Thermomechanical Cycling of Thin Liner High Fiber Fraction Cryogenic Pressure Vessels Rapidly Refueled by LH_2 pump to 700 bar

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"This presentation does not contain any proprietary, confidential, or otherwise restricted information"

Overview

Timeline and Budget

- Start date: January 2014
- End date: December 2016
- Total project budget: $5.45M
- Total recipient share: $1.5M
- Total federal share: $3.95M
- Total DOE funds spent: $1.8M*

*As of 3/31/15

Funded jointly by Technology Validation, Storage, and Delivery

Barriers

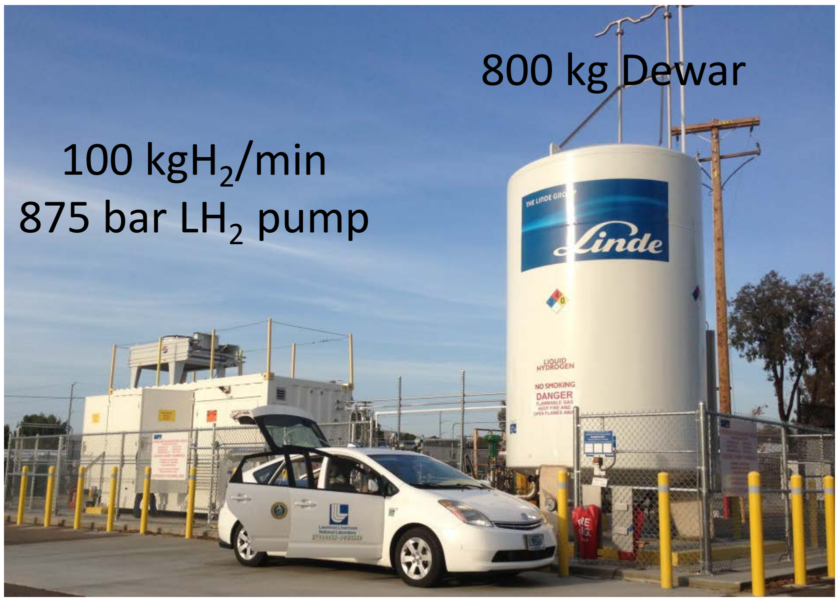
- A. System Weight & Volume
- D. Durability/Operability
- N. Hydrogen Venting

Partners

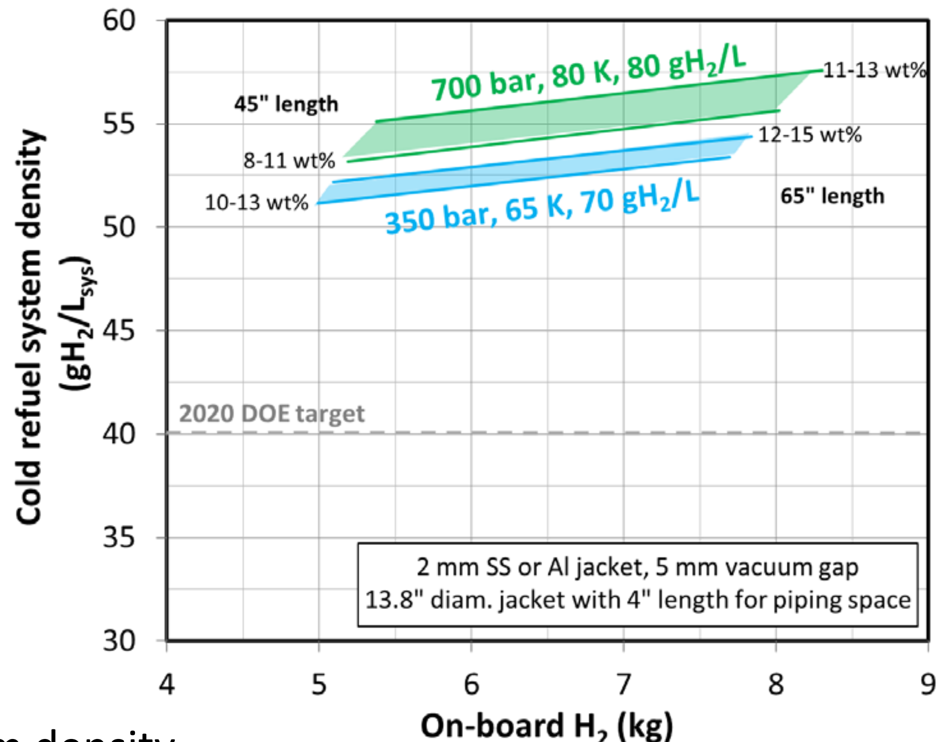
- Linde: LH_2 pump operation & maintenance, LH_2 delivery (~50 tankers)
- BMW: 350 bar cryogenic H_2 testing, system geometry, automaker perspectives
- Spencer Composites: design & build of 6 thin liner cryogenic prototype vessels

Cryogenic H_2 offers rapidly refueled onboard storage with volume, capacity, & safety advantages that outweigh technical challenges

- High density (cold) H_2 allows minimum vessel volume, mass, & cost with rapid refueling
- Larger capacities improve cryogenic valve/vacuum jacket cost, mass, & volume per kg of H_2
- Inert secondary containment, min burst energy @ max tension, on road safety factor of 5-10



