INL/CON-19-54498-Revision-0

Additive Manufacturing of Heat Pipes for Microreactor Applications

Donna Post Guillen, Clayton G Turner, Adrian R Wagner, Patrick J Moo

February 2020

Idaho National Laboratory

The INL is a U.S. Department of Energy National Laboratory
operated by Battelle Energy Alliance

INL/CON-19-54498-Revision-0

Additive Manufacturing of Heat Pipes for Microreactor Applications

Donna Post Guillen, Clayton G Turner, Adrian R Wagner, Patrick J Moo

February 2020

Idaho National Laboratory
Idaho Falls, Idaho 83415

http://www.inl.gov

Prepared for the
U.S. Department of Energy

Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

Idaho National Laboratory

Additive Manufacturing of Heat Pipes

Donna P. Guillen, Adrian Wagner, Patrick Moo
Idaho National Laboratory

Clayton Turner
University of Idaho

TMS Annual Meeting, San Diego, CA
February 23-27, 2020

~~Additive Manufacturing of Heat Pipes~~ Everything is easy until you go to do it

Donna P. Guillen, Adrian Wagner, Patrick Moo
Idaho National Laboratory

Clayton Turner
University of Idaho

and many others…

TMS Annual Meeting, San Diego, CA
February 23-27, 2020

www.inl.gov

Idaho National Laboratory

Outline

- Introduction
- Objectives and Scope
- Description of Printer
- Process Parameters
- Post-Processing
- Sample Characterization
- Summary
- Acknowledgements

Motivation for this Research

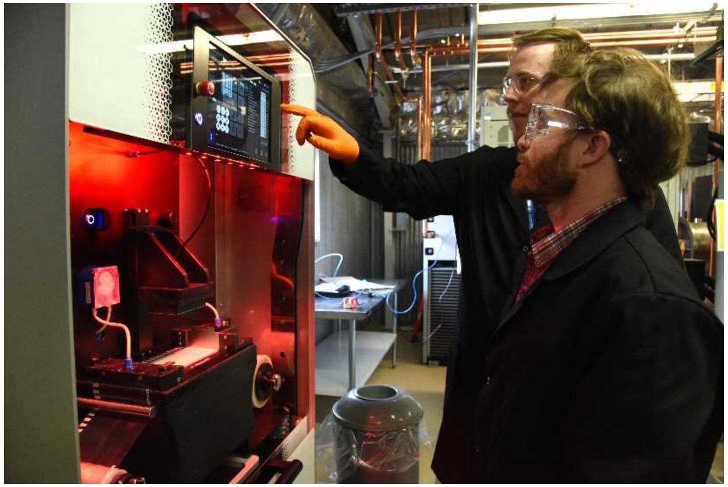
- **Problem addressed by this research**: Develop process for additively manufacturing heat pipes for advanced heat removal and transport for thermal energy conversion materials, devices, and systems
- **Significance**: The research performed by this project has the potential to impact numerous applications involving thermal transport, including nuclear reactors, electronics cooling, waste heat recovery, etc.
- **Objectives**:
    - Design and develop advanced manufacturing techniques for heat pipes with improved performance and lower cost
    - Incorporate heat pipes within a heat exchanger to minimize thermal resistances
    - Demonstrate the use of CoRE (configurable, removable, extractable) sensors and instrumentation to obtain real-time in situ/in operando data

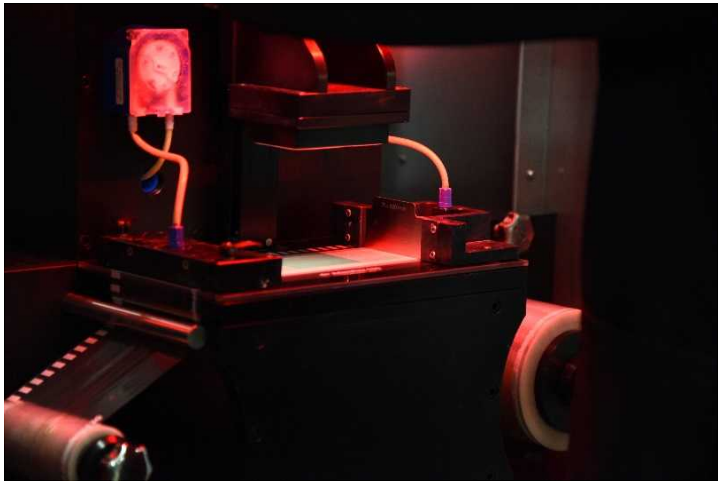
Benefits

- Advantages of 3D printing
  - Combine solid and porous regions within one part
  - Fabricate a freeform structure with complex geometry
  - Optimize internal pore structure
  - Integrate without introducing interfaces
- Key performance parameters affecting the heat transfer performance and capillary limit of a heat pipe wick are the:
  - porosity
  - effective pore radius
  - permeability
  - capillary pumping performance
  - wettability

Advanced Manufacturing

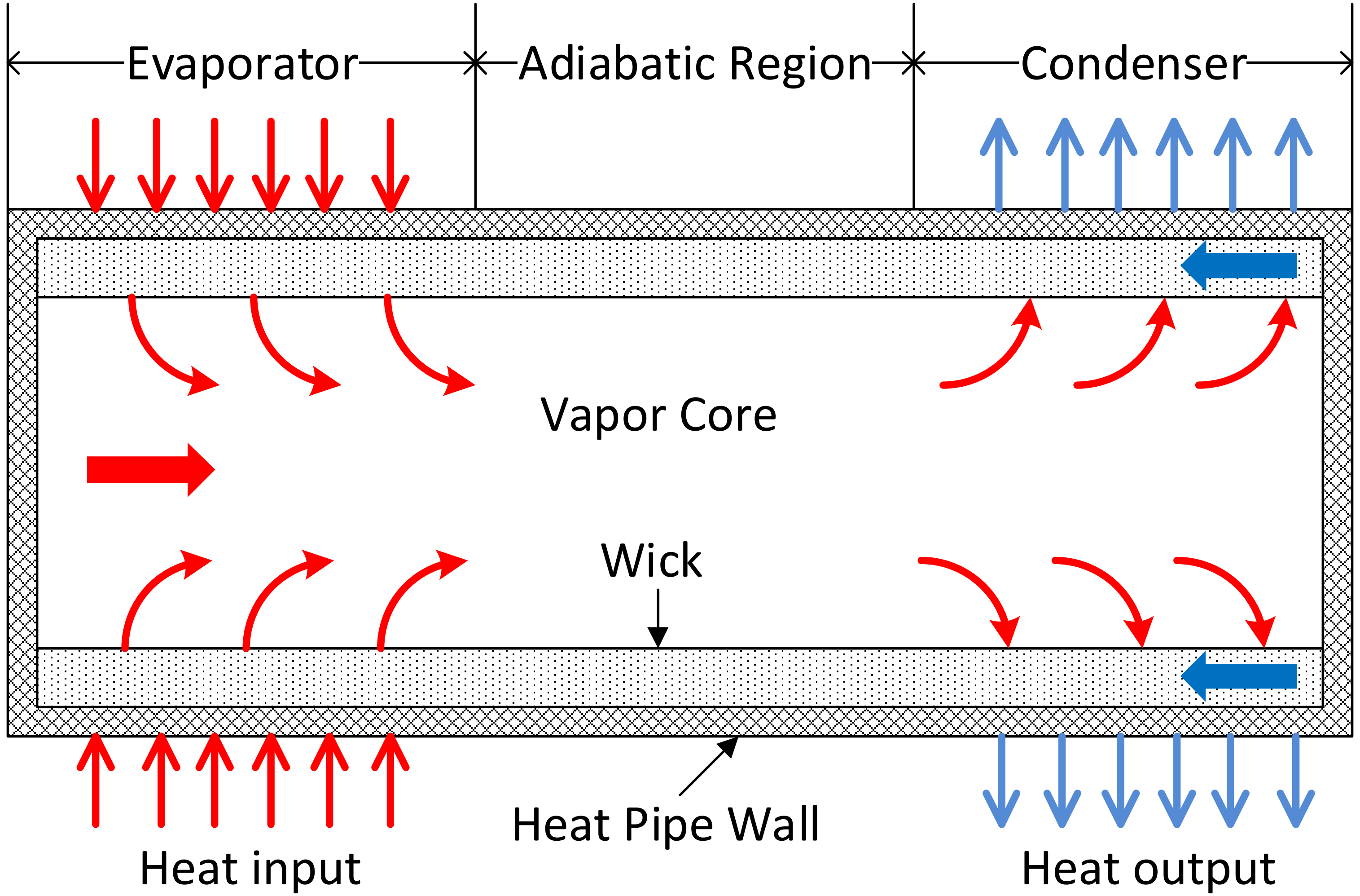
- The layer-by-layer deposition of material in the AM process creates complex processing conditions and can produce defect-laden microstructures that affect mechanical properties and performance
- Different print and post-processing parameters can result in materials with significantly different microstructures, defect populations and distributions, and internal stresses
- Materials synthesis and characterization/measurement tools will be used to collect data on additively produced materials uniquely tailored for heat pipe-heat exchangers (HPHX)





