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MagnetoCaloric Hydrogen Liquefaction

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EMERALD ENERGY NW: JOHN BARCLAY

AMES: JUN CUI, IVER ANDERSON, BRANDT JENSEN

JUNE 2017

PROJECT ID # PD131

Overview

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Timeline:

- Project Start Date: 10/1/2015
- Project End Date: 09/30/2018

Barriers addressed:

- H: High-Cost Low Energy Efficiency of Hydrogen Liquefaction

Budget:

- Total: $2.25M
- Federal share: $2.25M
- Planned funding in FY17: $750k

Partners:

- Emerald Energy NW, LLC:
- AMES/ISU

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Relevance: Increase figure of merit, reduce system cost, and meet DOE targets

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Project Objectives:

1) Demonstrate H_2 liquefaction system for 25 kg/day H_2 with a projected FOM >0.5
2) Identify pathway to installed capital cost < $70M 30 tonne/day

30 tonne/day (small facility)	Claude cycles (current)	PNNL's MCHL project cycle (new)	DOE Target (2017)[1]
Efficiency	<40%	***70~80%***	85%
FOM	<0.3 (small facility) 0.35~0.37 (large facility)	***~0.6 (small facility) ~0.7 (large facility)***	0.5
Installed Capital cost	$70M[1]	***$45-70M***	~$70M
O&M cost	4%	***2.8%***	?
Energy input	10-15[1] kWh/kg H_2	***5~6 kWh/kg H_2***	12 kWh/kg H_2

[1] DOE, *Multi-Year Research, Development and Demonstration Plan, 2015*

$$FOM = \frac{\dot{W}_{Ideal}}{\dot{W}_{Real}}$$

$$\dot{W}_{Real} = \dot{Q}_C\left(\frac{T_H}{T_C} - 1\right) + \frac{T_H \int_{T_C}^{T_H} \Delta S_{IRR}\, dT}{\int_{T_C}^{T_H} dT}$$

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Approach: Increase efficiency by using by-pass flow and layered materials

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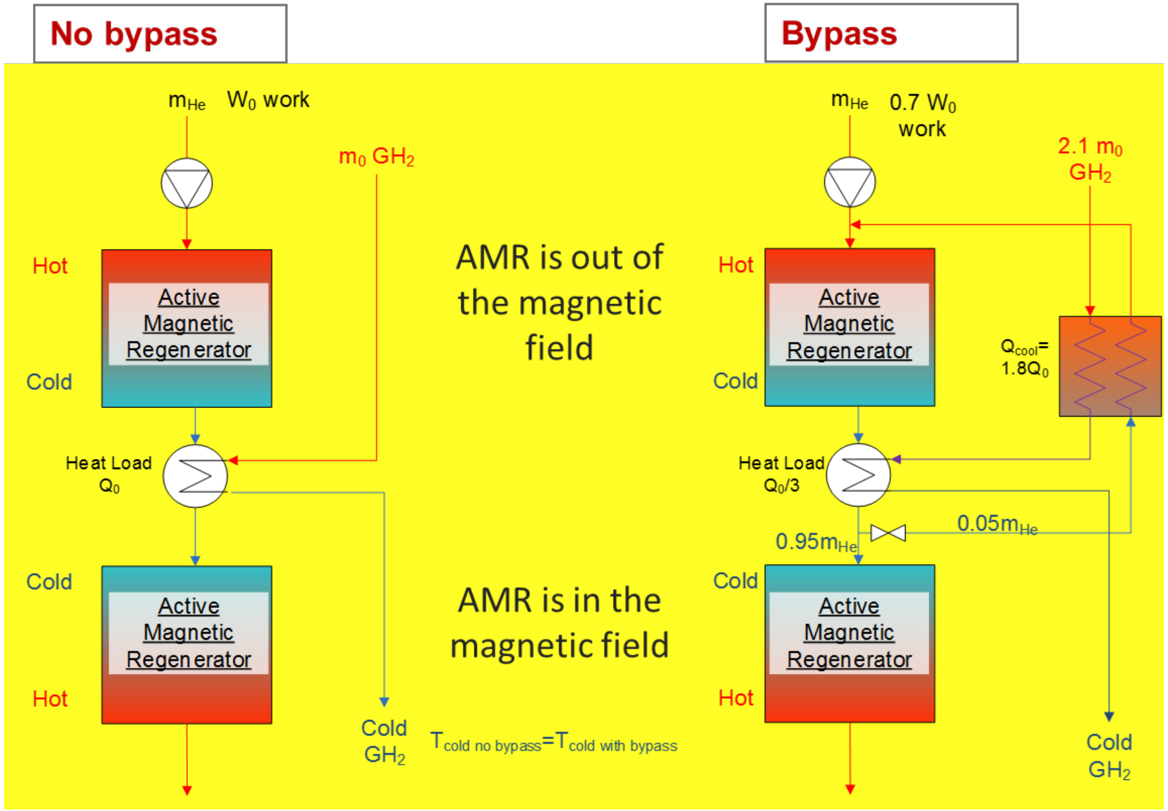
- *Reduces ΔT in process heat exchanger for large increase in FOM*
- *Eliminates intrinsic irreversibility of Active Magnetic Regenerator (AMR) cycle of magnetic refrigerants*
- ***88% reduction in magnetic material***
- ***Increase FOM from <0.4 to >0.75***



Approach: Analyze; Design; Build; Test Three Prototypes; Validate Models; Incorporate Results; Demonstrate LH_2

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Phase I (GEN-I & II) Phase II (GEN-III)

GEN-I (280-220 K) → GEN-II (280-120 K) → GEN-III (280-20 K)

- Refurbished Prometheus AMRR
  - Prepared high-bay lab @ PNNL
  - First successful GEN-I operation
- Measured performance @ 0.25 Hz
  - 3.3 T to 0.6 T; persistent mode
  - Dual 1.05 kg Gd regenerators
  - HTG - Helium @ 200 psia,
    - no by-pass flow of cold HTG
    - parasitic load; 285 K to 218 K
    - maximum T_{SPAN} ~318 K to 218 K
    - external load vs. T_{COLD}
  - **Measured by-pass flow up to ~10%**
    - **increased net cooling by ~25 %**
- Developed GEN-II design
  - Optimal use of bypass flow
    - **magnetic material drops ~85%**
    - **calculated FOM > 60%**
- Created design options for GEN-III
- Initial Cost analysis of LH2 AMRL

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- 5 invention PPAs; 2 patent appls
- **Modified GEN-I to liquefy propane**
  - Very successful demo of simple AMRL
- Rotary vs Recip AMR
  - Magnetic refrigerants to Ames lab
  - Choose belt vs wheel – solenoid magnets
  - Built test fixture for seal testing
    - can be sealed but friction too large
    - will modify GEN-I for GEN-II <$$
    - Fixed s/c magnet-cryocooler; 7 T
- Analyzed/Modeled GEN-II design
  - 8-AMRR stages with 1 mag mat'l/stage
    - Series design FOM > 50%
  - Invented 8-layer, 1-stage dual regenerator AMRR for 280 K to 120 K
  - Designed 8-layer dual reg; 1-stage AMRR
  - RDA spheres – successful-taken ~ 6 months longer than expected
- Prepared 2 journal papers;
- Detailed property data base done; Fortran performance model successfully running
- Assemble and test GEN-II;
- Validate Fortran model, update $$

- **Refine GEN-III design**
- **Design/build/test GEN-III**
- **Fully characterize AMRL for 1-25 kg/day LH2**
- **Fully Document AMRL**
- **Evaluate minimum of 2 non-rare earth based magnetocaloric materials (May be joint with CaloriCool)**
- **Complete Cost Analysis**
- **License Technology**

Green text is current year work, blue text is previous years, and copper is planned

AMRR= active magnetic regenerator refrigerator

AMRL: active magnetic regenerator liquefier

Approach: Project plans for H_2 liquefaction demonstration – Critical Path

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- Demonstrate & quantify by-pass operation (PNNL + EENW)
  - Why:
    - Increases cooling power
    - Reduces capital and O&M costs
  - Model results (EENW)

GEN-I

- Identify materials (PNNL+ EENW), synthesize & characterize materials (AMES)
  - Materials identification:
    - Assume 20K decrease per layer; total 13 layers in 2 stages
    - Need to have 2nd order phase transition
    - Need to have correct T_c
  - Materials synthesis and characterization
- Layer material demonstration (PNNL + EENW)
  - Why have other attempts not achieved expected results?
  - How to mitigate

GEN-II

FY18
- Ortho- Para H_2 (PNNL +EENW)
  - Catalyst selection
  - Design

EMER
- Demonstrate H_2 liquefaction (PNNL + EENW)