7 minute 10 kgH_2 fill to 70 g/L (350 bar, 65 K)

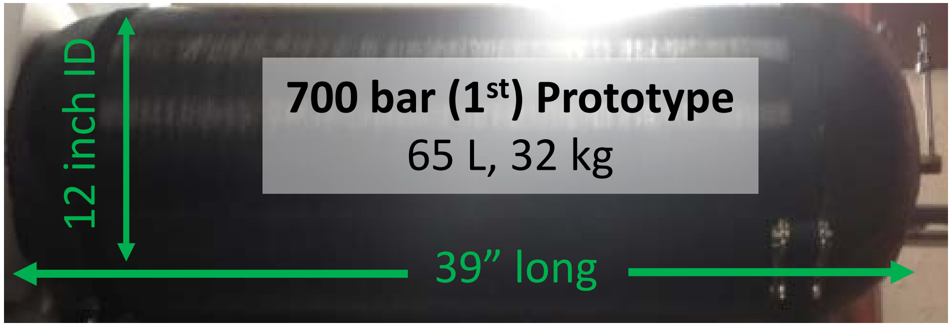


- *Compact* vacuum jacket necessary for system density
- Thermal *isolation* (parking) vs. suspension *strength* (acceleration)
- Temperature *variations* alter material properties, density, dormancy, H_2 burst energy

We will demonstrate a 5 kg H_2 system at 700 bar with 9+ wt% & 50 g/L

Volumetric efficiency improves system tradeoffs (pressure, dormancy, capacity & cost). Our objective is to explore thermomechanical limits of 12 inch vessels designed specifically for cryogenic H_2 storage

9 mm Al liner
Inner volume = 163 L
Outer volume = 233 L
163 L / 233 L = *70 % volumetric efficiency*



1.8 mm non-Al liner
Inner volume = 65 L
Outer volume = 80.36 L
65 L / 80.36 L = *80.9 % volumetric efficiency*

Ultra Thin liner (1.3-1.5 mm): necessary for small diameters
Non-Al liner: liner, piping, and weld durability under cryogenic H_2 cycling
Maximum fiber fraction: minimum wall volume & thermal inertia

We are demonstrating 700 bar prototype cryogenic vessels with a minimum 80% volumetric efficiency

Approach : Test cryogenic H_2 durability of four (65 L) prototype vessels before building a 5 kg 700 bar CcH_2 system demonstrating 50 gH_2/L

Phase 1 (proof-of-concept)

- Install instrumentation to determine LH_2 pump power, outlet temperature, & boil-off
- Safety plan for cryogenic H_2 cycling facility rated for 5kg H_2 prototype vessels
- 1600 bar cryogenic (LN_2) strength test of initial prototype design (2.28 safety factor)
- Fabricate 700 bar 163 L system using commercial vessel to test insulation & supports

Phase 2 (durability)

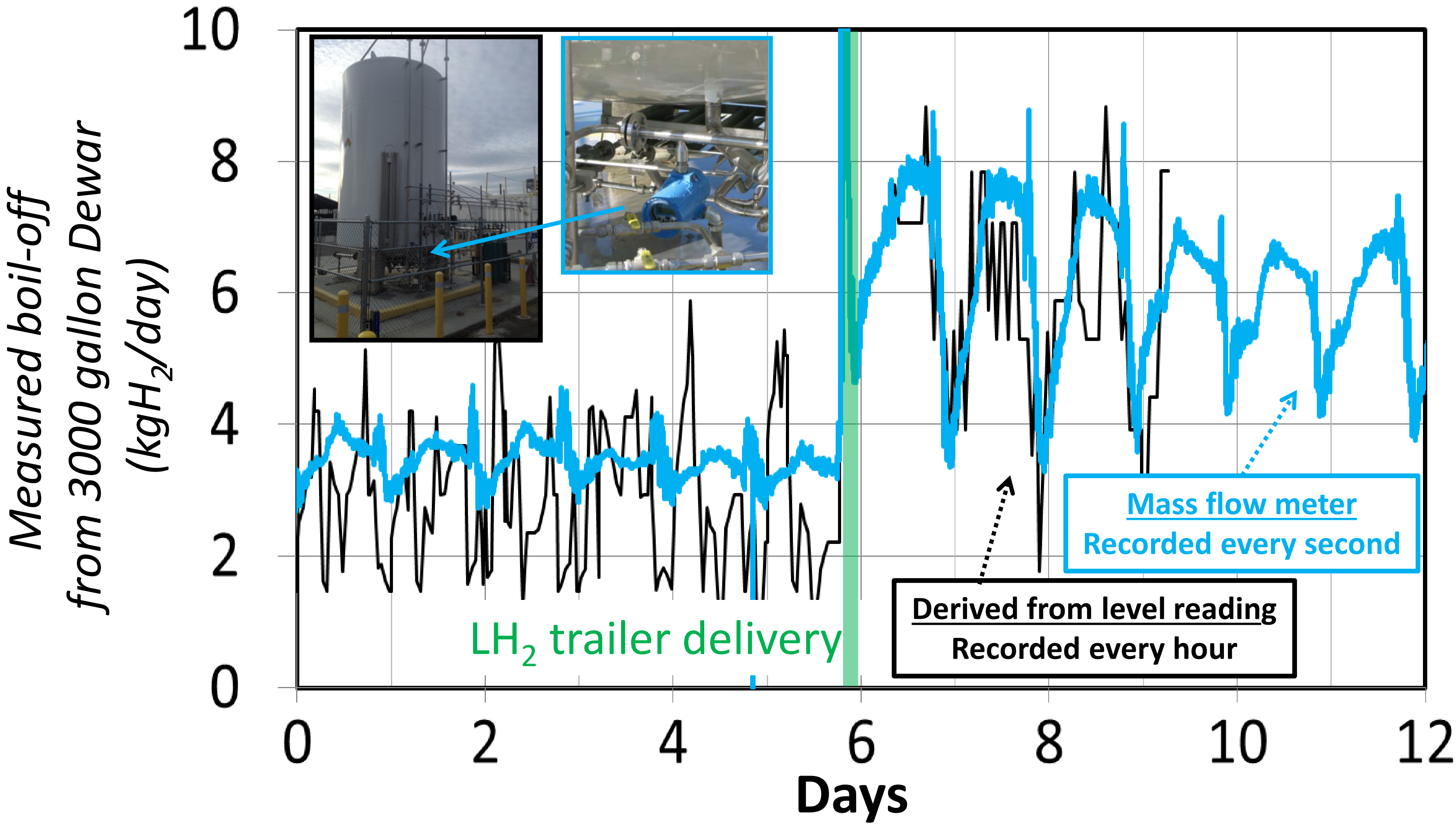
- Install containment for 1300 bar 160 Kelvin H_2 burst and 700 bar cycling to 300 Kelvin
- 1500 refuelings & cryogenic H_2 strength test (1.85 safety factor EOL) of two vessels
- Initial 700 bar characterization of LH_2 pump (peak density, kWh/kg, boil-off)

