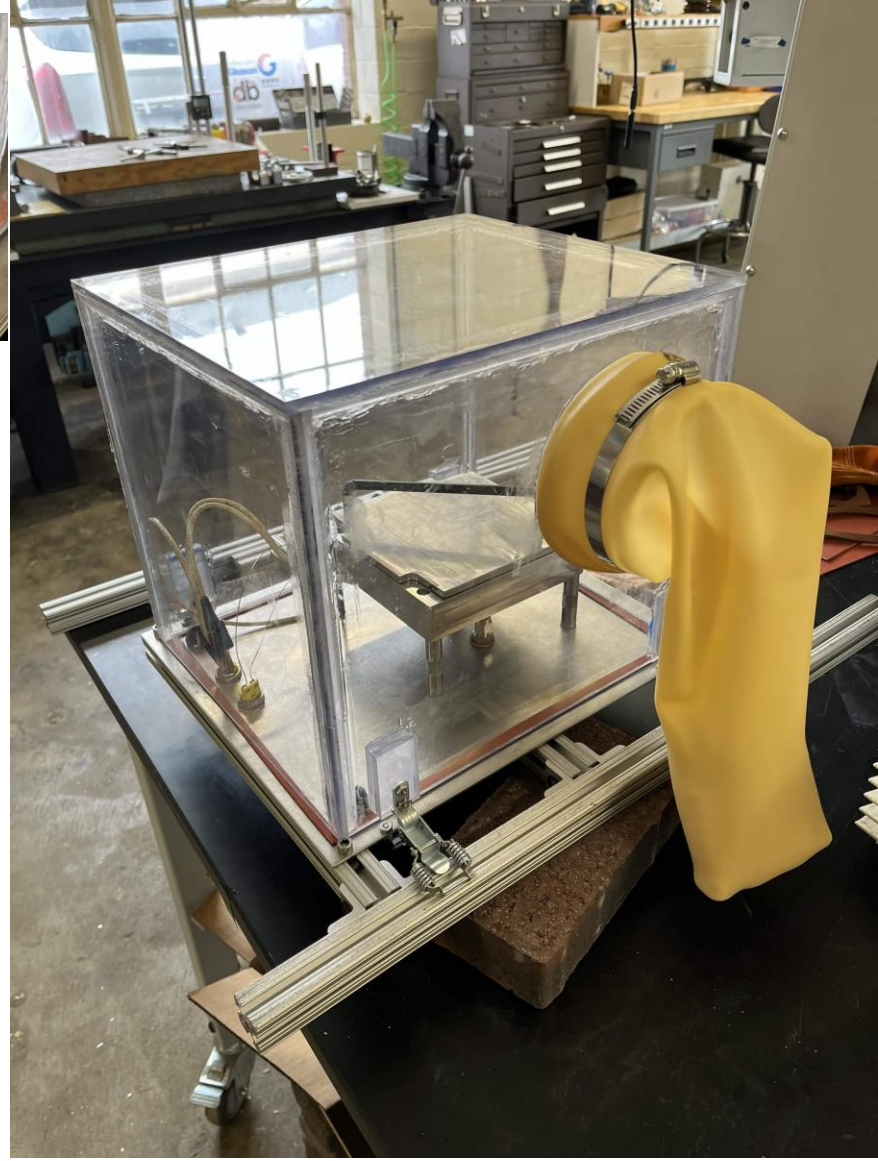










Customer
Dr. Zachary Robinson
Laboratory for Laser Energetics (LLE)

Project Overview

The team's objective is to create a reactor chamber and glovebox assembly that can be integrated into the existing Atomic Layer Deposition (ALD) system to reduce the duration of growth-rate curve experiments. The updated ALD system will allow the LLE to perform fusion energy related research more efficiently.

Problem Statement

The LLE is looking to decrease ALD experiment runtime thereby accelerating LLE research into clean, abundant fusion energy. Experiments with the current rig are inefficient because the system must cool down prior to removing samples.

A custom glovebox and reactor chamber that enable sample retrieval at working temperature would allow for single-day growth-rate experiments rather than multi-week.