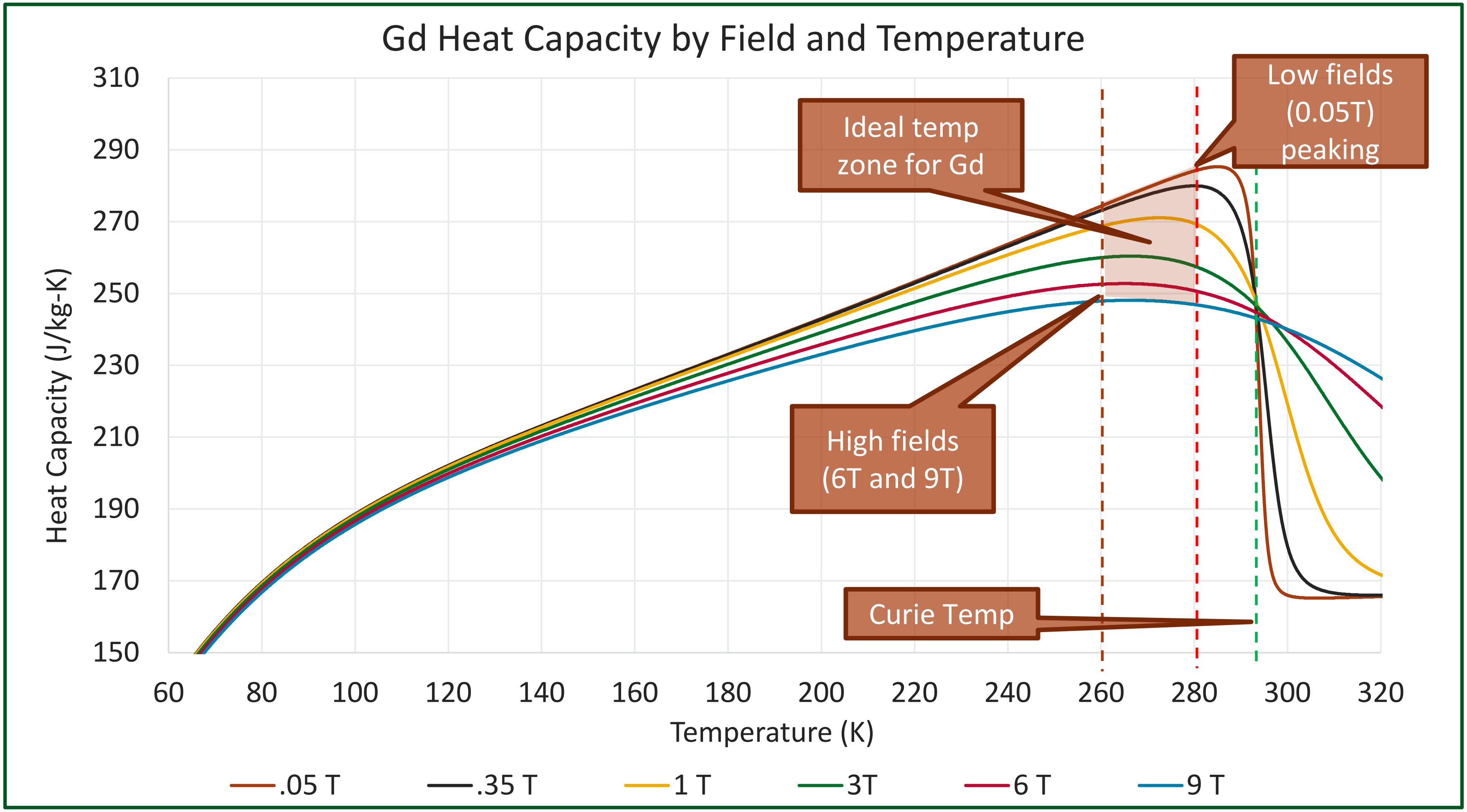
GEN-III

Major Milestones

Fiscal Year	Milestone	Status*	Comments
2017 Q1	PNNL will receive from AMES 8 alloy compositions reported in Q2 report. The amounts will be sufficient for 1 AMR (2 regenerators) plus 50-100 grams extra	75%	Taking longer to synthesize materials than expected
2017 Q2	Demonstrate GEN-II, as identified in FY16 Q3 report, operation. Demonstrate multi-layer (8+ layers) regenerator with by-pass and all subsystems operational with a measured ΔT of least of 45K. (note multi-layer regenerators of more than 3 layers have never been successfully demonstrated in the literature. This is a stretch milestone).	65%	Delayed to 06/01/17
2017 Q3	GEN-II demonstrate temperature span of 280-160 K	40%	Due 06/30/17
2017 Q4	GEN-III design is completed; Design is reviewed by program manager. Working with TDM, finalize path forward.	15%	

* Status as of April 2017

Accomplishment: Bypass is proportional to field dependent heat capacity



Data measured by AMES in FY17

AMRR cooling power is given by several key variables; e.g. Gd

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$$\dot{Q}_{Gd}(T_{COLD}) = \nu Fr_{COLD} M_{Gd} C_{Gd}(T_{COLD}, B_{0.6T}) \Delta T_{CD}(T_{COLD}) No_{Reg}$$

- $\dot{Q}_{Gd}(T_{COLD})$ is the cooling power in W
- ν is the AMR cycle frequency in Hz (typically 0.1-2 Hz)
- Fr_{COLD} is a steady-state fraction of the demagnetized regenerator colder than the average T_{COLD} before hot-to-cold blow of helium heat transfer gas (typically 0.1-0.2)
- M_{Gd} is the mass of Gd in each regenerator in kg
- C_{Gd} is the total heat capacity in J/kg K of Gd at T_{COLD} and low magnetic field after demagnetization, ΔT_{CD} is the adiabatic temperature change in K from T_{COLD} upon demagnetization from high-to-low field
- No_{Reg} is the number of magnetic regenerators in the AMRR (2 in GEN-I)
- **All variables in this equation are predetermined except Fr_{COLD} (coupled to heat transfer fluid flow) and best determined by modeling**

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Accomplishment (FY16-17): Performance simulation of the active magnetic regenerator

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Performance can be calculated by solving 1-D PDE equations for fluid and solid in magnetic regenerator

Mass, momentum and energy for heat transfer fluid and energy for porous magnetic solid refrigerant & regen wall

Eqn 1
$$\frac{\partial \rho'}{\partial t}+\frac{\partial \rho' u}{\partial x}=0$$

Eqn 2
$$\frac{\partial \rho' u}{\partial t}+\frac{\partial\left(\rho' u^2\right)}{\partial x}+\alpha\frac{\partial P}{\partial x}-\frac{f\rho(1-\varepsilon)|u|u}{d_p}=0$$

Eqn 3
$$\frac{\partial \rho' U}{\partial t}+\frac{\partial \rho' u U}{\partial t}+P\frac{\partial(\varepsilon u)}{\partial x}+\frac{6(1-\varepsilon)\alpha_{fs}}{d_p}(T-\theta)+\frac{4\varepsilon\alpha_{fw}}{d_i}(T-\psi)-\frac{f\rho(1-\varepsilon)|u|u^2}{d_p}-\frac{\partial}{\partial x}\left(\lambda_f\varepsilon\nabla T\right)=0$$

Eqn 4
$$\rho_s'\frac{\partial U_s}{\partial t}-E_s-\frac{6(1-\varepsilon)}{d_p}\alpha_{fs}(T-\theta)-\frac{4(1-\varepsilon)\alpha_{sw}}{d_i}(\psi-\theta)-\nabla\cdot\left[\lambda_s(1-\varepsilon)\nabla\theta\right]=0$$

Eqn 5
$$\rho_w'\frac{\partial U_w}{\partial t}-E_w+\frac{4(1-\varepsilon)d_i}{\left(d_o^{\,2}-d_i^{\,2}\right)}\alpha_{sw}(\psi-\theta)-\frac{4d_i\varepsilon\alpha_{fw}}{\left(d_o^{\,2}-d_i^{\,2}\right)}(T-\psi)-\nabla\cdot\left(\lambda_w\nabla\psi\right)=0$$

These are coupled 1-D non-linear partial differential equations solved by finite difference numerical techniques with all materials' and fluids' temperature, pressure, flow, magnetic field dependence updated each micro time step. Original model developed in FY16, but updated with measured values of Tc, Cp, and magnetic moment

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Accomplishment (FY17): Experimental results validate temperature profile changes with bypass flow

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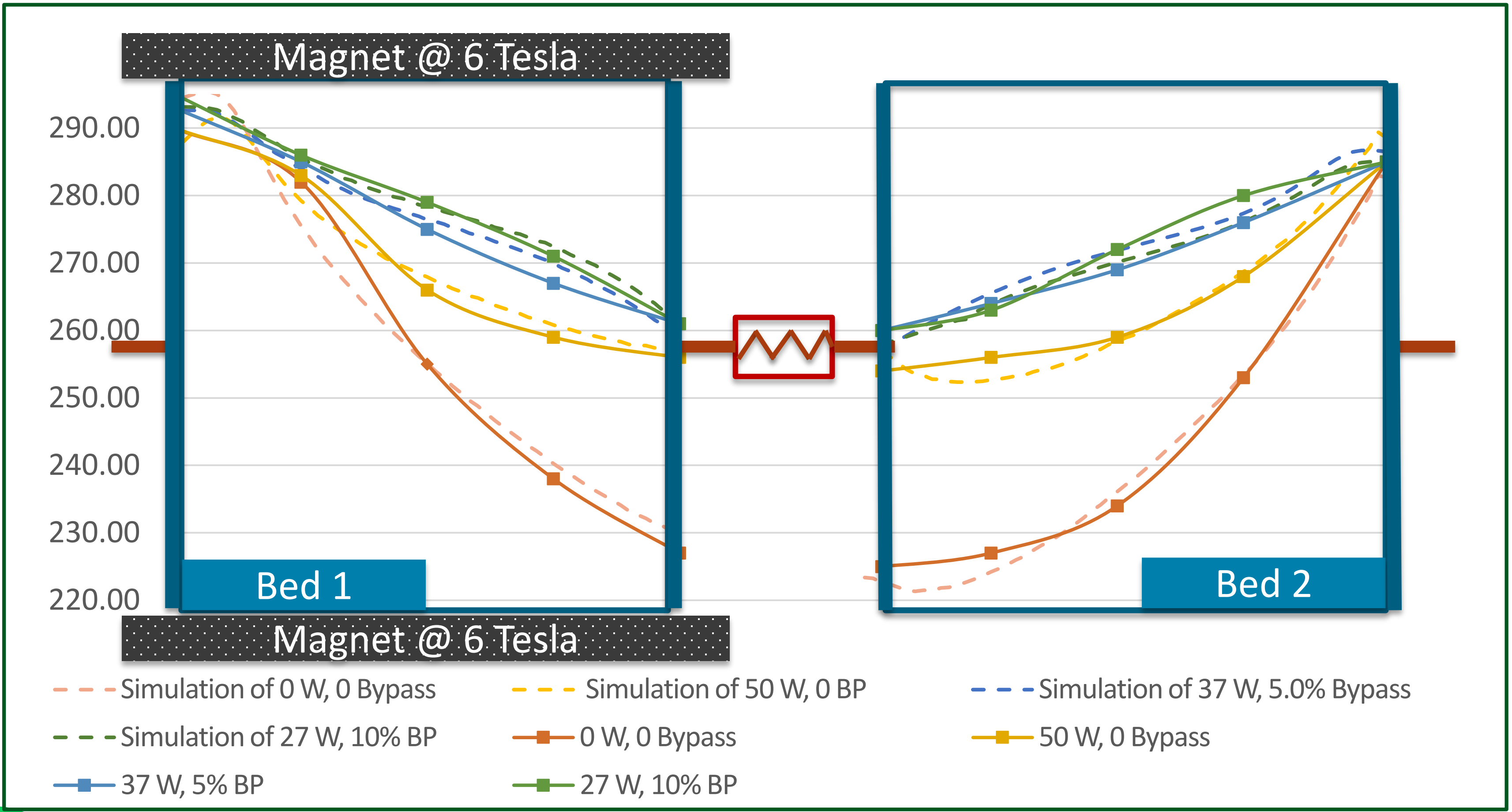
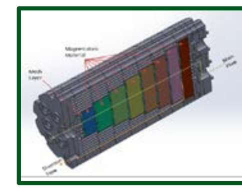
Temperature measurements from dual Gd regenerators
Configuration: 6 Tesla, 215 g Gd Beds, 0.25 Hz