Phase 3 (demonstration)

- Aggressively cycle then strength test two higher performance vessels
- Select and install final vessel design in lightweight compact vacuum jacket
- Performance demonstration (volume, peak H_2 density, dormancy, vacuum stability)
- Compare for any LH_2 pump degradation after 6,000 refuelings to 700 bar

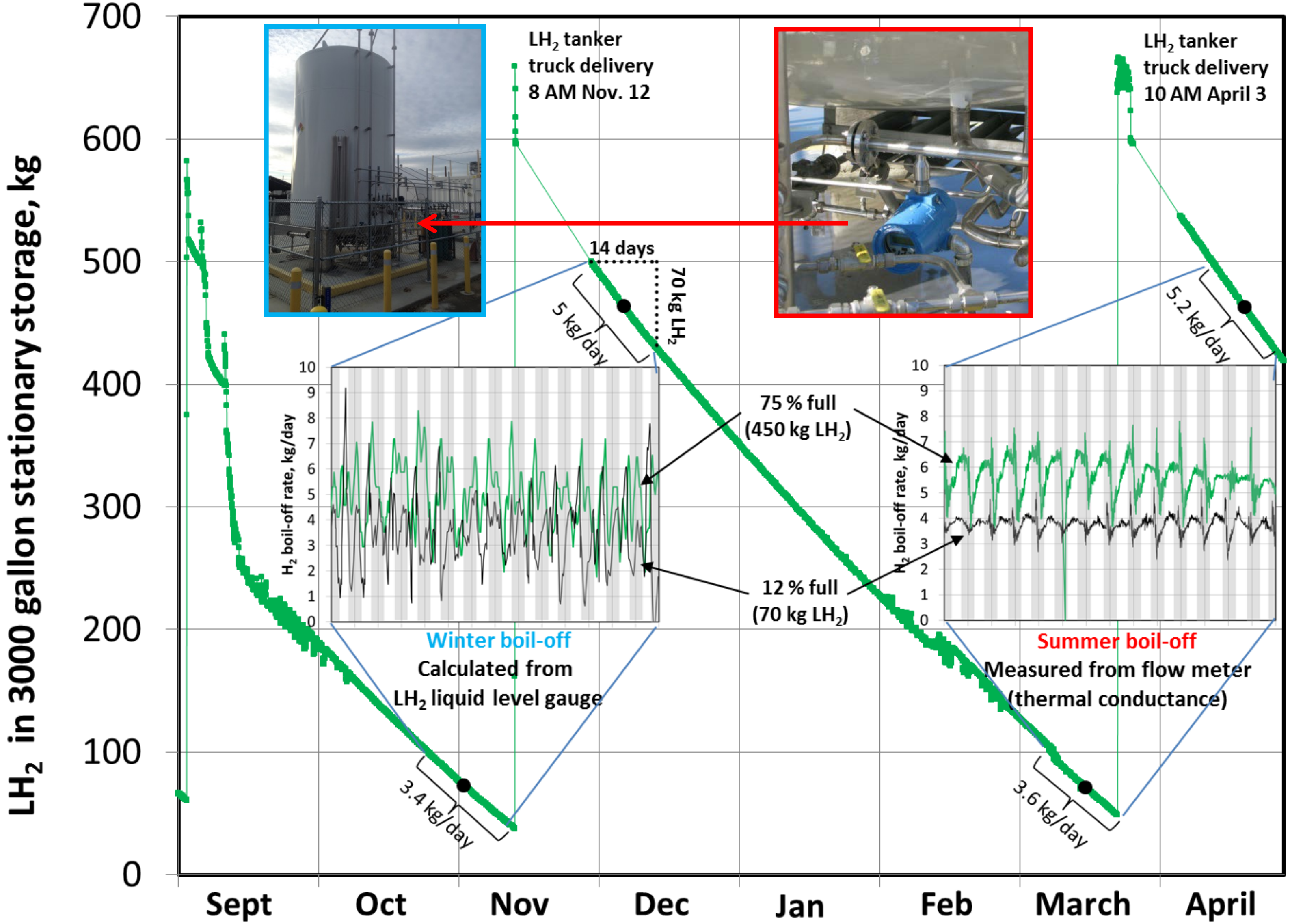
Phase 1 go/no-go successfully completed
Phase 2 go/no-go in winter 2015