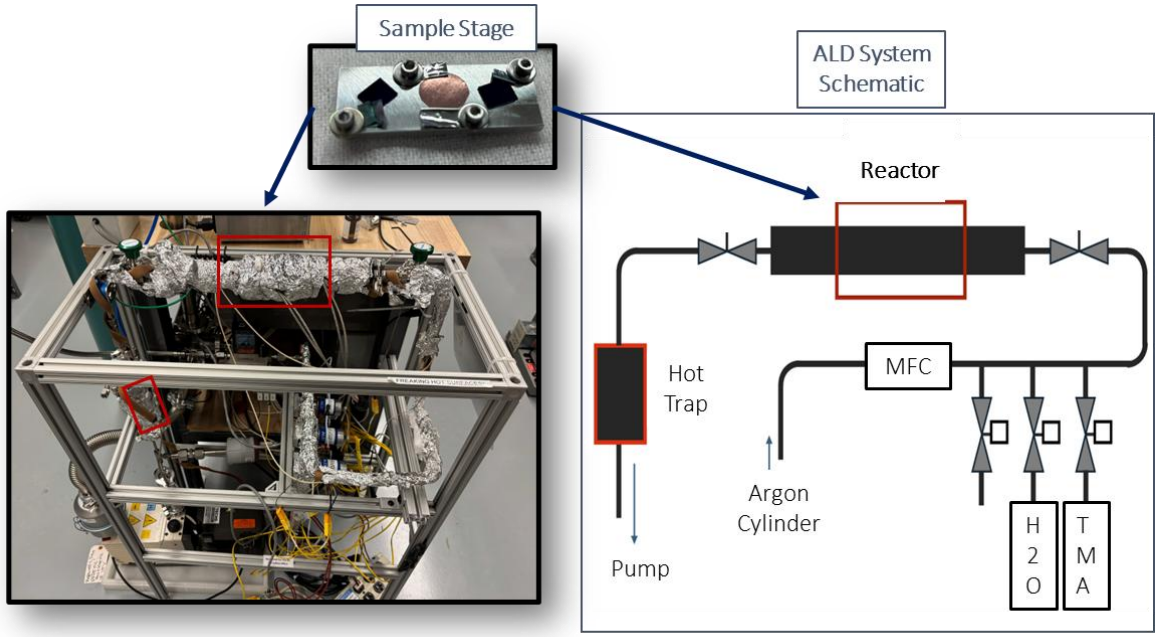
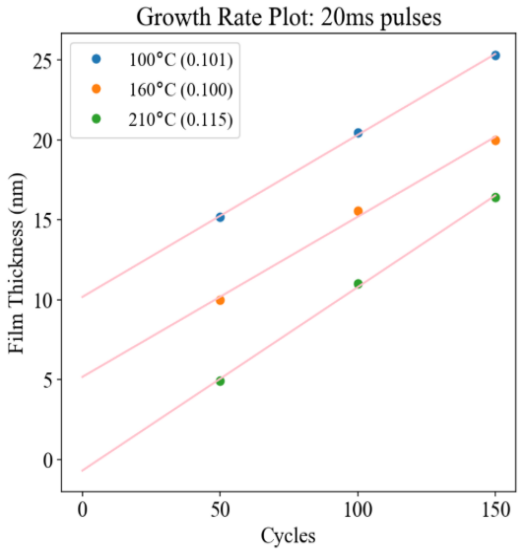


Image of existing ALD reactor system at LLE



ALD is characterized by linear growth rate.
A plot like this currently takes many days of ALD.

Example of laboratory glovebox system to prevent sample contamination

Deliverables, Requirements and Specifications

The team worked with Dr. Robinson to develop a number of design requirements and testable specifications for the project to meet.

Deliverables:

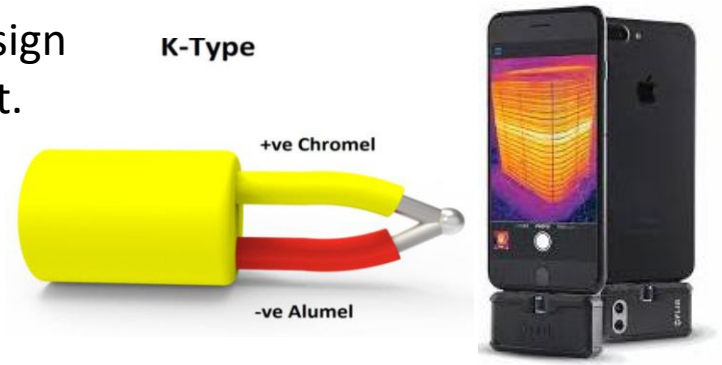
- Reactor chamber
- Glove box
- Integration with existing LLE setup

Requirements:

- Assembly must allow for of mid-run sample removal.
- Reactor temperature must be uniform across sample area
- All components must be designed with consideration of chemical, thermal, and pressure conditions.
- All components must be compatible with existing ALD system.
- The system must be approved for safety by Dr. Robinson.