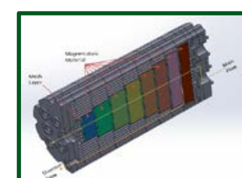
Accomplishment (FY17): Identified materials for 2-Stage H_2 liquefier

Material	Operating Temperature Span	Curie Temperature
	K	K
Gd	280-260	293
$Gd_{0.90}Y_{0.10}$	260-240	274
$Gd_{0.30}Tb_{0.70}$	240-220	253
$Gd_{0.69}Er_{0.31}$	220-200	232
$Gd_{0.32}Dy_{0.68}$	200-180	213
$Gd_{0.15}Dy_{0.85}$	180-160	193
$Gd_{0.27}Ho_{0.73}$	160-140	173
$Gd_{0.16}Ho_{0.84}$	140-120	153
$Gd_{0.23}Er_{0.77}$	120-100	132
$Ho_{0.90}Gd_{0.10}Co_2$	100-80	110
$Ho_{0.95}Gd_{0.05}Co_2$	80-60	90
$Gd_{0.5}Dy_{0.5}Ni_2$	60-40	70
$Dy_{0.75}Er_{0.25}Al_2$	40-20	50

Stage 1
(GEN-II)



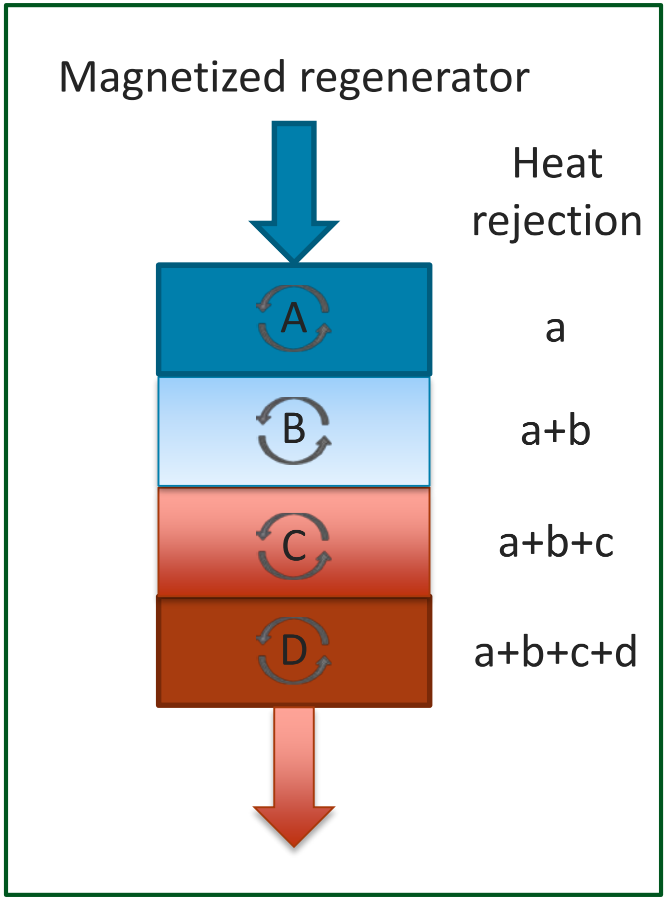
Stage 2
(GEN-III)



Accomplishment FY17: GEN-II design uses *unique* 8 layer design with bypass to achieve ΔT ~160K operational span

- Multi-layer design
  - Others have tried, but never worked as expected.
  - Used same volume of material for each layer
  - Used same flow through each layer

- ***We hypothesize that one needs to consider each layer as an individual cycle***
  - Change the material amounts as needed
  - Control the heat transfer fluid flow for each layer
  - Provisional patent submitted



Accomplishment FY17: GEN-II design uses *unique* 8 layer design with bypass to achieve ΔT ~160K operational span

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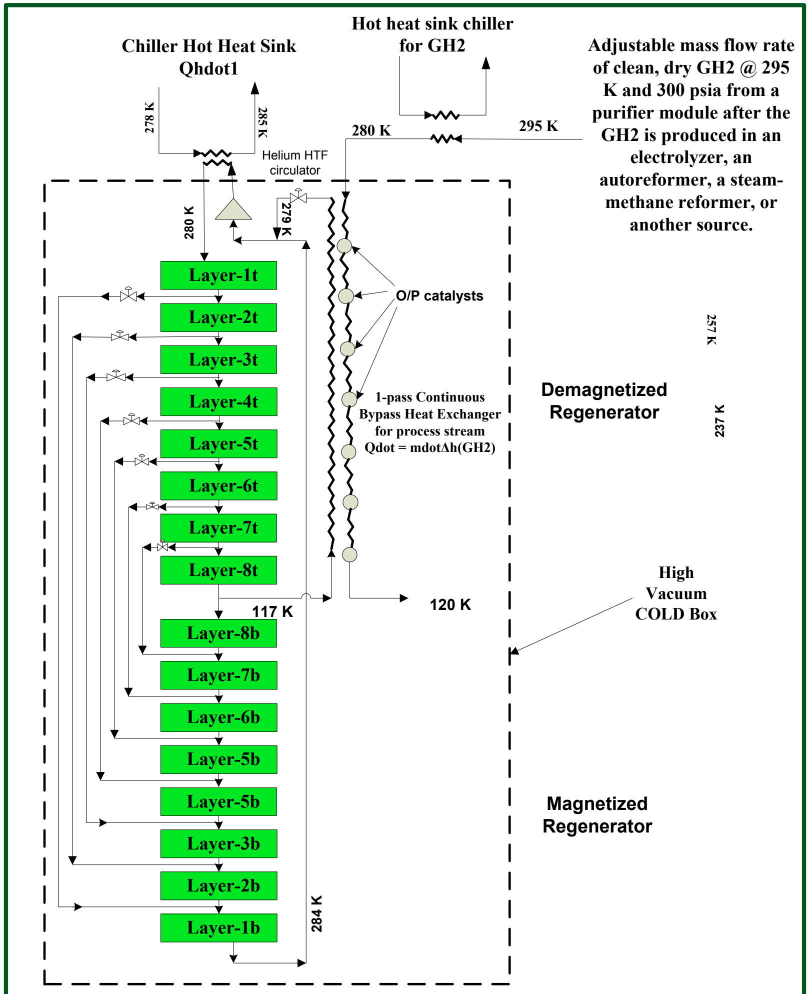
- GEN-II Design
  - Heat transfer fluid flow to get heat and cold out of each layer
  - Diverted flow since each layer requires a different amount of flow
  - By-pass flow comes out after 8th layer



BPFD Schematic of a 8-layer dual AMR for AMRR with continuous bypass flow operating between 280 K and 120 K. JAB-030916

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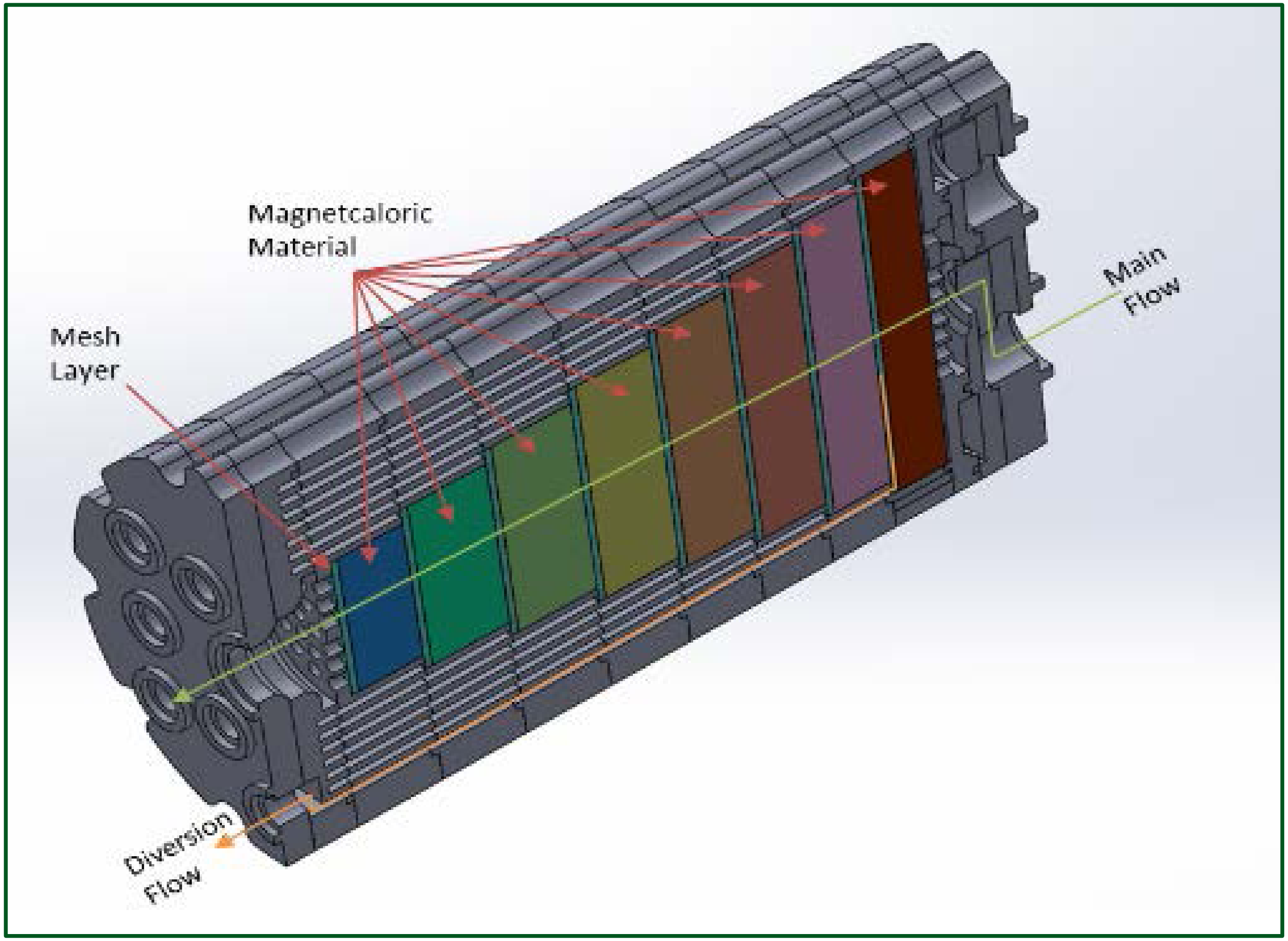
Accomplishment (FY17) GEN-II Design is unique 8 layers with heat transfer fluid bypass & diversion flow