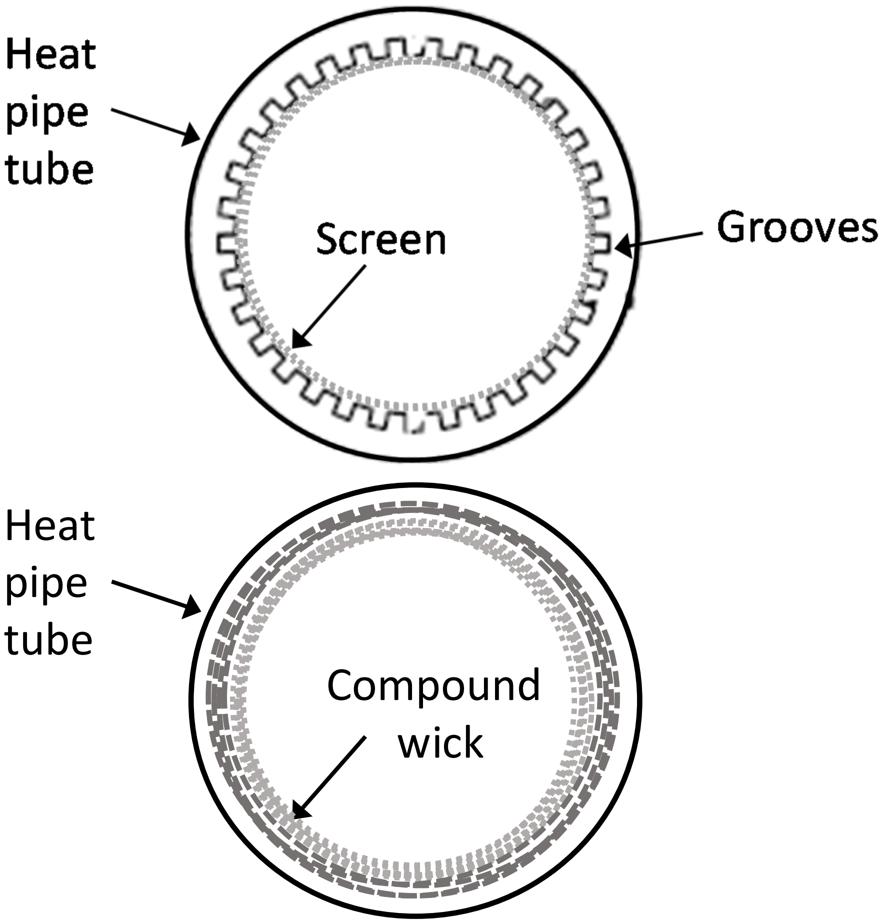
Basic Operation of Heat Pipes

- Heat pipes are highly efficient devices used to transport heat from one point to another
  - No moving parts, long lifetimes, little to no maintenance required