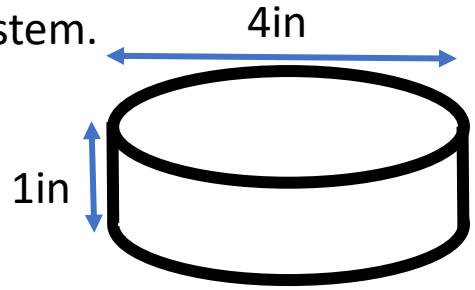
Specifications:

- Maximum dewpoint of -40°C inside N_2 glovebox
- Reactor chamber must pass Helium leak check
  - (Less than $1*10^{-9}$ torr*L/s vacuum leak rate)
- Reactor chamber must achieve at least 250°C
- Chamber dimensions 4” diameter, 1” height

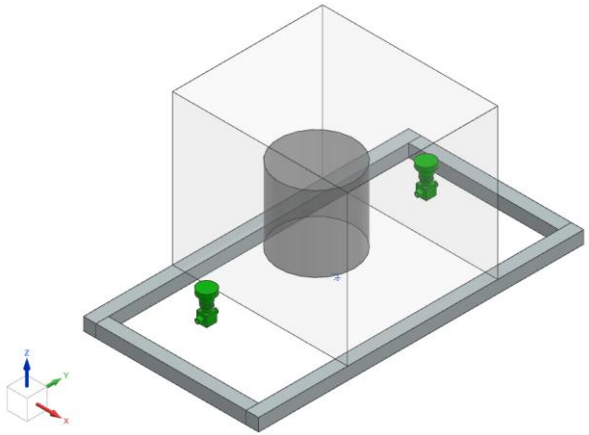


K-type thermocouple and thermal camera for measuring reactor temperature

Edwards Spectron 600D leak rate tester can verify vacuum sealing