Model predicts the 8 layer device will achieve 160K temperature change

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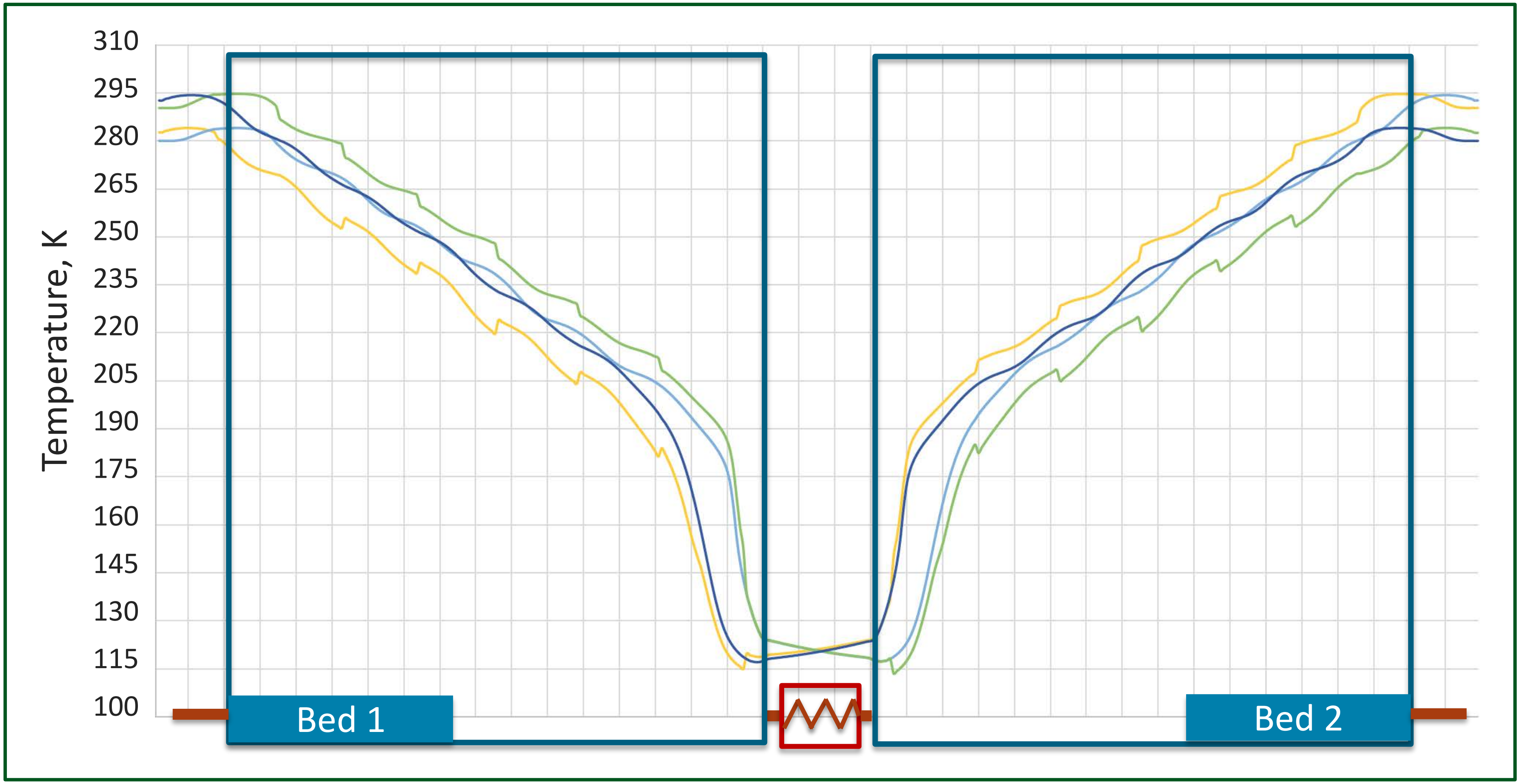

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Accomplishment FY17: 8 optimal magnetic refrigerants identified for GEN-II design

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AMES Synth-esized*	Layer	Material	Average T_{HOT}/T_{COLD} (K)	Curie Temp (K)	Mass of magnetic material/layer (grams)	Work rate/layer (W)	Q_{HOT} /layer (W)
Y	1	Gd	280/260	293	268	11.0	132
Y	2	$Gd_{0.9}Y_{0.1}$	260/240	274	258	9.9	110
	3	$Gd_{0.3}Tb_{0.7}$	240/220	253	235	8.8	90.7
	4	$Gd_{0.69}Er_{0.31}$	220/200	232	202	7.6	71.9
	5	$Gd_{0.32}Dy_{0.68}$	200/180	213	172	6.3	54.4
Y	6	$Gd_{0.15}Dy_{0.85}$	180/160	193	139	4.9	38.4
Y	7	$Gd_{0.27}Ho_{0.73}$	160/140	173	100	3.4	23.9
Y	8	$Gd_{0.16}Ho_{0.84}$	140/120	153	57	1.8	11.0

*Synthesized as of 04/20/2017, details in back-up section

different heat transfer fluid flow required in each layer

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Accomplishment (FY17): Heat capacity measurements complete and used to refine by-pass flow prediction/design at high fields (AMES)

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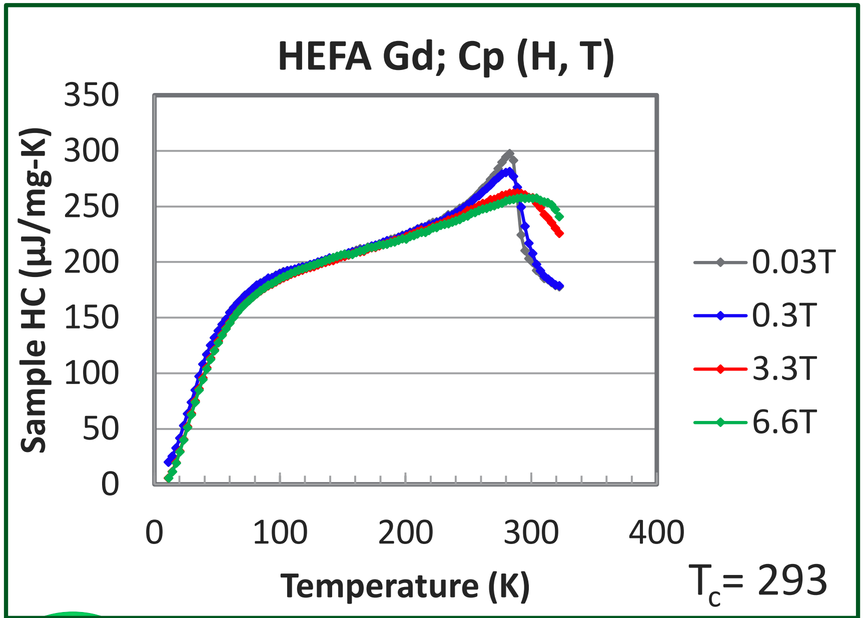
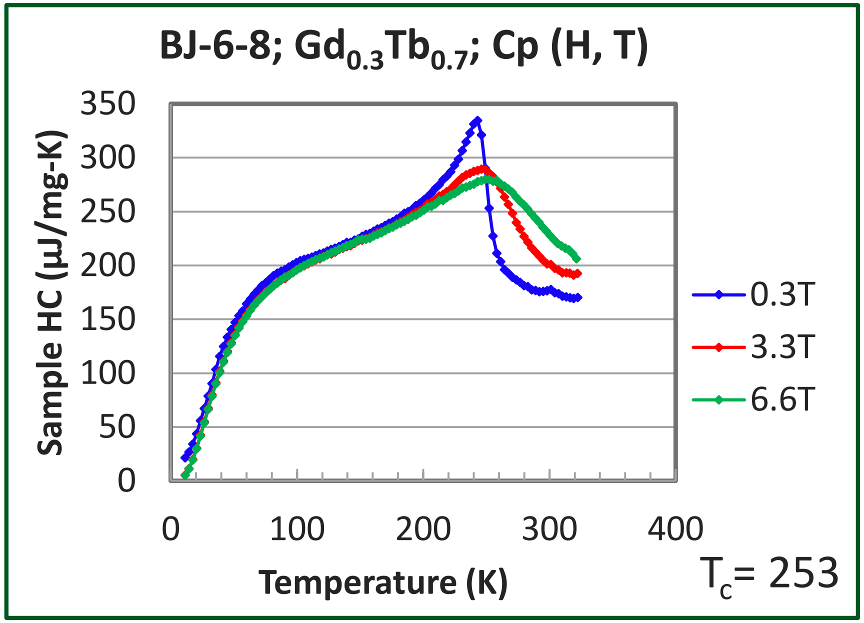
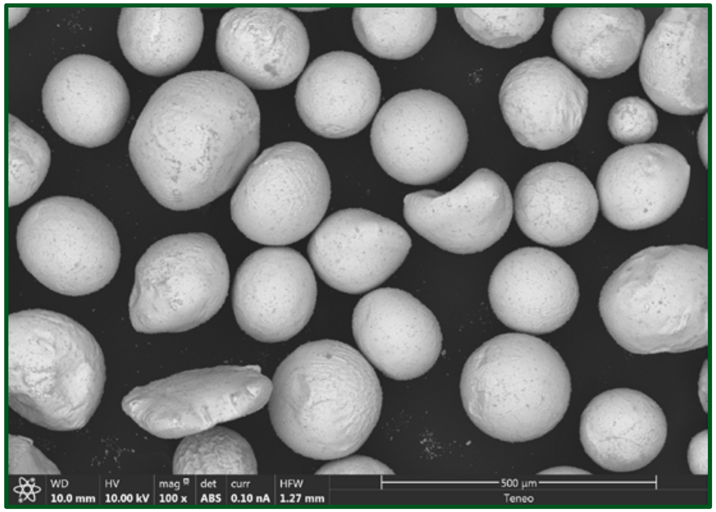
- Characterization of Cp, T_c, and magnetic moment for each material
- Heat capacity measurements – all 8 alloys completed
  - Very *accurate* but *time consuming* - 1 week per sample
  - Hardware adjustments were necessary
  - C_p (H, T) validates our molecular field model