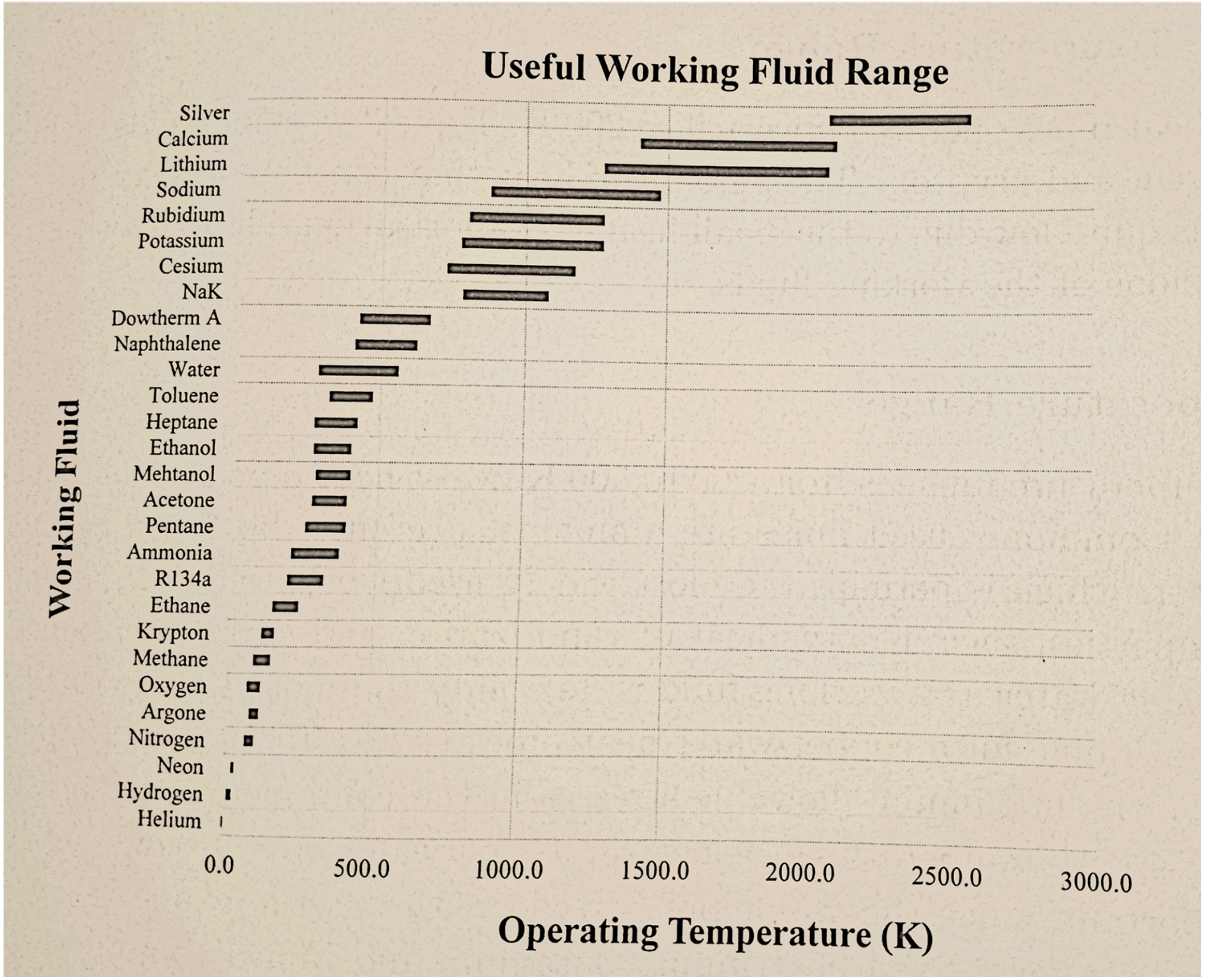
Heat Pipe Architecture

- Design and operating variables include:
  - Orientation, heat pipe size, heat pipe wall material, wick or wickless (thermosyphon), wick material, heat pipe working fluid
  - Wick structure − wick surface porosity, wick thermal conductivity, wick wire size, effective pore radius of the liquid-vapor interface, wick thickness, number of grooves, and groove sizes



Heat Pipe Working Fluid Range

Ref.: Faghri, A. Heat Pipe Science and Technology. 2nd ed., Global Digital Print, 2016.



Recommended Heat Pipe Wall and Wick Materials

Working Fluid	Material	Working Fluid	Material
Ammonia	Aluminum	Potassium	Nickel
	Carbon steel		SST
	Nickel		Inconel
	SST		Titanium
Acetone	Copper		Refractory metals
Methanol	Copper	Lithium	Nb-1%Re
	SST		Tungsten
Water	Copper		W-26%Re
	Monel		SGS Tantalum
Sodium	SST	Mercury	SST
	Nickel	Silver	W-26%Re
	Inconel 800		
	Hastelloy X		
	Haynes 188		
	Molybdenum		
	Tungsten		

Ref.: Faghri, A. Heat Pipe Science and Technology. 2nd ed., Global Digital Print, 2016.

HTPIPE, Heat Pipe Simulation Tool

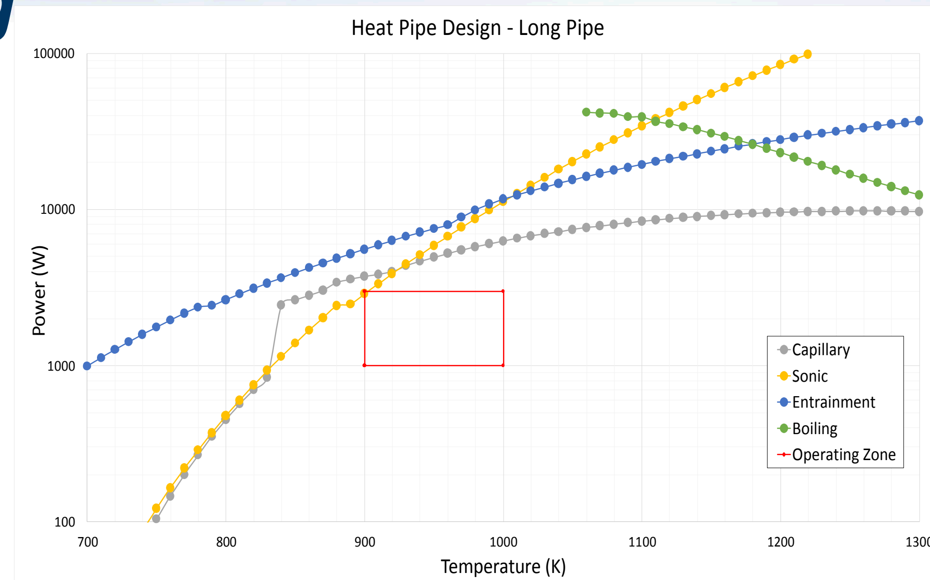
- Computer program developed by LANL that has been extensively validated with experiments
- Calculates a steady-state approximation of four limits within a heat pipe:
  - **Capillary limit** − reached when the wick structure cannot overcome gravitational, liquid, and vapor flow pressure drops
  - **Sonic limit** − occurs when the vapor flow reaches sonic velocity as it leaves the evaporator and the flow becomes choked
  - **Entrainment limit** − occurs when high velocity vapor flow strips liquid from the wick
  - **Boiling limit** − occurs when the working fluid boils in the wick, preventing liquid return to the evaporator
- These limits indicate the maximum heat flow into the heat pipe at a given temperature before the performance is degraded
- The maximum heat transport ability of a heat pipe at a given temperature is determined by the most constraining limit