Reaction chamber minimum size specifications

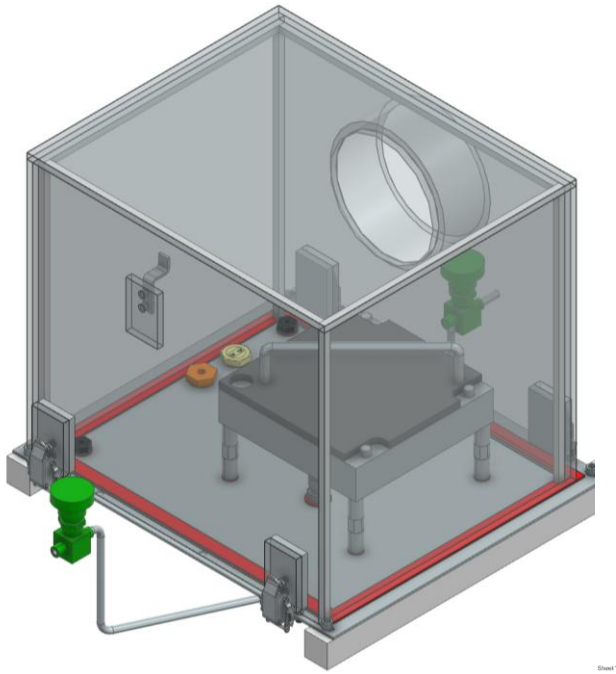


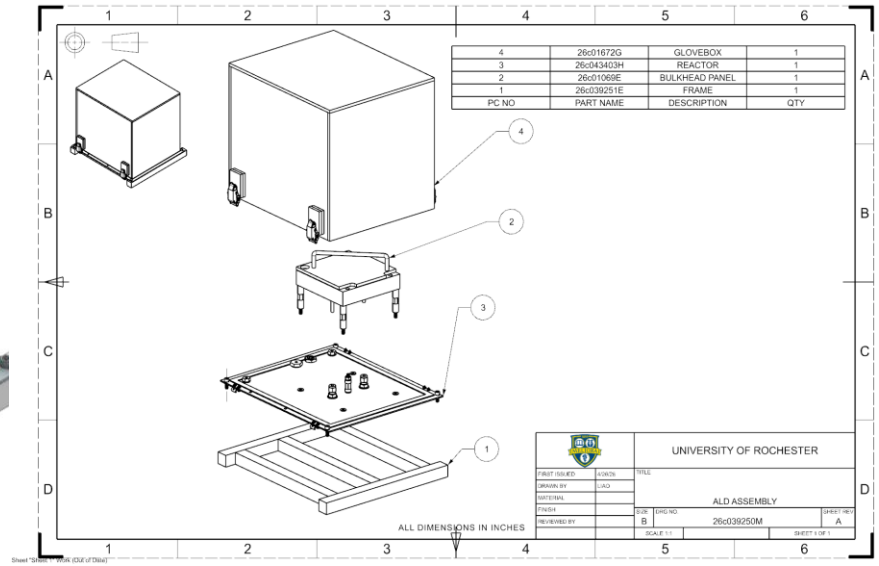
Space envelope created from existing ALD system

Current Project Status

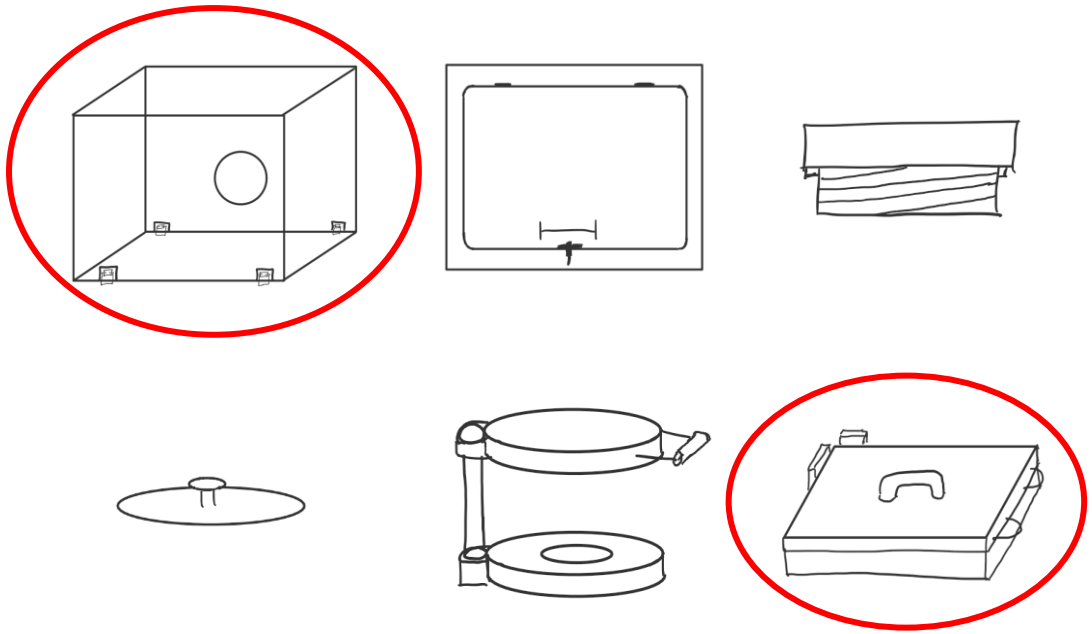
The team went through a number of design iterations before settling upon a final configuration for both the glove box and reactor.

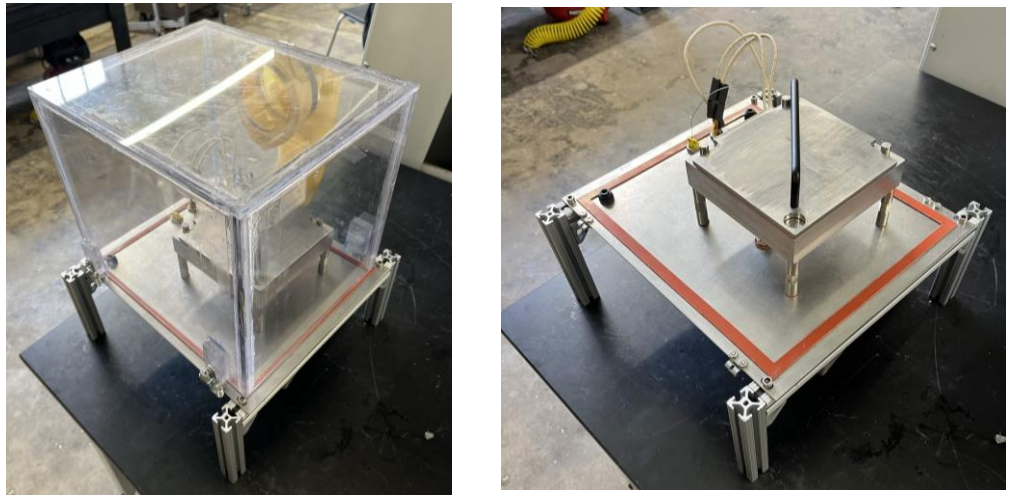
Drawings were made for each part of the system, then components were bought or manufactured.