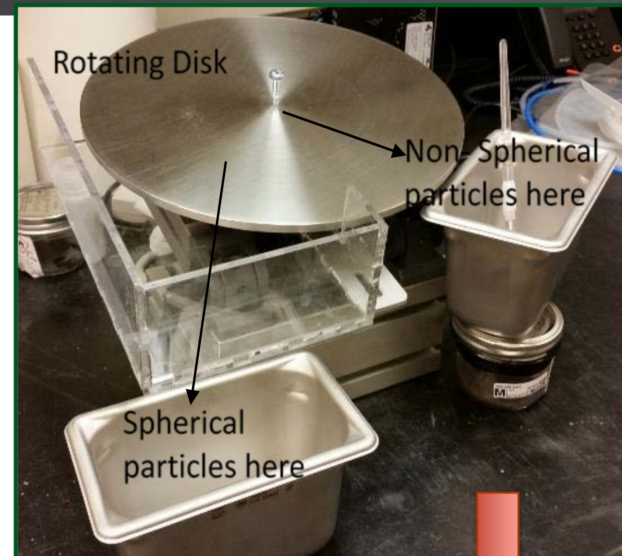




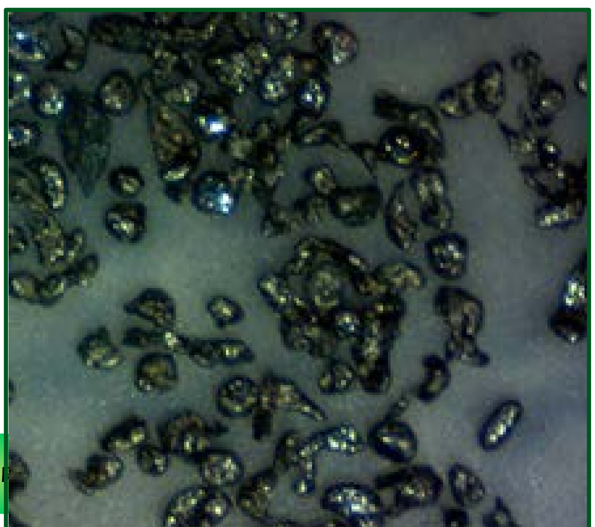
Accomplishment (FY17): PNNL shape separator developed to remove non-spherical particles

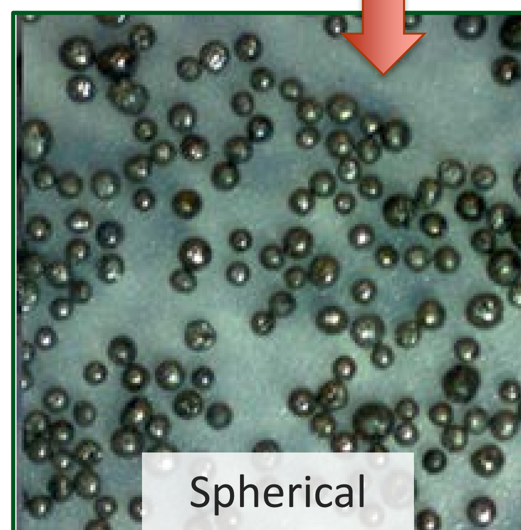
Materials from AMES





Separation after 3 passes





Reviewer Responses

Review	Response
The authors need to select a cascade of materials with appropriately located phase transition temperatures to make this work. It is unclear whether these materials have been selected and properly characterized. It is also unclear whether the performance predictions take this into account.	We have identified materials for a 2 stage liquefaction system. The first stage, GEN-II, has 8 materials which were characterized by AMES in FY17. AMES will synthesize and characterize the stage 2 (GEN-III) materials in FY18. The performance predictions take this into account.
The investigators should clearly identify key challenges of magnetic refrigeration that were not attempted or satisfactorily solved…	Key areas which were not satisfactory resolved in the Prometheus work include: 1. By-pass operation demonstration. Prometheus proposed this, but never demonstrated. 2. Multi-layer operation. 3. Advanced heat exchangers and Ortho-Para catalyst integration. 4. System design to go from room temperature to liquid
Inclusion of industry partners.	Emerald Energy NW (John Barclay) is an industrial partner who works closely with the PNNL staff. We are in discussions with several industry groups who expressed interest, but wanted to see the multi-layer operation.
The project has too many risks and assumptions for one project and should be narrowed	We focused the work on the demonstration of two critical aspects- by-pass operation and multi-layer design. We eliminated other issues to narrow the focus.
Would like to see a breakdown of the proposed system costs	This is included in the reviewer only section

Collaborations

Partner	Project Roles
DOE	Sponsorship, steering
Emerald Energy Northwest LLC	Working with PNNL on: - Design - Data analysis - Cost analysis
AMES Laboratory / ISU	Materials characterization Material synthesis
HDTT	Provide critical feedback and direction
CaloriCool (EMN)	Working with organizers to ensure new materials for our application will be developed

We are focusing our efforts on the key remaining challenges and barriers

- FY17
  - 8 layer operation
    - This has been a challenge for research groups in the past
    - Our data reveals the problem is in the HTF design used
    - Mitigation: Demonstrate our hypothesis and test new HTF design
  - Rotating disk atomization
    - Literature is for single compounds
    - Alloy atomization is very different

- FY18
  - GEN-III demonstration of liquefaction
  - Ortho-Para catalyst selection and integration – we have developed several concepts for integration and will test them in the next year

Proposed Future Work

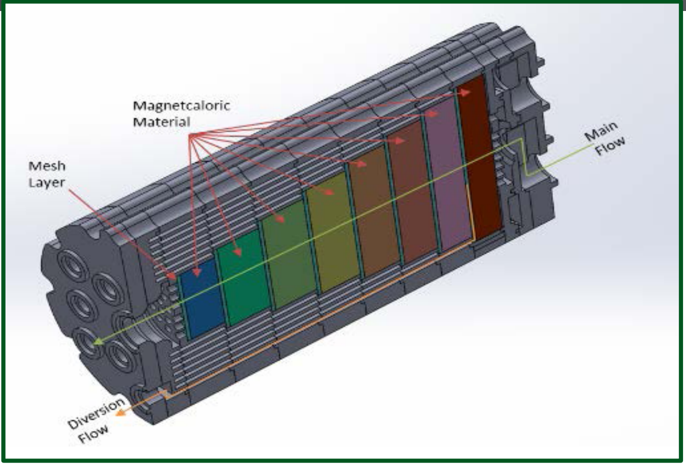
- FY2017
  - Integrate magnetic materials into GEN-II
  - Test GEN-II
  - Revise GEN-III concept design based on learnings from GEN-II
  - Find additional commercial support
- FY2018
  - Receive remaining compositions for the multi-stage GEN-III system
  - Construct, commission and test GEN-III system
  - Hydrogen liquefaction
  - Complete economic analysis
  - License patented technology with collaborative agreement to develop first commercial plant

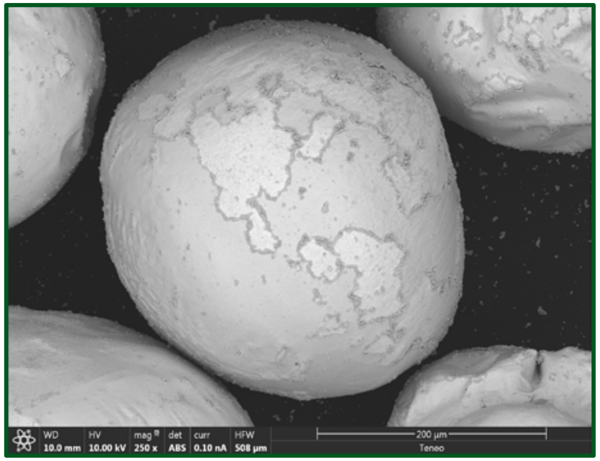
Technology transfer activities resulting in multiple patents and industrial interest

- Industrial
  - Met with multiple companies to discuss application of MCHL
    - Hydrogen liquefaction
    - Stranded NG
    - High value gas separation and recovery
- Potential future funding
  - Reaching out to other DOE agencies (EERE-AMO & FE) for alternative applications
- Patents and Licensing
  - 5 invention disclosure reports submitted
  - 2 patents applications submitted
  - 2 provisional patent applications submitted

Summary

- 6 T operation demonstrated
- Selected 8 materials (down selected from 16)
- 280 K- 120 K system invented
  - Modeled system
  - Unique flows design– diversion flow invented
  - 8 layered
- Measured key material properties
- 5 materials converted to powders
  - New separator demonstrated
- Mapped regenerator temperature profile
- Multiple papers and patents pending





Mission

We transform the world through courageous discovery and innovation.