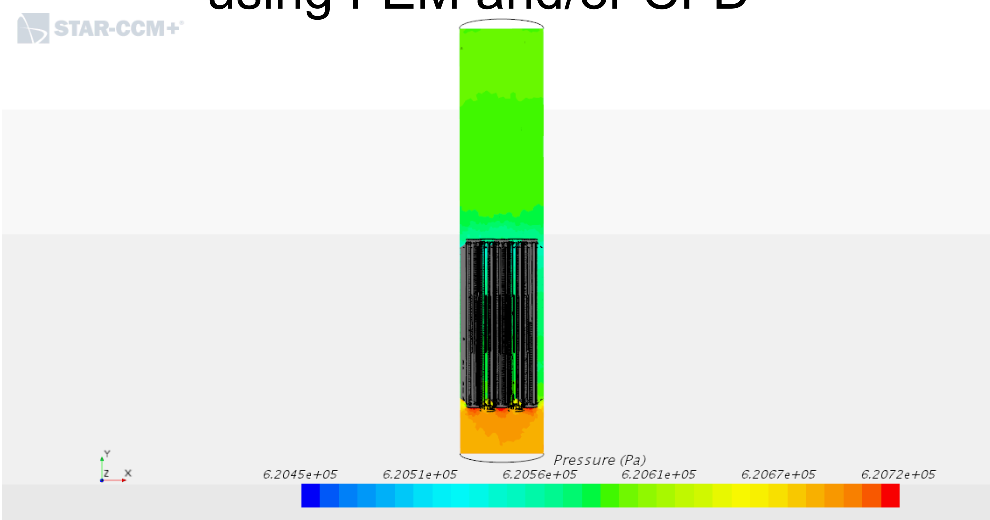
Mechanical Design

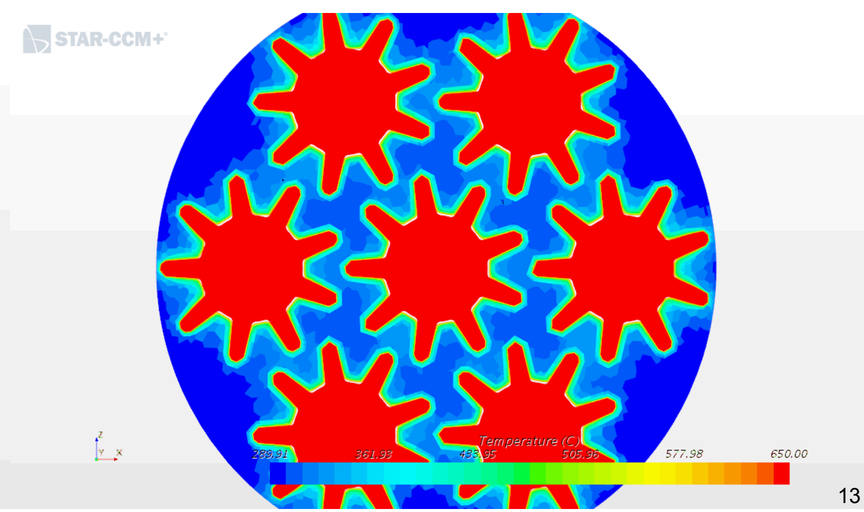
- Just because you can print it doesn't mean it's a good design!
- Use good engineering design rules
  - Radius corners to alleviate stress concentrations
  - Add ribbing to strengthen walls where needed
  - Wall thickness (e.g., calculate hoop stress, etc.)
- Printer resolution
  - Min. wall thickness = 500 μm
  - Min. feature size = 250-300 μm

Computational Modeling

- Heat pipe/HX scoping studies
  - Resistance network analogy
  - Excel model to determine heat exchanger performance
- Component design and topology optimization
  - Heat exchanger heat transfer and fluid dynamics using FEM and/or CFD

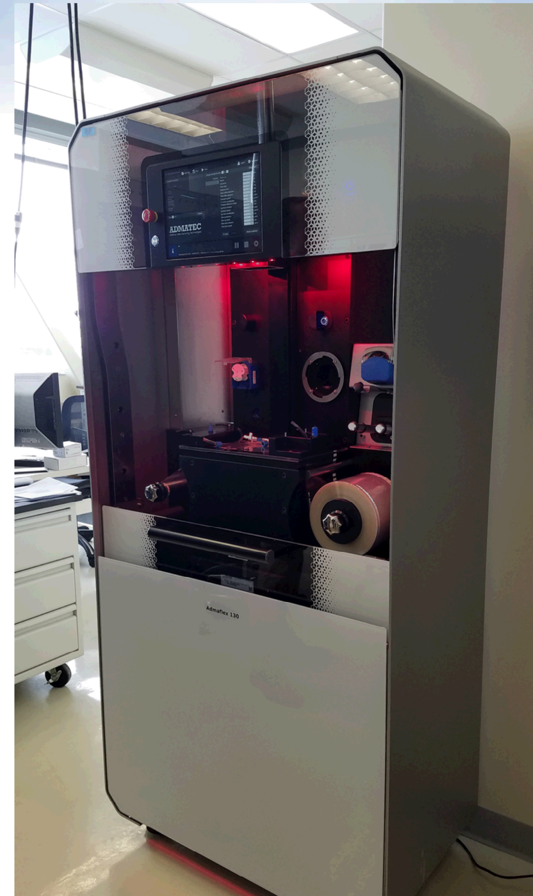


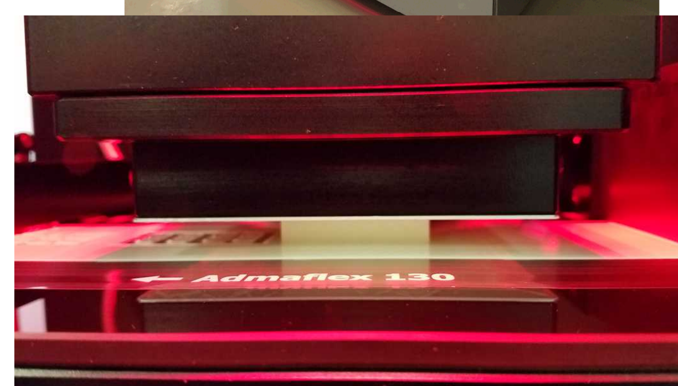




Admaflex 130 Digital Light Printer

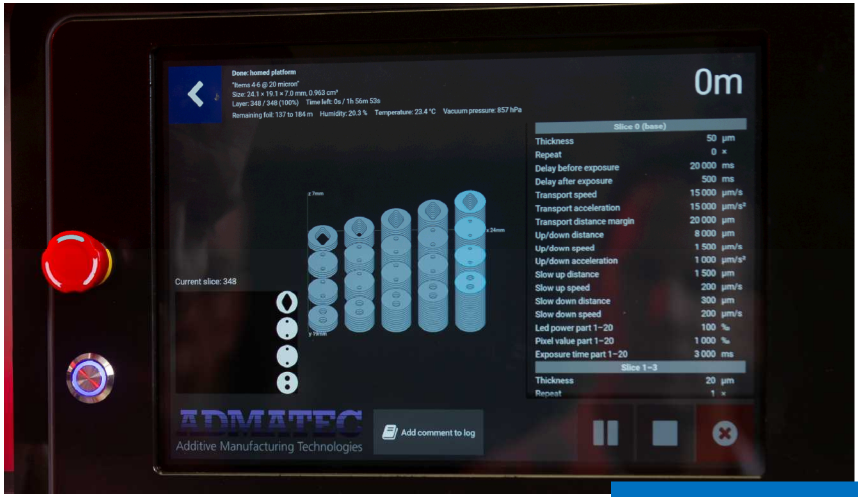
- The Admaflex technology provides an **open platform** that allows the production of a **multitude of materials** via UV curing layers of the liquid material on a build platform
- The AdmaPrint feedstock is specially formulated with a mixture of photosensitive resins and a solid load of powder (ceramic or metal), called **slurry**. The use of light curing and slurries allows achieving **high resolutions and very fine surface roughness** in printed products. In addition, it prevents health hazards and (cross)contamination related to the use of dry powders. The AdmaPrint feedstocks can be used to print complex geometries, large and fine structures and a wide variety of functional products.
- Custom-developed slurries can also be used