Final design image and assembly drawing





The team fabricated designs into a final assembly

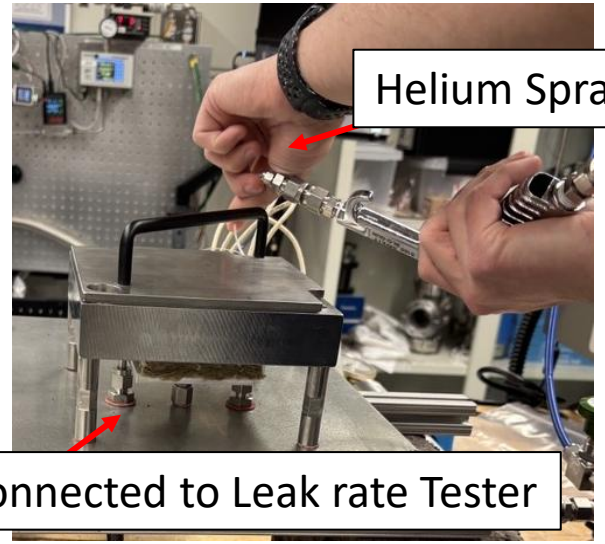
Current Project Status

Leak Rate Maximum	Leak Rate Measured	Test Result
1E-9 torr L/sec	4.7E-10 torr L/sec	PASS

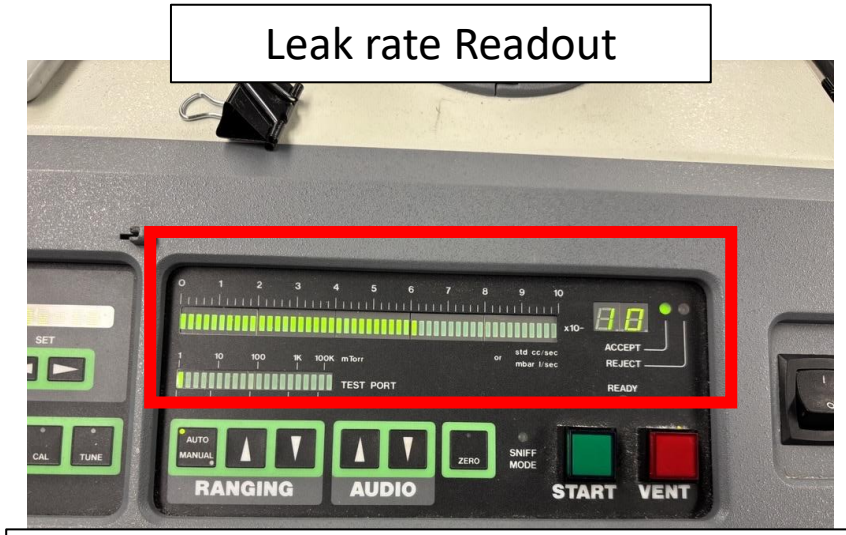
Vacuum Testing:
The system vacuum was tested using an Edwards Spectron 600D leak rate tester.

The test was successful in demonstrating the system has a leak rate below the maximum required.