Accomplishments : Boil-off flow meter installed on 3000 gallon Dewar



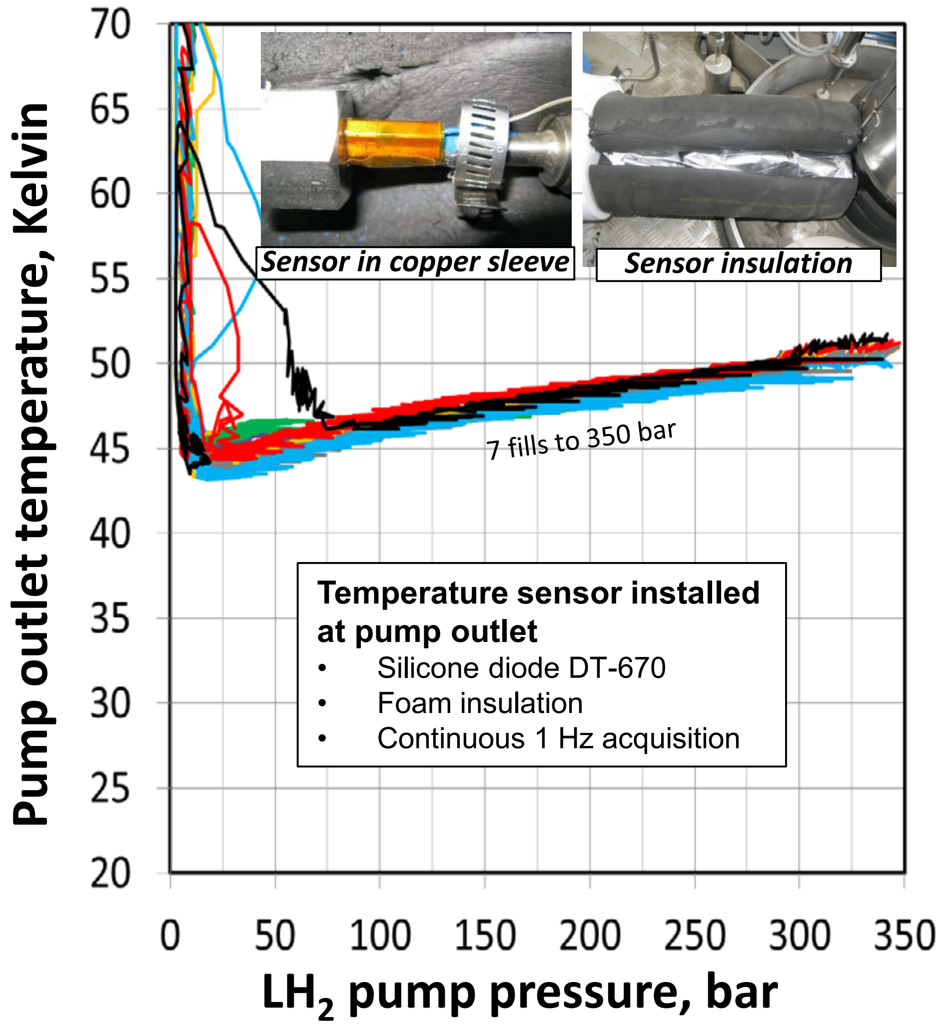
Directly measuring evaporation is more accurate than inferred evaporation from LH_2 level changes