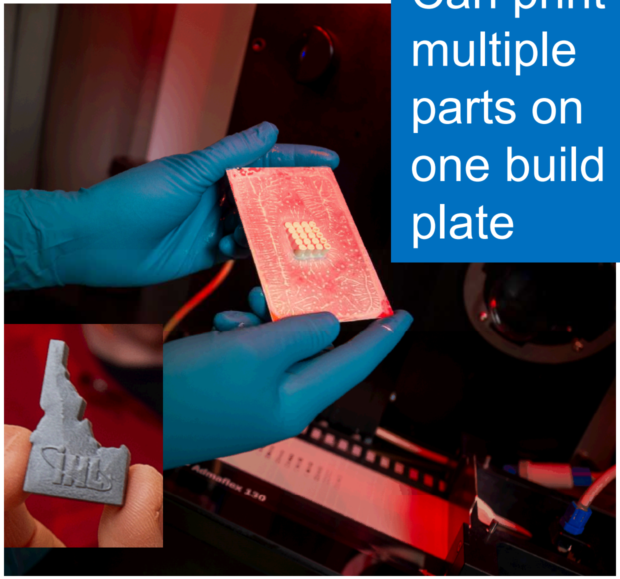


Procedure

- Import CAD file as .stl
  - Slice into layers (.slc file)
- Load slurry, reservoir, wiper
  - Metals require use of mixer to avoid particle settling
  - Metal slurry is less viscous than ceramic slurry
- Use filter with ceramic resin (don't need for metal resin)
- Perform depth of cure (DOC) study
  - Set exposure and light intensity
    - Note that age of resin affects DOC
    - DOC=2*layer thickness
- Layer thickness
  - Metal = 20 μm
  - Alumina = 50 μm



Can print multiple parts on one build plate



Print Parameters

Scale Factors

- Al_2O_3 @ 50 μm Layers X, Y = 1.306 Z = 1.367
- Al_2O_3 @ 30 μm Layers X, Y = 1.297 Z = 1.388
- ZrO_2 @ 20 μm Layers X, Y & Z = 1.41
- All Metals X, Y & Z = 1.30

Feature/part sizes:

- Minimum Feature size = 250-300um
- Minimum wall thickness = 500-750um
- Maximum print volume = 55d x 95w x 390h mm (roughly 2” x 4” x 16”)

Overhangs that can be tolerated without supports:

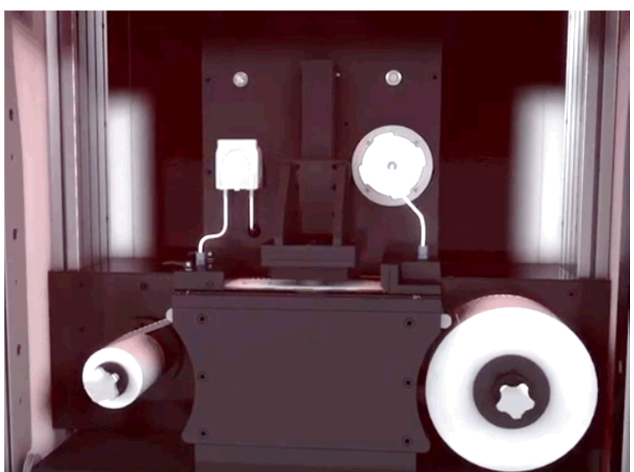
- Alumina & Zirconia 50^o
- Silica 80^o
- Metals 45^o

Practical Printing Considerations

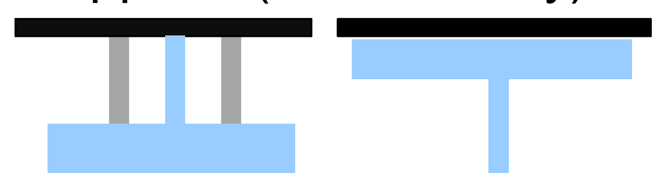
- Air doesn't affect the slurry, but humidity does (hygroscopic)
- Protect resin from stray UV light
  - Apply UV-protective film coatings to all exterior and interior windows in lab, as well as printer windows
- Resin shelf life is ~ 3 mos. (note European day/mo/yr vs. U.S. mo/day/yr)
- Resins are shear thinning (like Jello or yogurt), but with different μ
- Pauses should be kept to < 15 mins. to avoid particle settling
- There are differences between printing ceramics and metals:
  - The metal powder is dark; thus, particles absorb more light much faster than ceramic
- Be very conservative when printing the base layer - If base layer fails, entire part fails!
  - Base layer: exposure time = 4x DOC (only for ceramics, no base layer for metals)
  - Layers 1-5: exposure time = 1.5x DOC
  - Subsequent layers: exposure time = DOC

Mechanics of Printing

- Retract print head by:
  - 8 mm for first layers to clear slurry bed
  - 5 mm for subsequent layers
- Print head moves slower as it approaches the layer to avoid delamination
- Film-style vs. vat-type → uses film roll

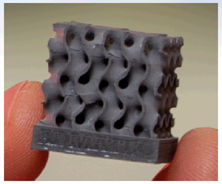


- Base plates:
  - 1/16” Al6061 sheet metal
  - Must coat plates with initial layer of resin when using metal slurry (don’t need to do this with ceramic slurry)
- Supports (can be tricky)



Supports req’d No supports

Controlled Porosity



- Want porosity to be:
  - Contiguous (not ping pong ball type) so that fluid flows from pore to pore
  - Homogeneous
  - Graded
- Ways to achieve porosity:
  - Adjust the amount of sintering
    - Incorporate additives which reduce sintering behavior
    - Hollow base grains or agglomerates
      - Multiple pore sizes can be achieved by using selectively sieved particles
  - Sacrificial particles and photocurable resin are mixed
  - Particles can be anything that evaporates or decomposes at the furnace temperature

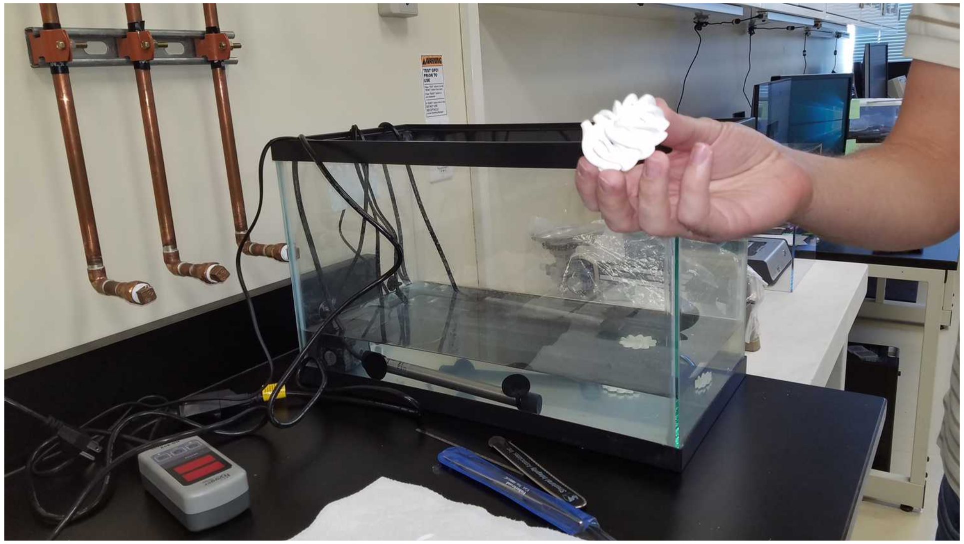
After the green body is printed……..