Vision

PNNL science and technology inspires and enables the world to live prosperously, safely and securely.

DISCOVERY *in Action*

Values

integrity

CREATIVITY

courage

Impact

COLLABORATION

PNNL-SA-94292

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Technical Back-up slides

Approach:
Magnetocaloric liquefaction has potential to increase the FOM by 2x compared to conventional Claude process

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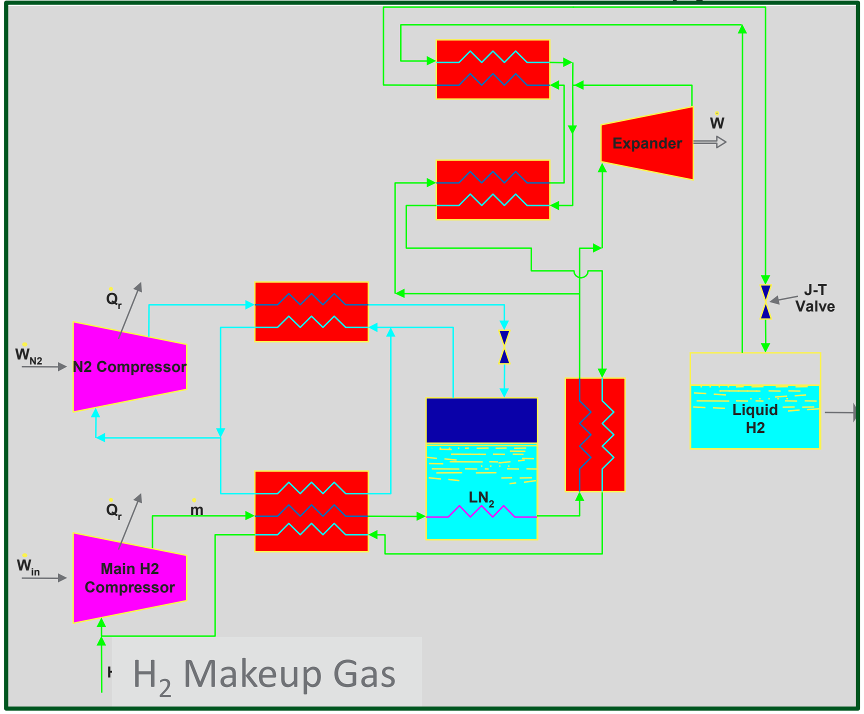
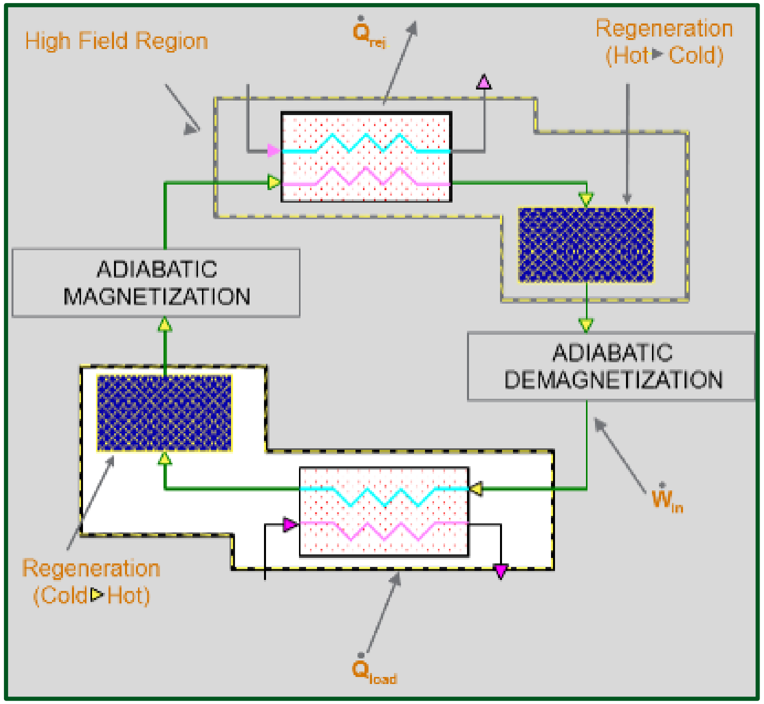
- Conventional- Claude Process
  - Low efficiency, FOM = 36%
    - Theoretical 4 kWh/kg H_2
    - Real 11 kWh/kg H_2
  - Why?
    - LN_2 pre-cooled Claude cycle
    - 50% irreversible entropy

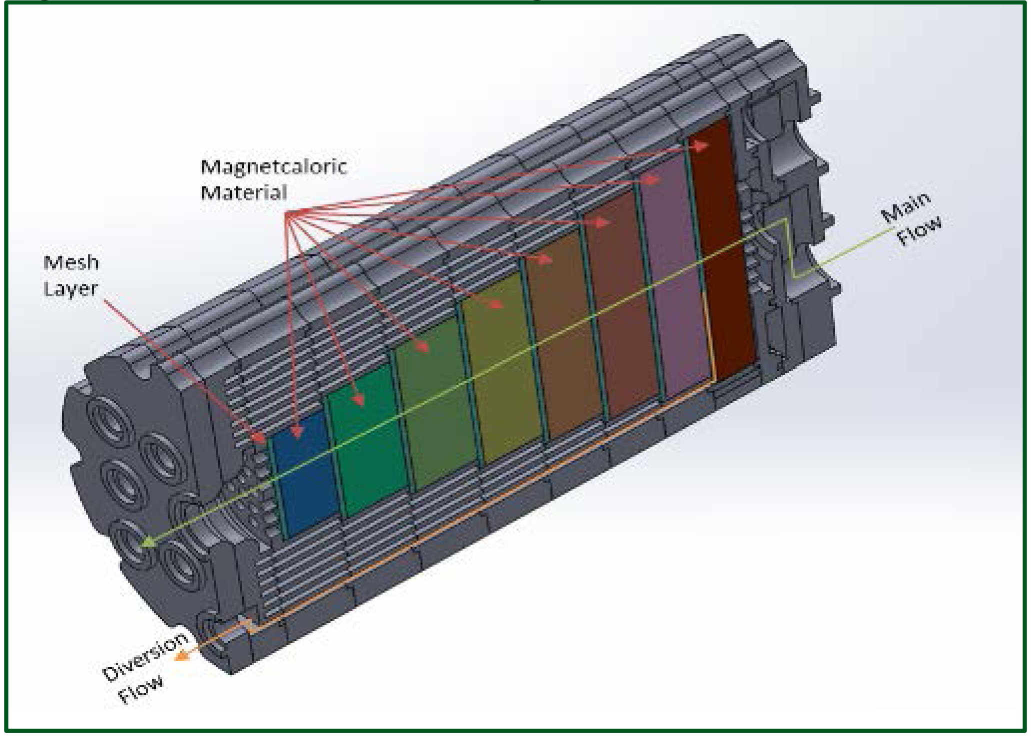


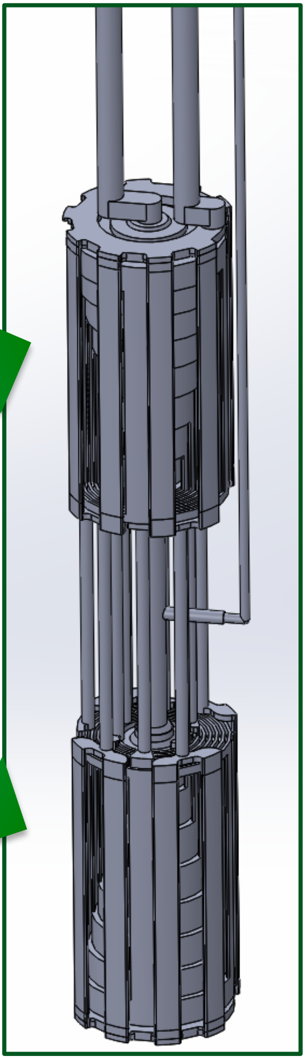
- MagnetoCaloric Liquefaction
  - FOM = 60+% (projected)
  - Solid magnetic materials
  - Entropy manipulated by magnetic fields – high reversibility
  - Bypass flow is unique to MCL



GEN-II Design is unique 8 layers with heat transfer fluid bypass & diversion

- GEN-II Design
  - Heat transfer fluid flow to get heat and cold out of each layer
  - Diverted flow since each layer requires a different amount of flow
  - By-pass flow comes out after 8th layer
  - The regenerator and the piping are shown in these two figures