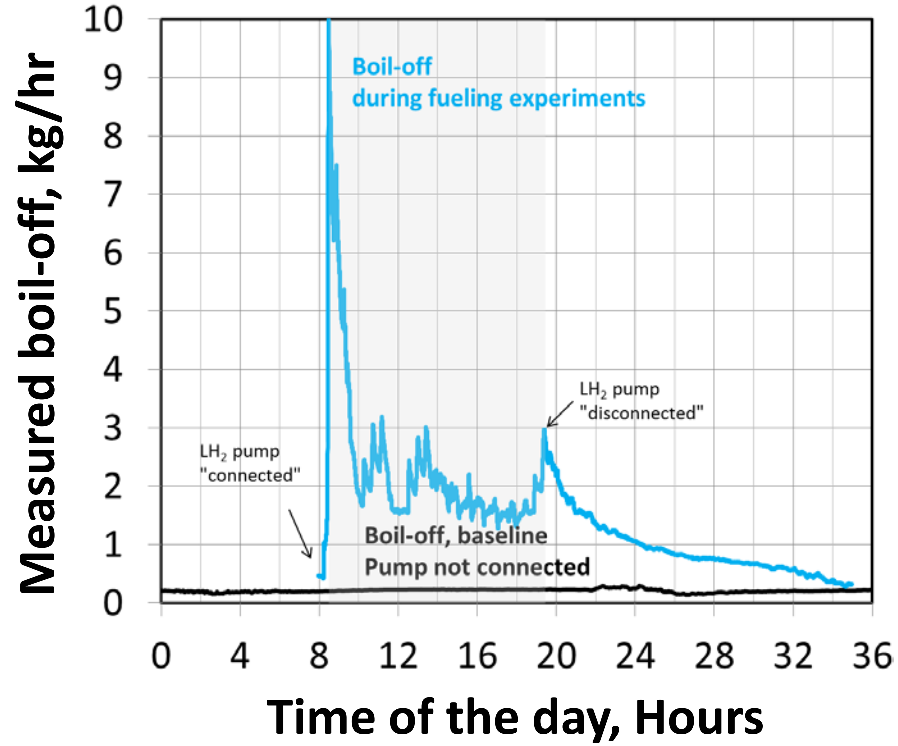
Accomplishments : Boil-off flow meter installed on 3000 gallon Dewar



LH$_2$ stationary storage fill level controls boil-off rates. Seasonal impacts are negligible

Accomplishments : LH_2 pump has been instrumented (outlet temperature, electricity usage) to evaluate performance degradation

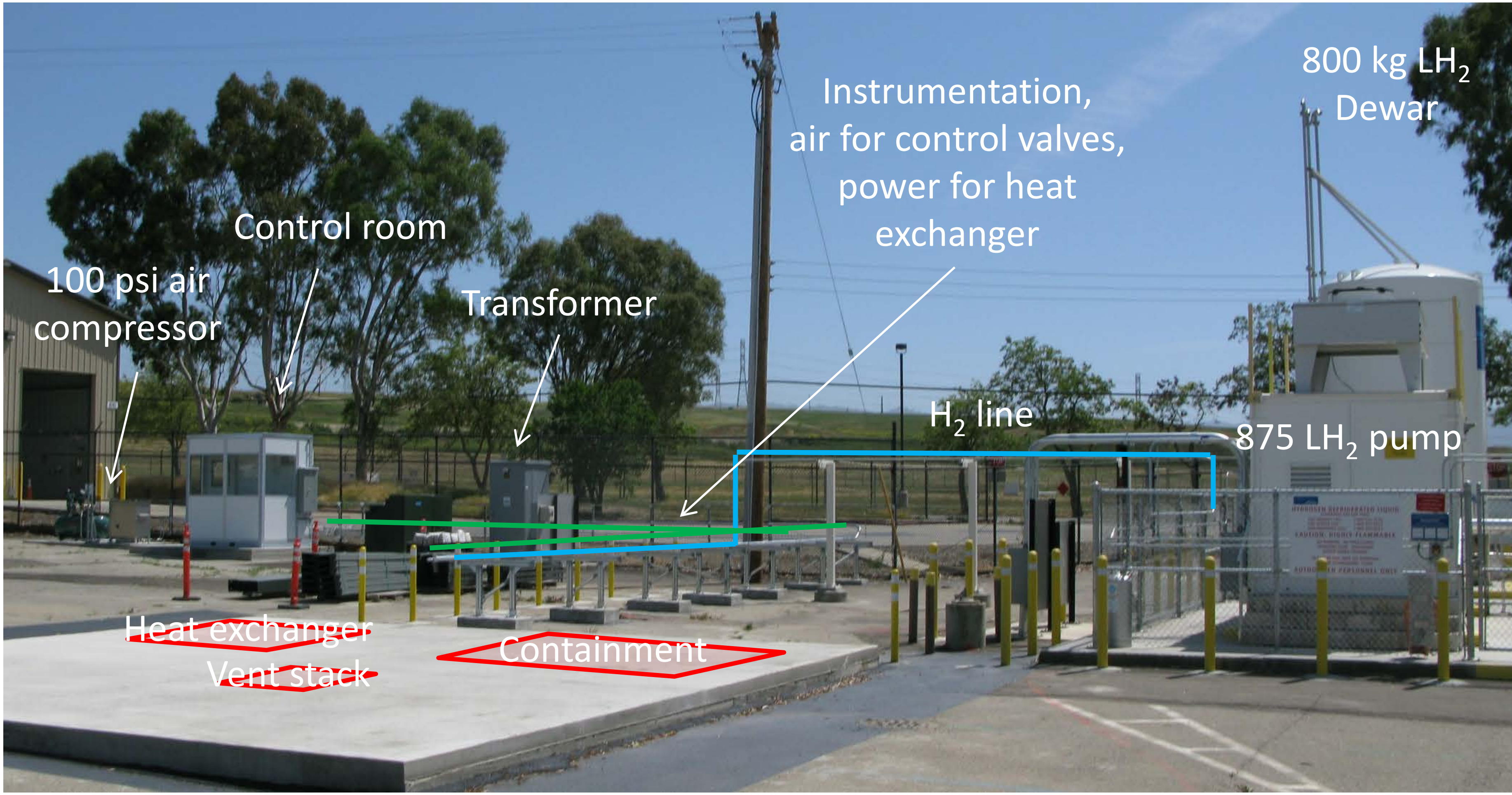




Power Analyzer
- 20 kHz sampling rate
- Memory limited (24 hours)