- Post-processing and heat treatment: Water and thermal debinding/sintering
- Metals: SST, Inconel, Copper
- Bonding/joining of components and sections
- Mechanical testing: Tensile, creep, fatigue. Testing at room temperature and elevated temperature. Compare to wrought materials.
- Thermal properties: Thermal conductivity and expansion.
- Microscopy: Examine structure and bond with SEM, XCT. Characterize porosity, defects, surface finish.
- Sensors: Determine best methods to incorporate fibers/sensors, high-thermal conductivity polymer composite filaments, create channels or pores, into AM parts. Evaluate impact on microstructure. Assess sensor performance.
- Fill heat pipes with working fluid and weld to seal

Rinse and Water Debind

- Rinse in tap water
- Water debind in deionized H_2O
  - Creates channels/pores that debind faster

Takes
24 hours



Thermal Debinding - Metals

- The printed metal parts need to be thermally debinded prior to sintering
- Metals require atmospheric control
  - Thermal process in a "forming gas" atmosphere (5% H_2 in N_2)

Step	Target Temp. (ºC)	Rate (ºC/hr)	Time (hr)
1	150	50	2.6
2	320	12	14.2
3	Dwell		4.0
4	400	12	6.7
5	Dwell		10.0
6	500	12	8.3
7	900	60	6.7
8	rT	100	9

Takes ~3 days

- Alumina tubes can only withstand rather slow heating and cooling rates (5-10ºC/min)

Carbon Removal

- The removal of the carbon that remains from the resin is crucial to the further processing
- Additional actions to remove the remaining carbon:
  - Reduce the pressure in the oven
  - Bubble the forming gas through a volume of water to introduce a minimal amount of oxygen in the debinding chamber

Sintering - Metals

- The debinded parts can be sintered under the following conditions
- Thermal process in a “forming gas” atmosphere (5% H_2 in N_2)

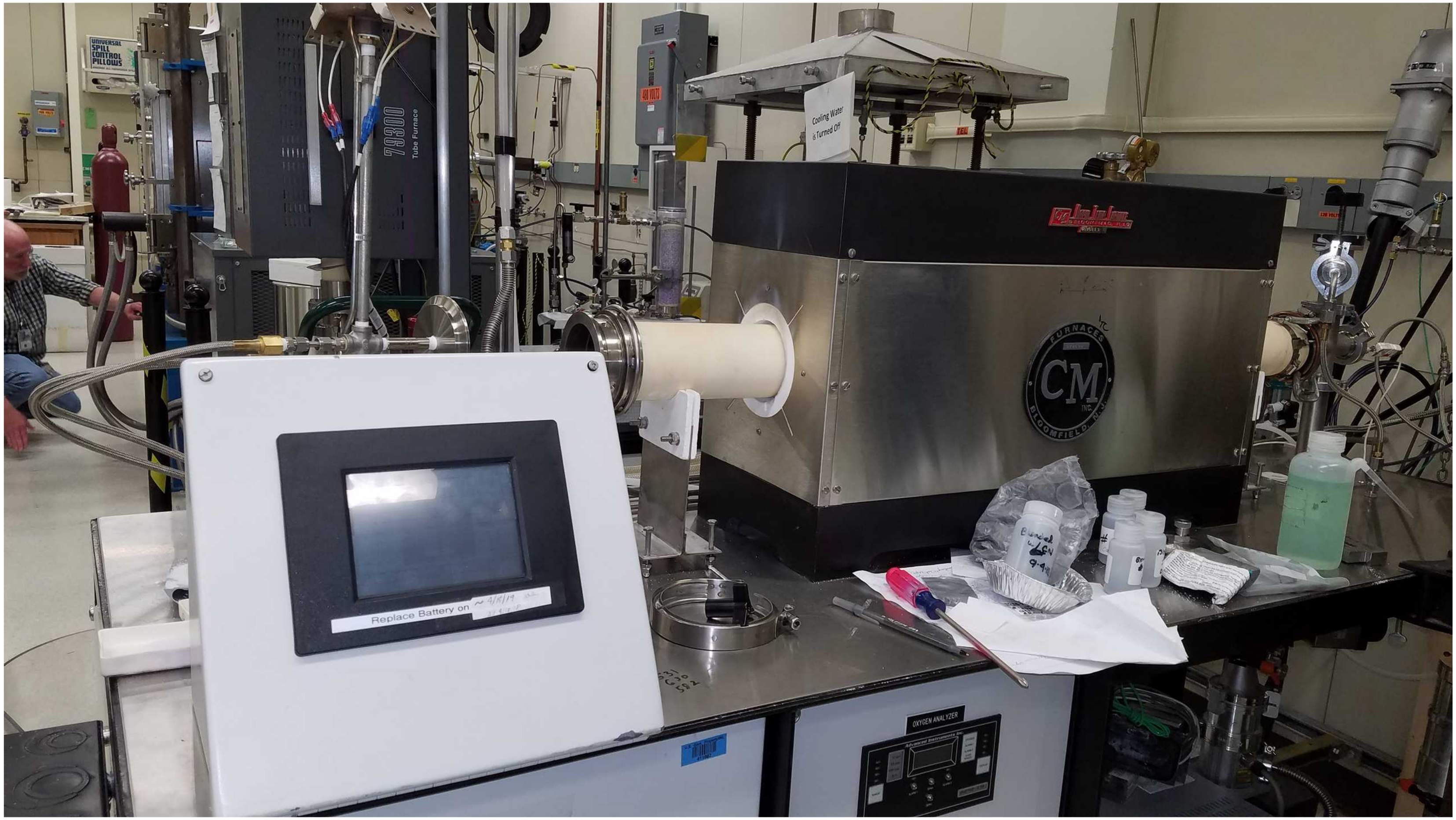
Step	Target Temp. (ºC)	Rate (ºC/hr)	Time (hr)
1	900	300	2.9
2	Dwell		3.0
3	1250	175	2.0
4	Dwell		0.83 (50 min)
5	kT	300	2.0

Takes ~11 hrs.

- The 5% hydrogen environment is considered flammable by our safety people and therefore will require a burnoff unit and interlocks
- The non-flammable limit is 2.8% hydrogen

Furnaces

Small CM Tube Furnace 1600ºC, air, N2, inert, inert-reducing, low vacuum, up to 100% H2, limited height ~2" and 8" long heated zone



Instrumentation Lab X-Ray CT

- Adapted Energy Computed Tomography
- 3D X-ray inspection for complex geometry specimens
- 225 kV microfocus X-ray tube
- Rotating through ports for long components and internal stage for smaller components
- Rotating stage for parts up to 0.5 in (13 mm) in diameter
- Resolution can exceed 15 microns depending upon specimen

Post-Fabrication Thermal-Mechanical Testing

Thermal-mechanical testing of materials and physical simulation of processes
Diffusion bonding
Tensile, torsion testing