Rotating Disk Atomization System for RE MC Powders

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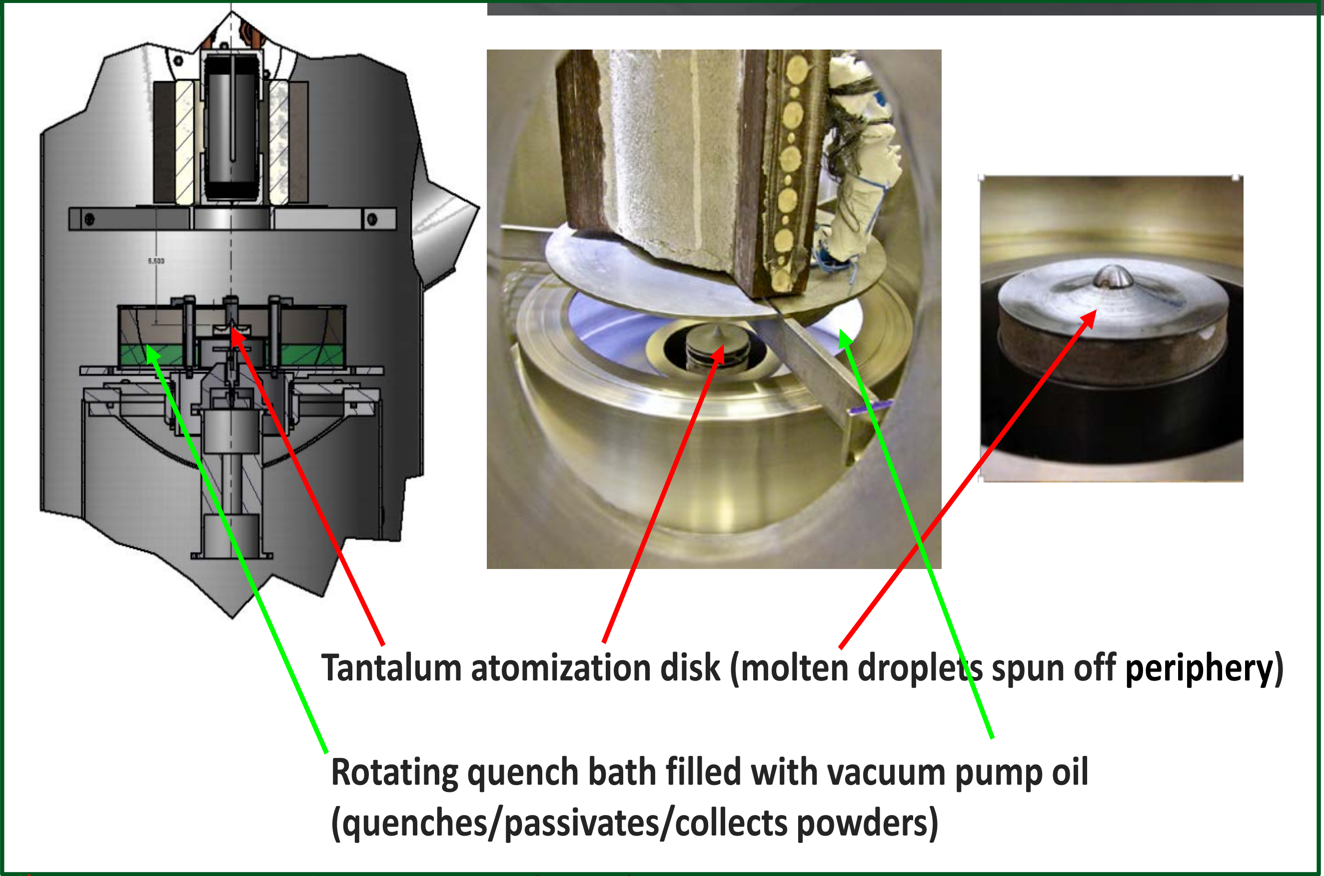

- Rotating disk atomizer (RDA) makes reactive metal powders.
  - Co-rotating oil quench bath envelopes powders instantly, providing passivated surface film.
- This unique capability was adapted for making pure rare earth (RE) and RE-RE alloys as spherical powders in the right size for magnetic refrigeration.

Atomization mode selection for control of spherical powder size

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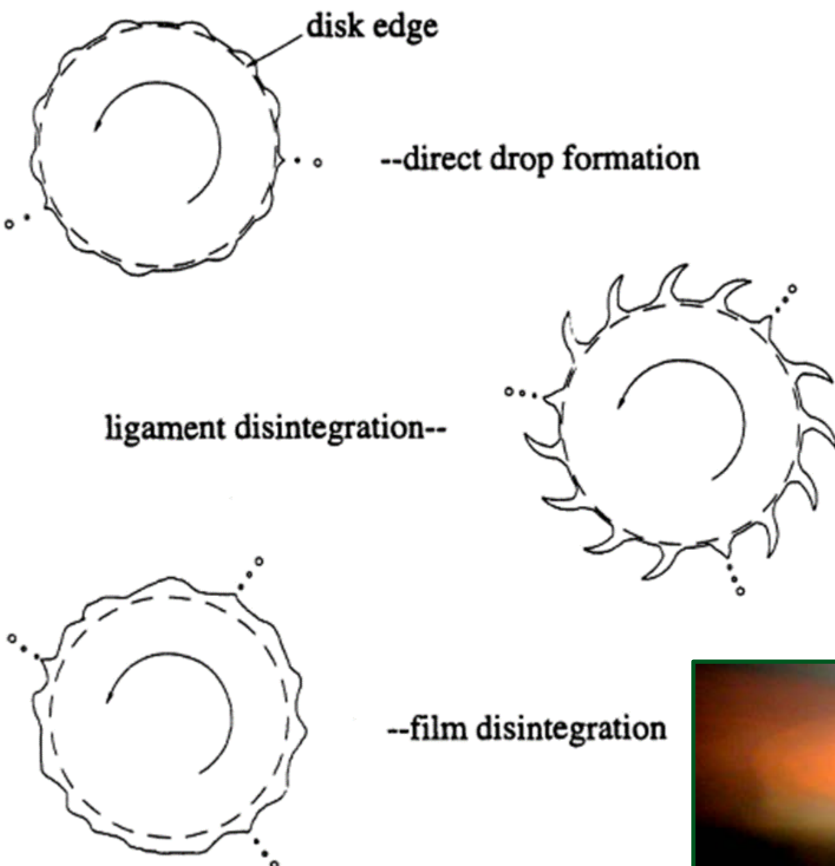

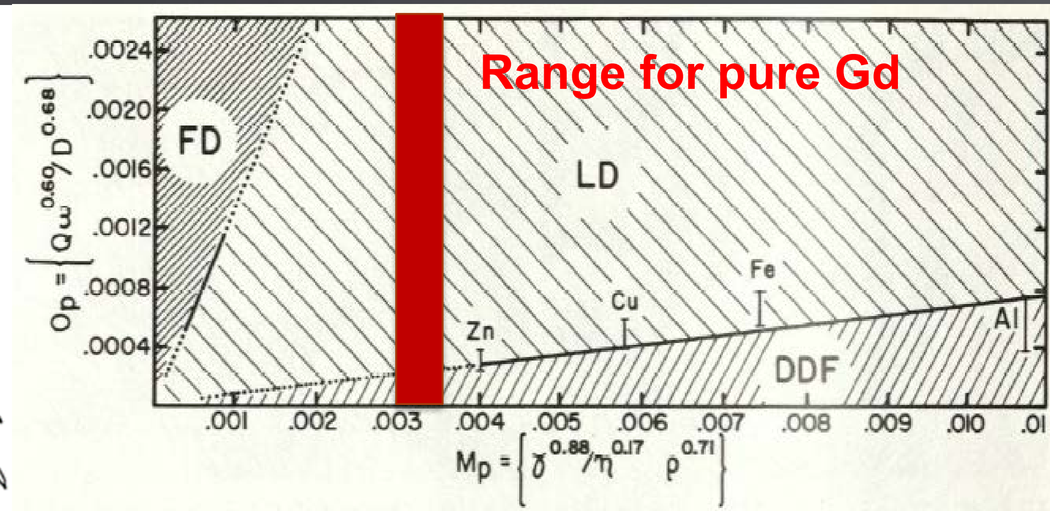

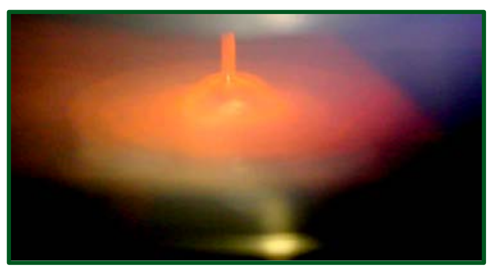

Champagne and Angers predicted critical liquid flow rates for transitions between 3 modes of centrifugal atomization: lowest flow rate/narrowest size distribution.

Ligament or direct drop disintegration mode preferred.

Target = 200µm

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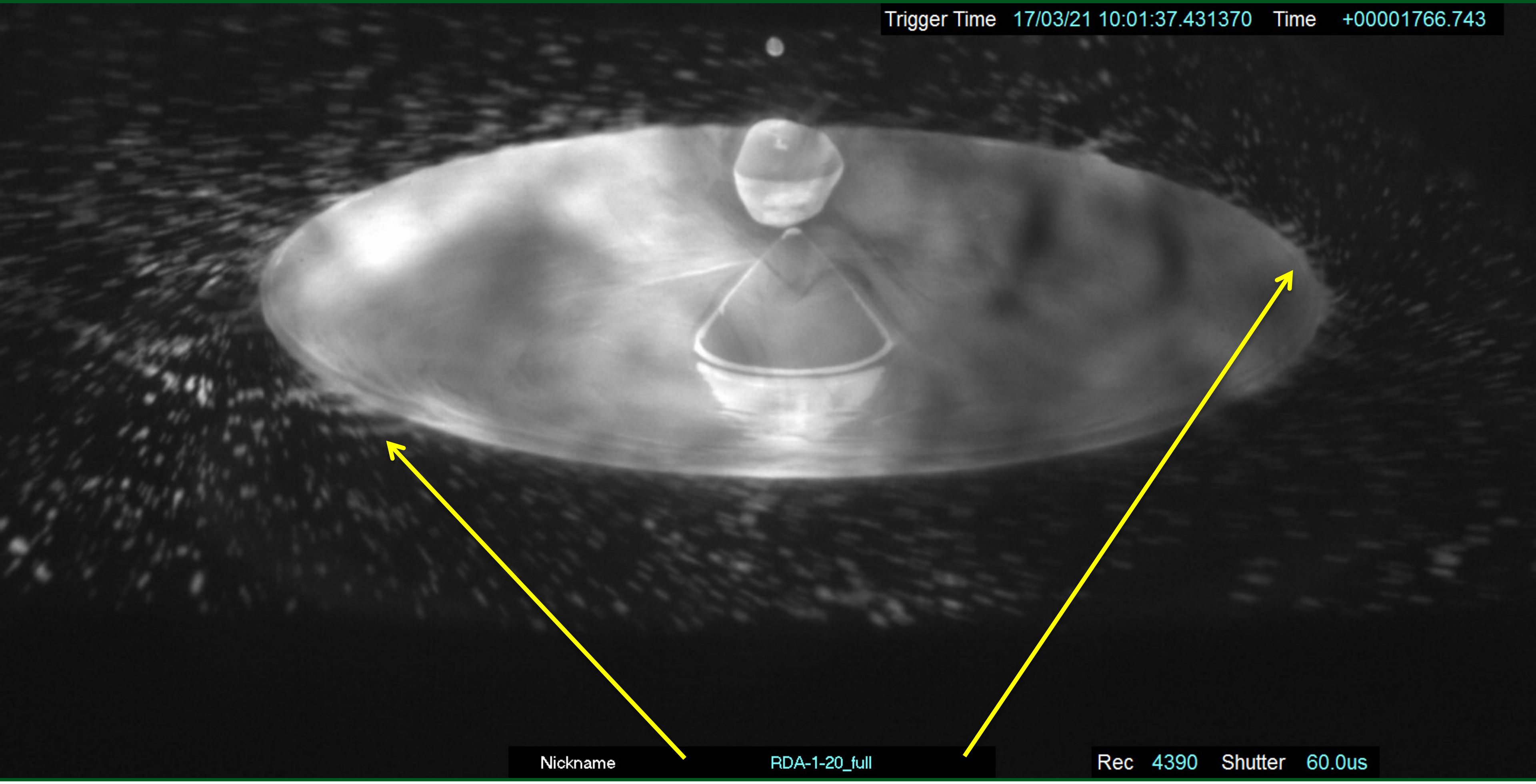
$Gd_{0.16}Ho_{0.84}$ RDA-1-20 Image of Atomization Process



Evidence for ligament/direct drop "mixed mode" disintegration from HS still frame.