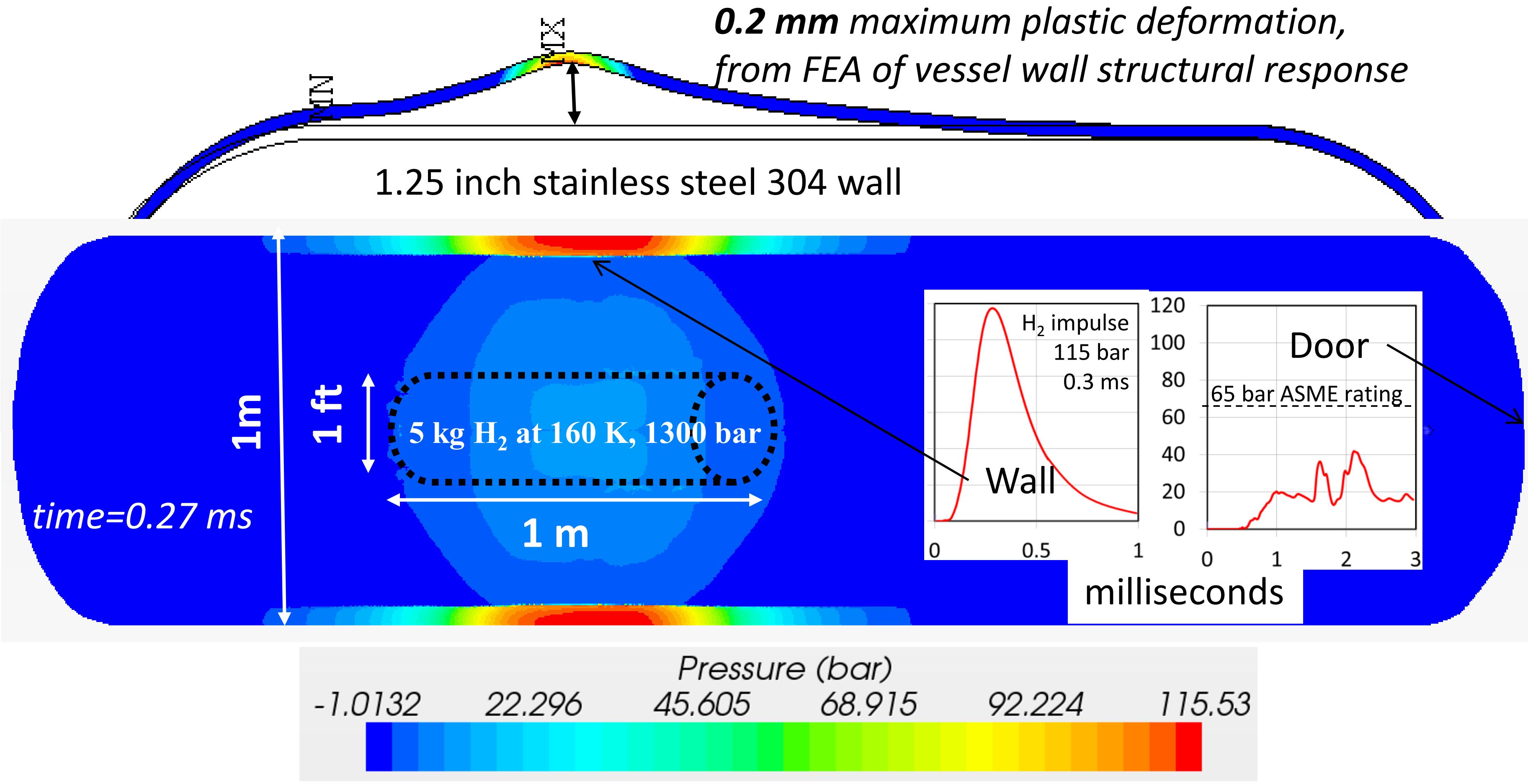
Boil-off, electricity consumption and pump outlet temperature will be measured over 6,000 H_2 refuelings (prototype cycling in phases 2 & 3)

Accomplishments: Site construction & control room for cryogenic H_2 cycling within 2.8 m^3, 65 bar containment using 875 bar LH_2 pump