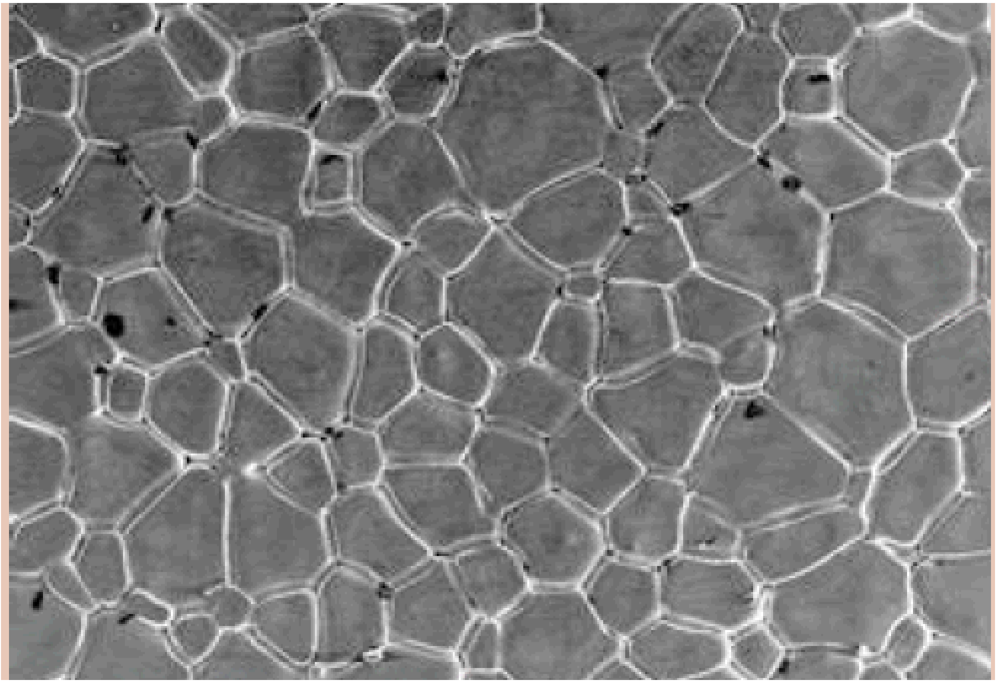
Thermal creep

Electric Field Assisted Sintering / Spark Plasma Sintering

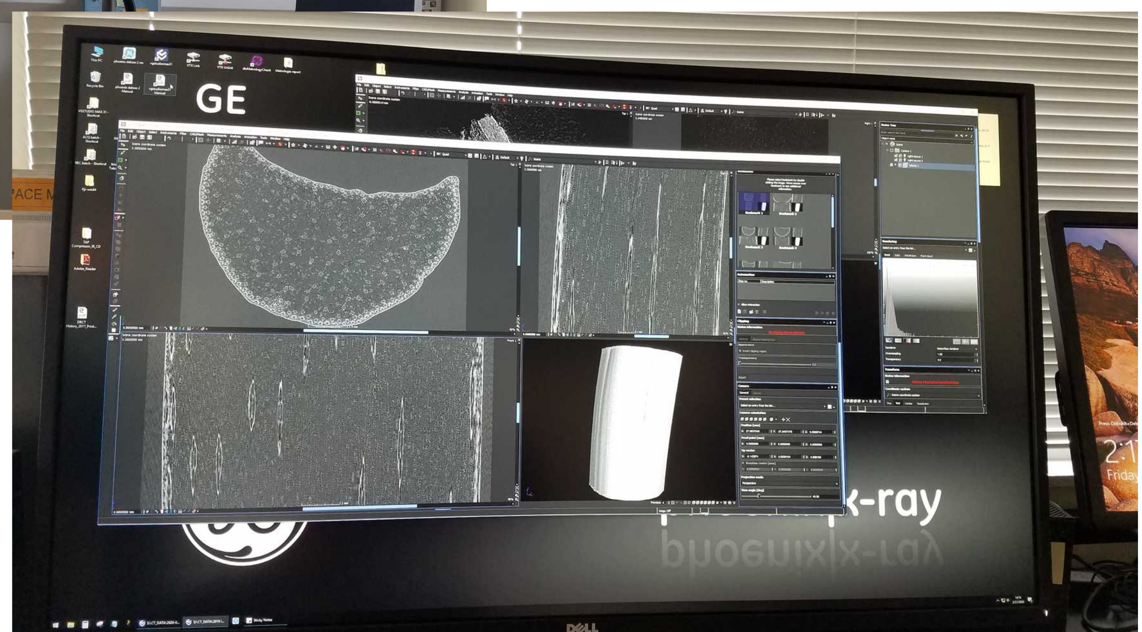
- INL currently has two main SPS installations.
  - Fuji Dr. Stinter SPS-515S with a 5kN load and 1800 amp capability located at the Center for Advanced Energy Studies
  - Thermal Technology SPS 25-10 capable of 250 kN (10 Ton) of axial load and 10,000 Amps of applied current located in the Radiological Spark Plasma Sintering (RSPS) facility at the new Advanced Fuel Fabrication (AFF) laboratory located at Idaho National Laboratory's (INL) Materials and Fuels Complex (MFC). . The system is capable of operating based on thermocouple feedback for low-temperature sintering or optical pyrometry feedback for high-temperature sintering. To maintain high quality powdered materials and to facilitate the use of radiological materials, the sintering furnace is integrated with an inert atmosphere glovebox enclosure and purification system to maintain oxygen and moisture levels in the parts-per-million (ppm) range.
- Additionally, INL has recently invested nearly $3M in procurement of a large-format Spark Plasma Sintering unit capable of fabricating commercial-scale parts of up to 0.9m in diameter and over 500kg. This unit, a Thermal Technology DCS-800, is the largest known SPS in the world with 150,000 amp and 800 ton load capabilities. INL is also developing an analytical, beam line SPS for in situ characterization of EFAS processing using neutron or X-ray sources during sintering.