Preliminary test shows vacuum generates enough force to list reactor



Leak testing reactor at LLE



Measured Leak Rate: 6.2E-10 std cc/sec (4.7E-10 torr L/sec)

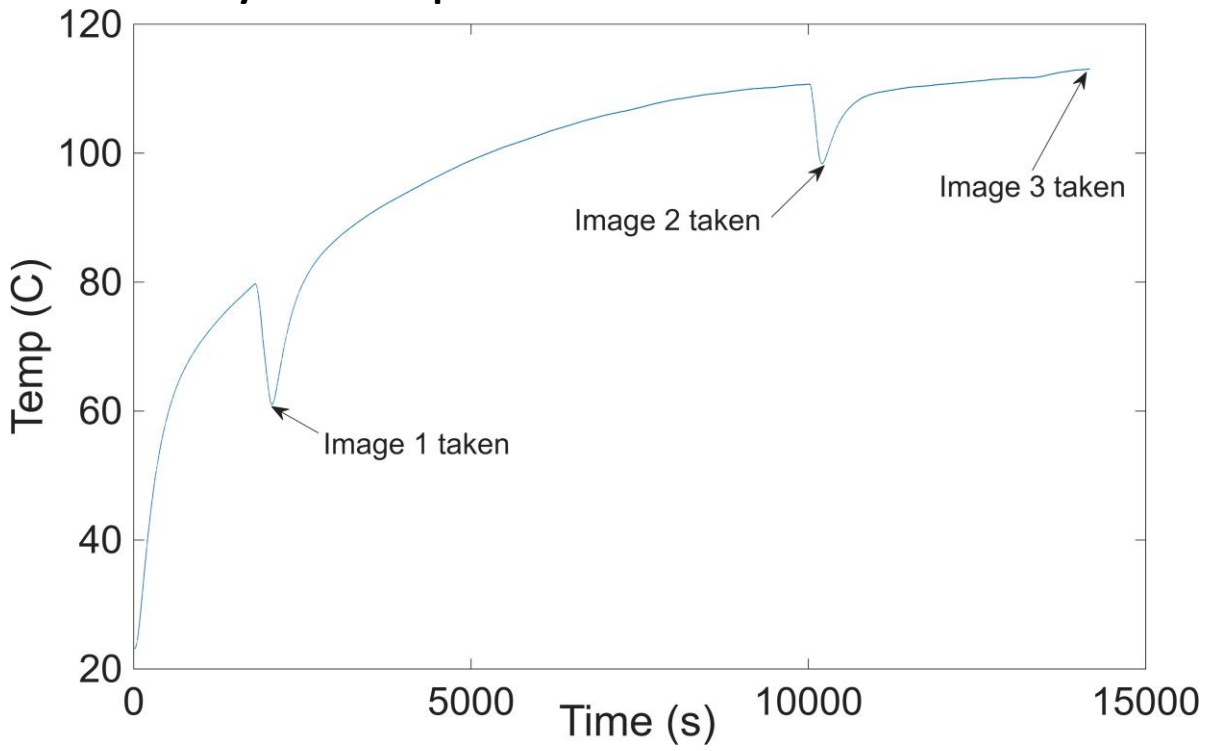
The leak tester measures the amount of helium sucked in through leak points in vacuum system

Current Project Status

Thermal Testing:

Because a high temperature Viton O-ring could not be sourced during testing, the heater was run at ~11% of full power. The following data was taken to improve thermal simulations and make predictions about final heating capacity.

Steady state temperature of bottom of reactor over time



Thermocouple placed at the bottom of the reactor measured exterior temperature

Min Temp Requirement	Max Temp Measured	Test Result
250°C	Exterior 112°C, Interior 39°C	**Not tested to full capacity**

Thermal Camera Images of Reactor Interior

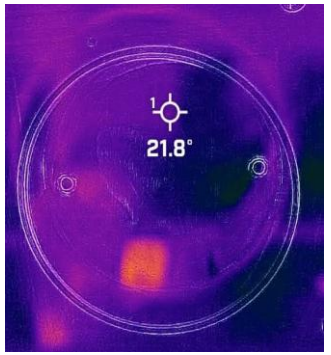


Image 0: Before Heating.

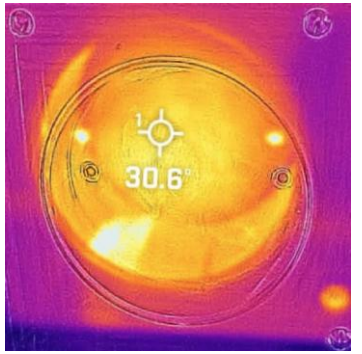


Image 1: At 2041s.