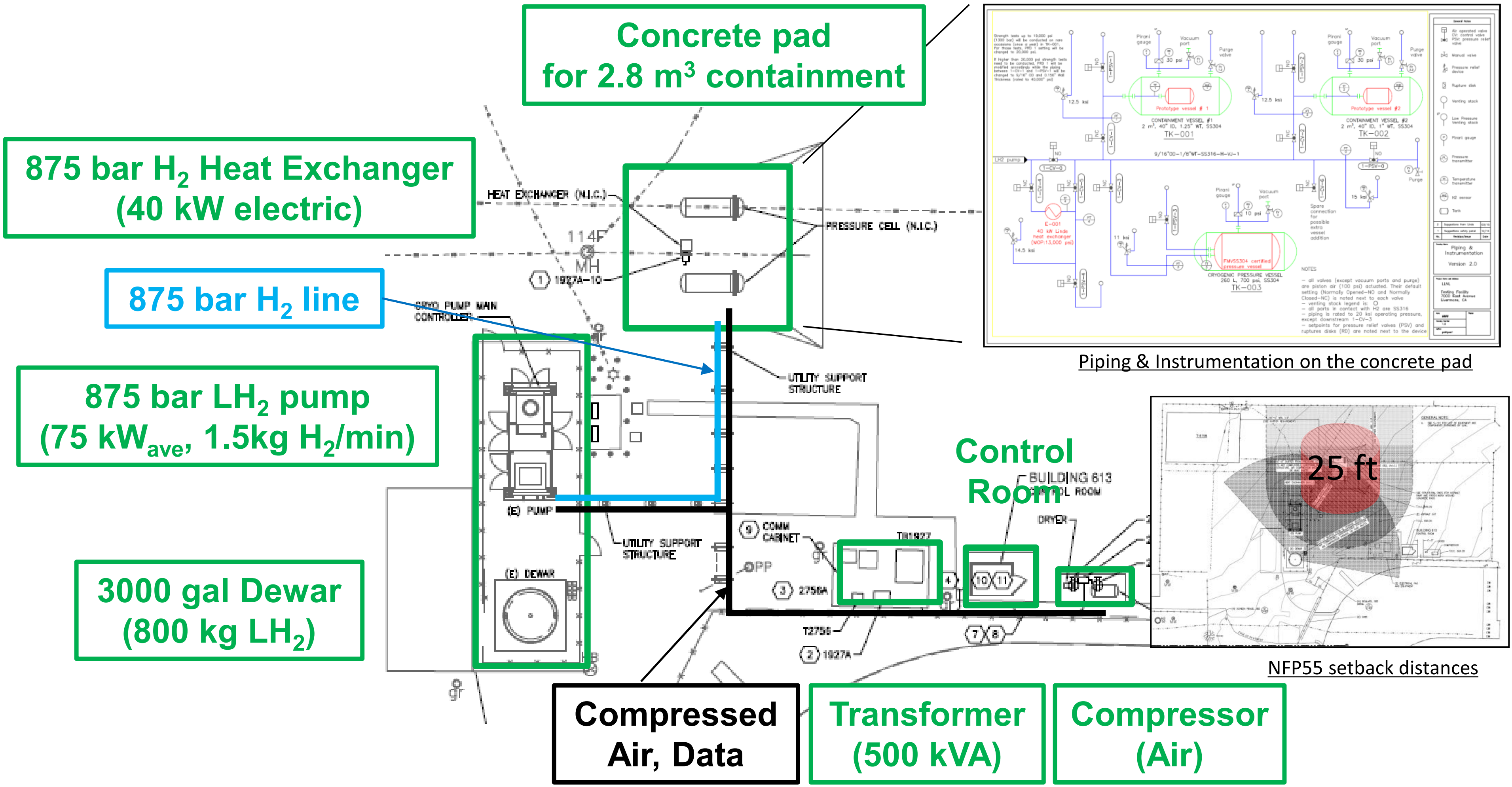
Civil site construction for test facility was completed in early 2015
Containment, LH_2 lines etc., and heat exchanger to follow in FY15

Accomplishments : LLNL safety approval of 65 bar containment required transient H_2 peak pressure & dynamic vessel wall loading analyses for 2.5-5 kgH_2 over the full pressure and temperature range



1300 bar 5 kg cryogenic H_2 burst is worst case scenario (P_{wall}=115 bar)
700 bar 2.5 kg H_2 burst during 300 K cycling is lower (P_{wall}< 100 bar)

LH_2 cycling facility with 1 m dia. containment & 40 kW heat exchanger for 2.5-5 kg 700 bar prototype vessel testing, remotely operated



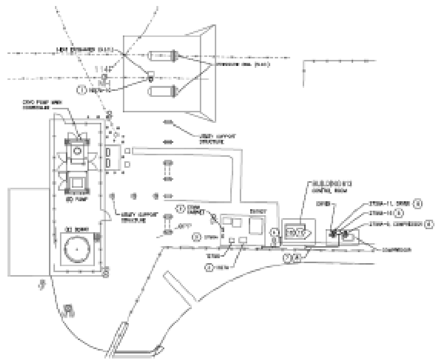
1 m diameter 65 bar containment, Class 1 Div. 1 power and controls, 25 ft. exclusion zone & remote operation are used to mitigate H_2 risk

Accomplishments: 100+ page DOE safety plan submitted for 800 kg LH_2 testing facility. Visit by DOE H_2 Safety Panel in August 2014