Microstructure Examination

- Examine 3D printed structure using scanning electron microscopy



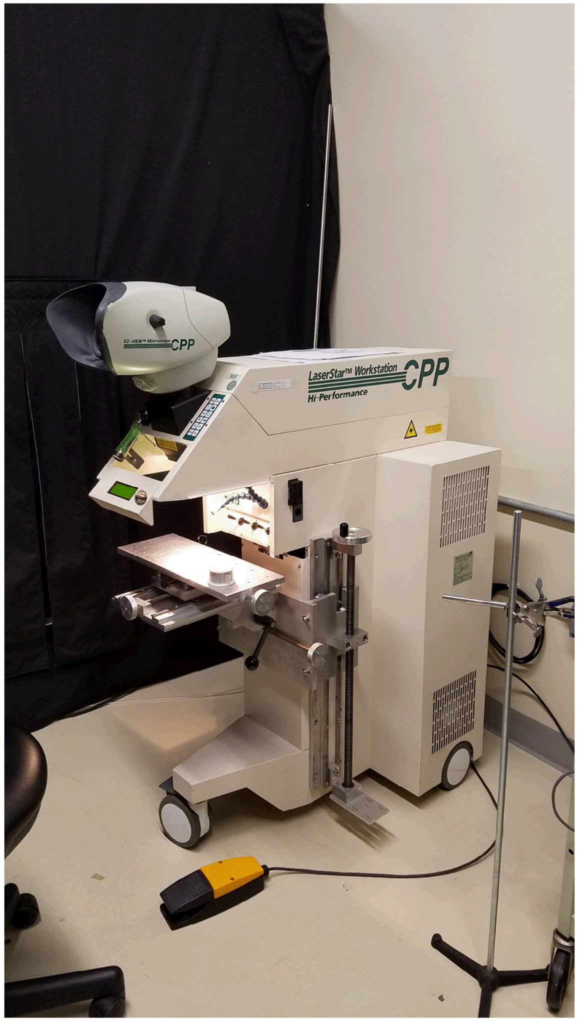
GE Phoenix vtomex X-Ray CT



High power for dense materials
Resolution: 500 nm to several microns

Optical and Laser Microscopes

Laser Welding of Heat Pipes

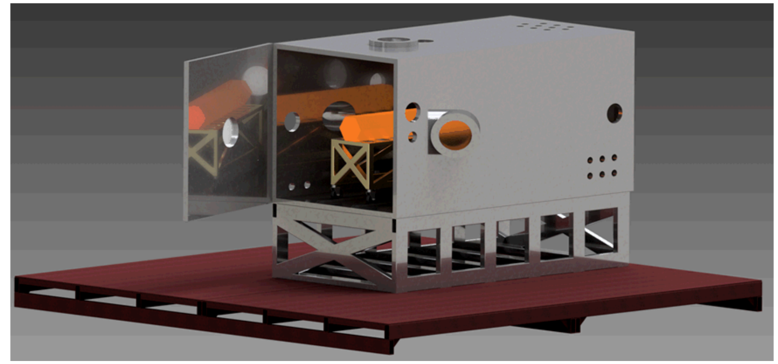


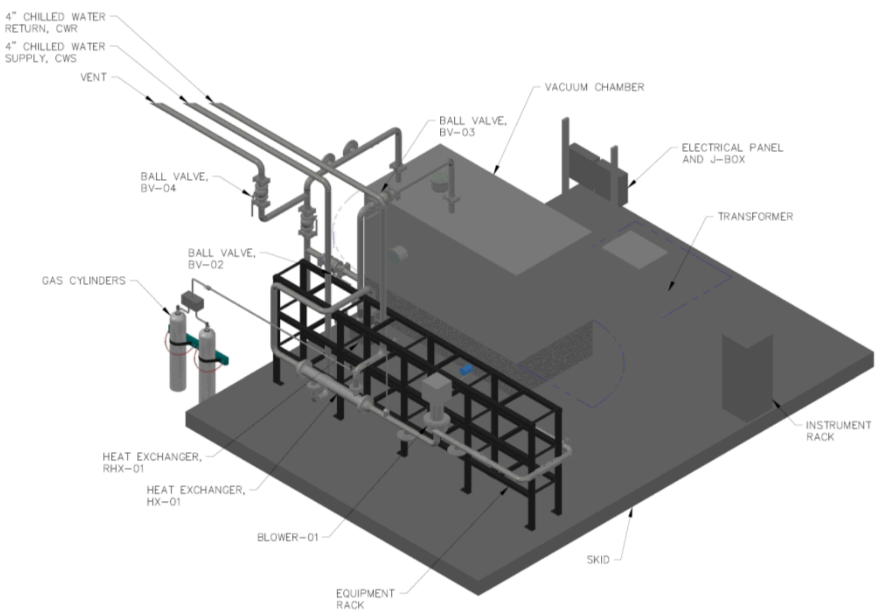
CoRE (Configurable, Removable, Extractable) Sensors

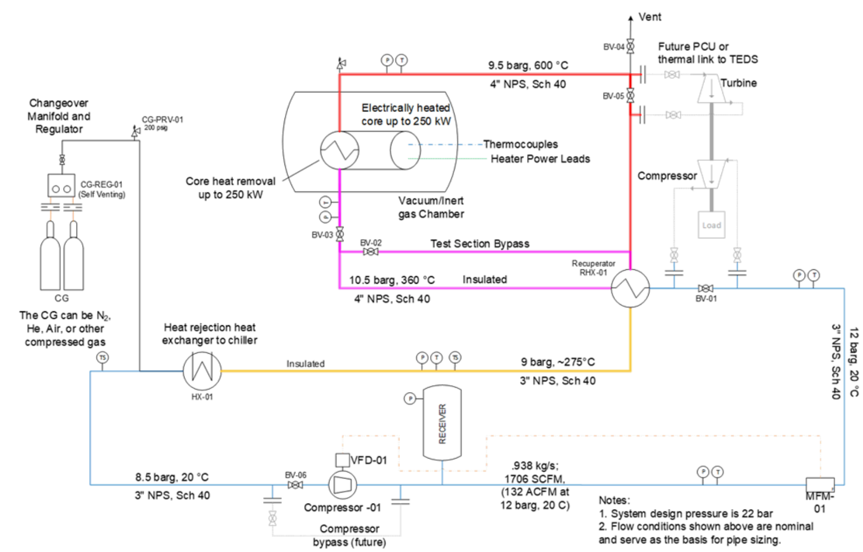
- INL is developing advanced sensor technology designed specifically to withstand the harsh conditions of high temperature and chemical exposure expected to be encountered in heat pipe-heat exchanger (HPHX) applications
- Robust fiber optics, CoRE instrumentation and printed sensors are some of the technologies being researched to monitor the in situ, operando performance of systems
- *Key* research questions that remain to be addressed:
  - What methods can be used to integrate sensors that don't create defects in the material or structure?
  - What are the best sensors to use for HPHXs?
  - How long do the sensors last under harsh operating conditions?
  - Can they be used for real-time sensing?
- Use data to evaluate performance and validate models

Performance Testing

- Conduct performance test of heat exchanger
- Obtain validation data for models
- Iterate and improve design

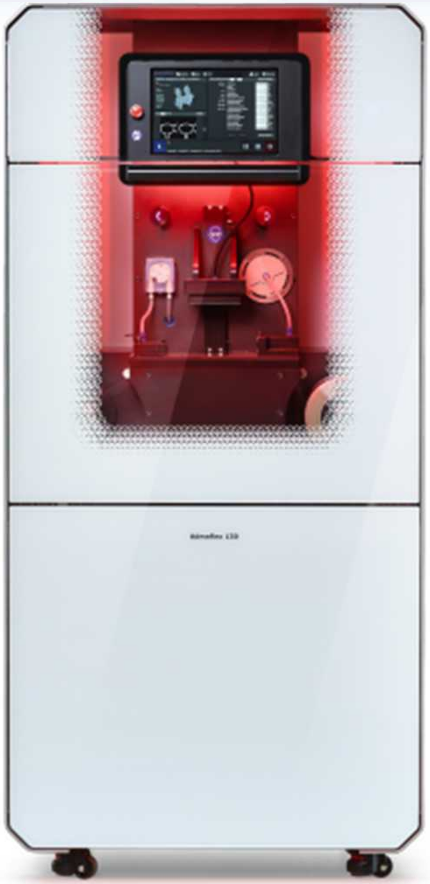






In Summary…..

- Perform thermal analyses and design
- Print and characterize coupons
- Thermal-mechanical testing of parts
- Fill heat pipes with working fluid
- Bond/join smaller sections
- Test entire heat exchanger



Acknowledgement

Work supported under DOE Idaho Operations Office Contract DE-AC07-05ID14517.


INL
Idaho National Laboratory