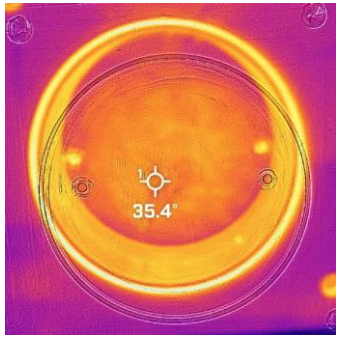


Image 2: At 10193s

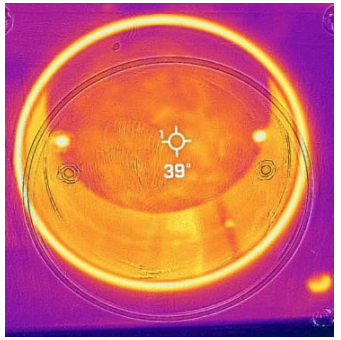


Image 3: After heating stopped, 14133s

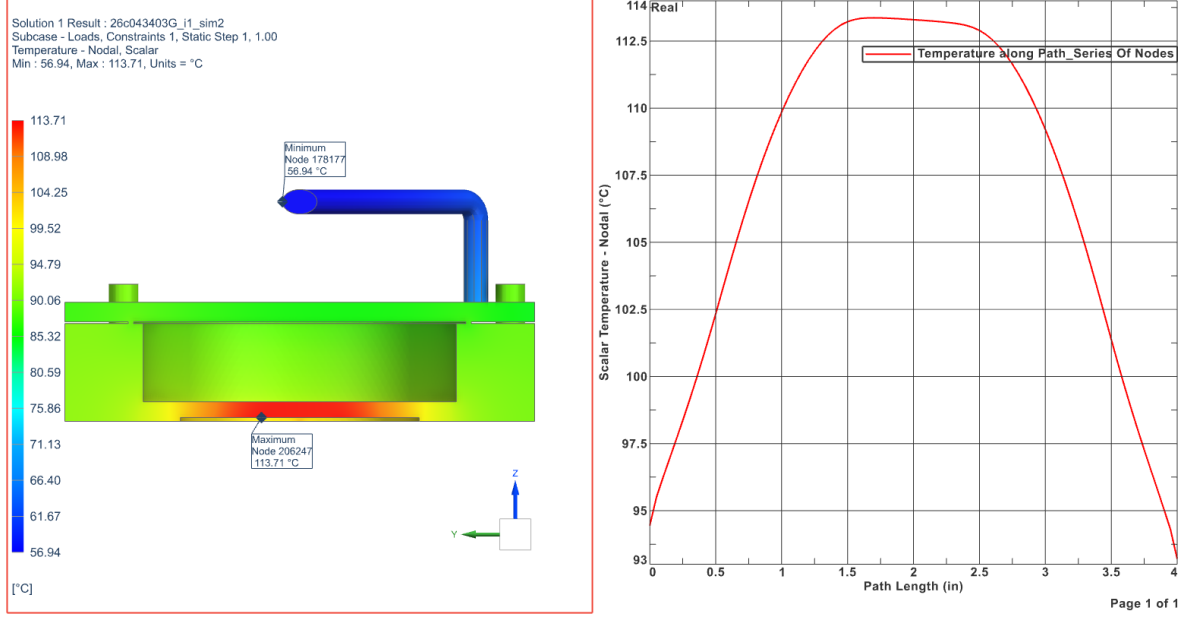
Chamber interior did not reach the same temperatures as exterior thermocouple during this test