Accomplishment (FY17): RDA Production of magnetocaloric powders (as of 4/2017)

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Layer	Material	Mass of magnetic material/layer (grams)	2.5x	Ames Produced (grams)	# Lots	Powder Average/ Ingot T_{Curie} (K)	Powder Average/ Ingot $M_{sat.}$ (emu/g)
1	Gd	268	670	490	3	289/293	256/267
2	$Gd_{0.91}Y_{0.09}$	258	645	512/605	4	274/274	247/228
3	$Gd_{0.3}Tb_{0.7}$	235	588	759	2	250/253	267/254
4	$Gd_{0.69}Er_{0.31}$	202	505		2	---/230	---/264
5	$Gd_{0.32}Dy_{0.68}$	172	430		2	---/214	---/279
6	$Gd_{0.15}Dy_{0.85}$	139	348	Pending Sieving	2	---/192	---/283
7	$Gd_{0.27}Ho_{0.73}$	100	250	350	1	172/173	306/290
8	$Gd_{0.16}Ho_{0.84}$	57	143	286	1	154/153	303/297

- ***Table reveals that magnetic properties of atomized powders have been consistent with bulk ingot measurements, indicating retained high purity.***
- ***Unexpected shift in mushy fluid (L+S) behavior of alloy on rotating disk changed predicted atomization results that required extra experimental effort/time.***

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Accomplishment (FY17): AMES measured key characteristics for 8 materials required for our models

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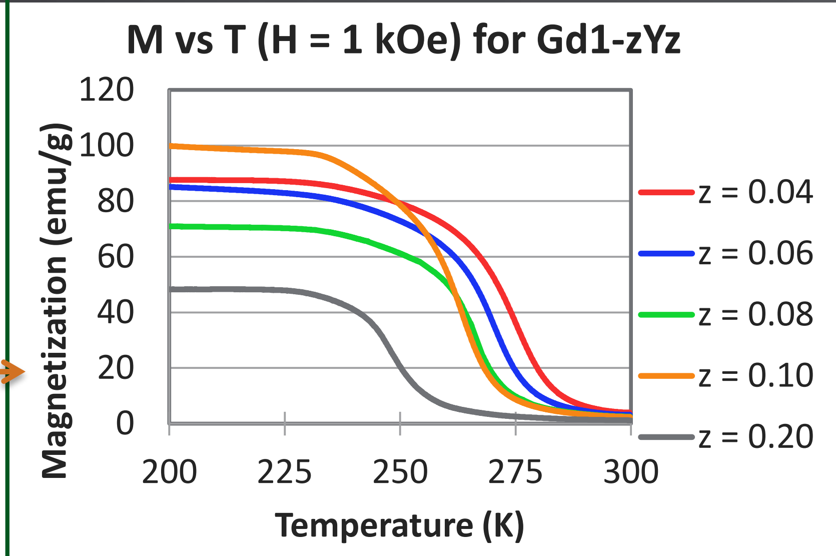
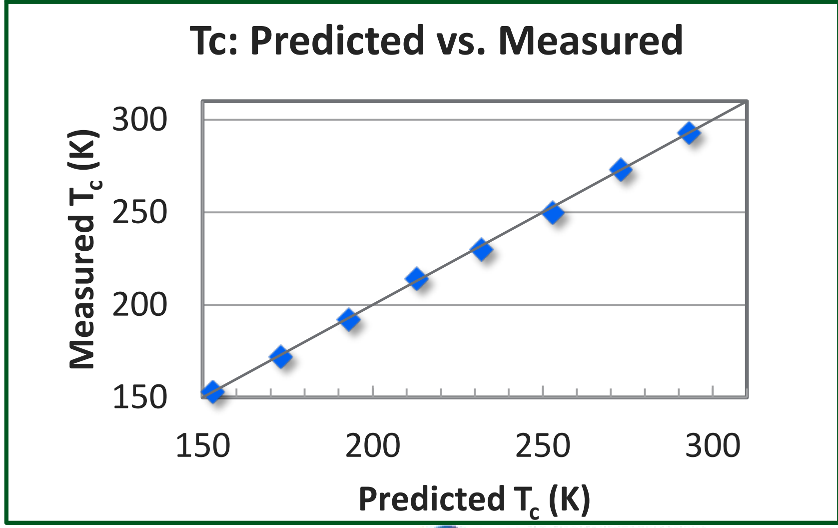
- Each material is fully characterized
  - T_c
    - M vs T, $\mu_0 H = 0.1$ T
    - Some alloys needed adjustment
      - $Gd_{0.90}Y_{0.10} \rightarrow Gd_{0.91}Y_{0.09}$
      - $Gd_{0.69}Er_{0.31} \rightarrow Gd_{0.67}Er_{0.33}$
    - mm-sized needle shaped samples and powders
  - C_p (H, T)
    - Quantum Design heat-pulse calorimeter
    - Temperature span: 319 K – 10 K
    - $\mu_0 H = 0.3$ T, 3.3 T, 6.6 T
    - mm-sized needle shaped samples
  - Magnetic Moment (M_s)
    - M vs H, T = 2 K, $\mu_0 H = 9$ T
    - mm-sized needle shaped samples and powders





Accomplishment (FY17): Characterization Magnetic Moment (AMES)

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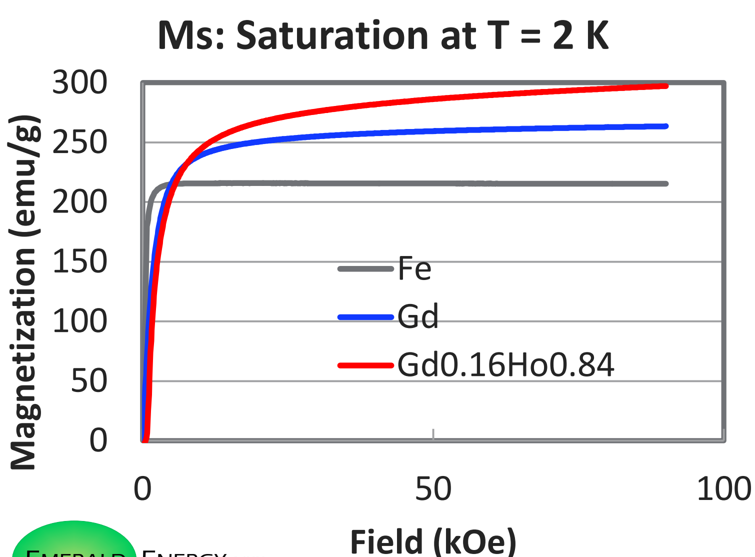
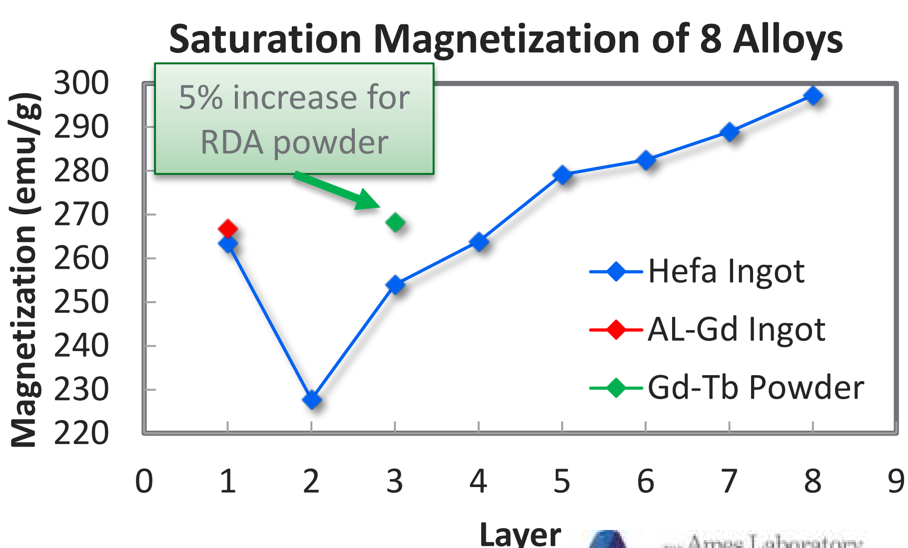
- Saturation Magnetization
  - M_s measured at T = 2 K, $\mu_0 H$ = 9 T
  - M_s values plug into AMRR performance model
  - RDA powders also being measured for comparison

Layer	Chemistry
1	Gd
2	$Gd_{0.9}Y_{0.1}$
3	$Gd_{0.3}Tb_{0.7}$
4	$Gd_{0.67}Er_{0.33}$
5	$Gd_{0.32}Dy_{0.68}$
6	$Gd_{0.15}Dy_{0.85}$
7	$Gd_{0.27}Ho_{0.73}$
8	$Gd_{0.16}Ho_{0.84}$





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