Current Project Status

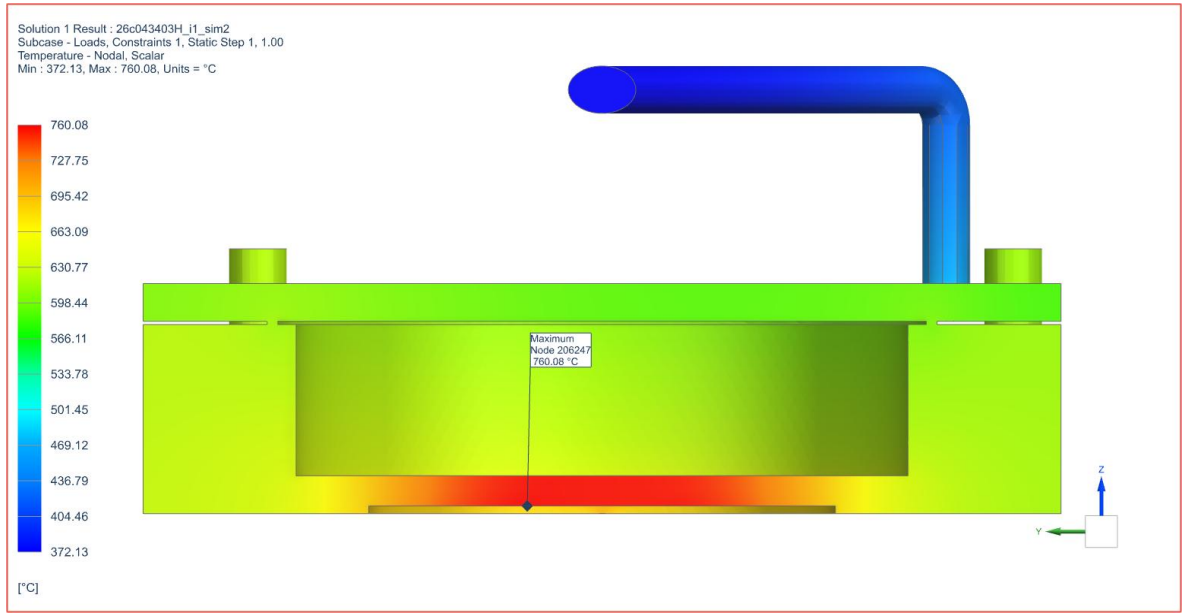
Heat Transfer Simulation:
The team performed rough heat transfer simulation to fit to experimental trial. Preliminary results suggest target temperature of 250°C is well within reach and thermal uniformity within the chamber is within specification.

Further thermal testing with a direct contact thermocouple inside the chamber and a measurement of the active resistance of the heater would provide more accurate data to validate these simulation results.

Transient simulations would allow the team to compare experimental heat up data and do additional tuning of the simulation parameters for accuracy.



Revised heat transfer simulation shows reactor temperature uniformity within 20°C



Full heater power - 760°C max temperature

Conclusions/Future Work

This project was successful at meeting some of the requirements and specifications. The design of the system is sound according to simulations and preliminary testing of requirements. The system requires some minor improvements to meet all specifications and be ready for a full ALD growth.

Future Work:

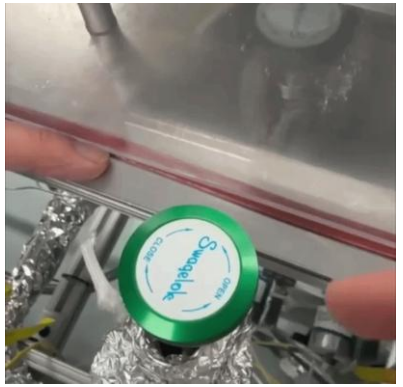
- Acquire a high temperature O-ring to test full heater power
- Fix small leak in glovebox seal
- Add insulation to protect glovebox interior from hot components

Acknowledgements:

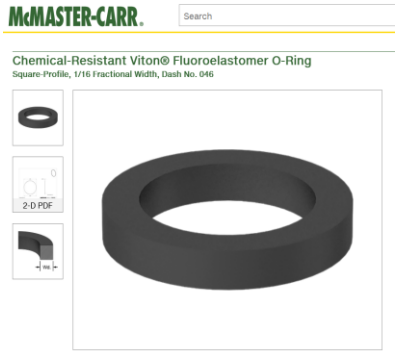
- Authors acknowledge the help of Dr. Zachary Robinson, Professor Christopher Muir, Christine Pratt, Samantha Kriegsman, Jim Alkins, Bill Mildenberger, Jeff Leffler, Paul Osborne, and Rashad Ahmadov for their aid in the completion of this project.

Specification	Value	Measurement	Result
Glovebox max dewpoint	-40°C	Leak prevents testing	**FAIL**
Chamber max leak rate	1E-9 torr L/sec	4.7E-10 torr L/sec	**PASS**
Chamber min achievable temperature	250°C	Tested to 111°C with low temperature O-ring	Not tested to full capacity
ALD chamber min internal diameter (circle)	4in	4in	**PASS**
ALD chamber min internal height	1in	1in	**PASS**

Final summary of test results and specifications



Glovebox gasket does not fully seal



Viton high temperature O-ring