LLNL Proprietary – Do not distribute

LLNL Cryogenic and High Pressure Hydrogen Vessel Testing Facility

Safety Plan



Submission date: June 3, 2014

Authors: S. Aceves, G. Petitpas. V. Switzer

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

Comprehensive plan includes key aspects of safe operation

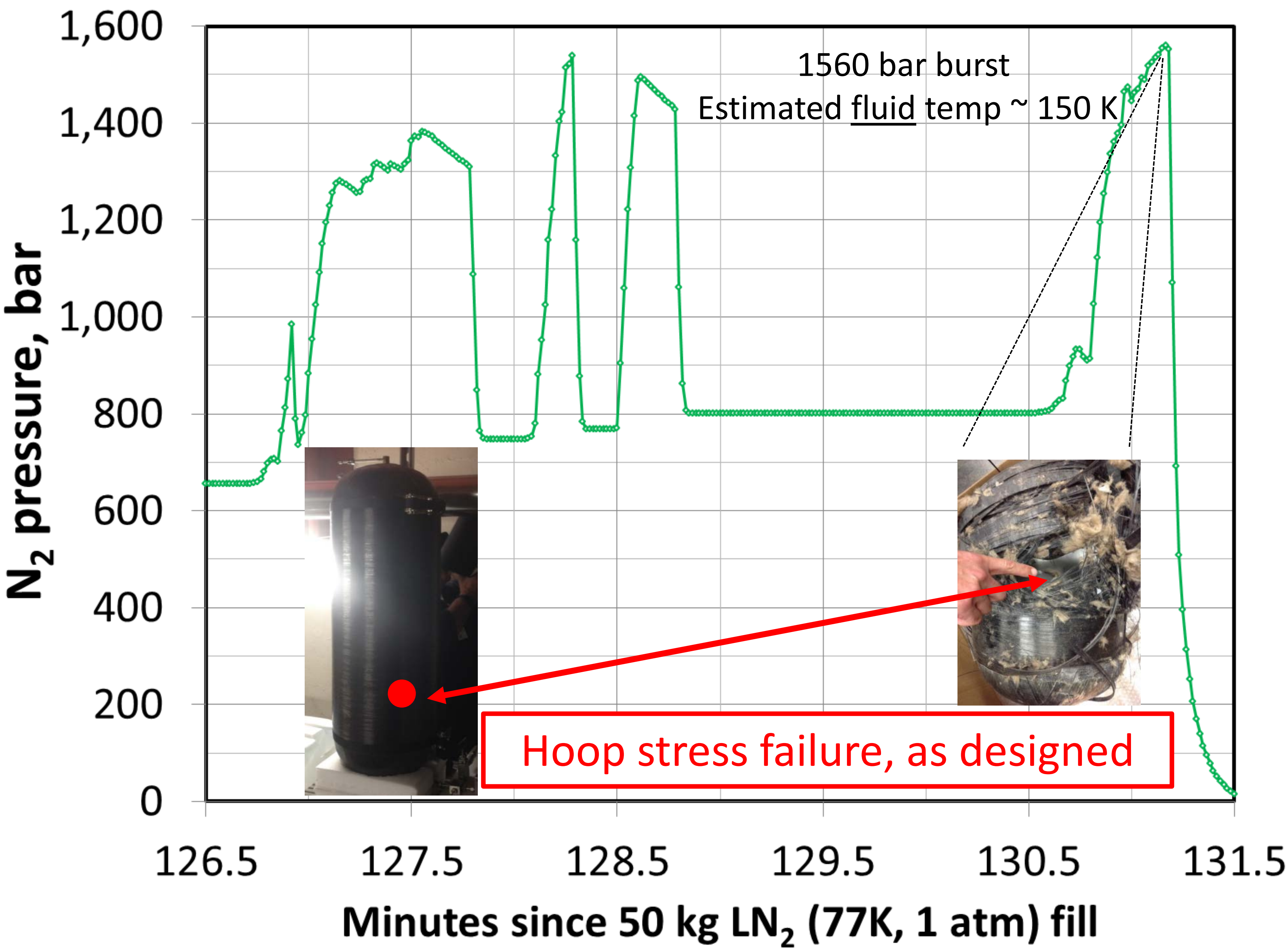
- Failure Mode and Effect Analysis
- Piping and Instrumentation Diagram
- Site layout
- Safety distances for H_2
- Design Calculations
- Components specifications

Safety plan was very well received by DOE hydrogen safety panel

- Requested authorization to use as an example of completeness and thoroughness

Internal LLNL safety plan for ASME 65 bar containment submitted and approved

Accomplishments : Cryogenic strength test of 65 L prototype with 81% volumetric efficiency burst at 1560 bar



Accomplishments : Cryogenic strength test of 65 L prototype

Phase 1 go/no-go :

Cryogenic N_2 strength test of full scale 700 bar prototype vessel

	vessel ID	Volume Efficiency	Inner Volume	Outer Volume	Vessel Wall	% of goal	Minimum Cryogenic Strength	% of goal
Target	12”	*80.0 %*	65 L	*81.25 L*	*16.25 L*	*100 %*	*1600 bar*	*100 %*
Result	12”	80.9 %	65 L	80.36 L	15.36 L	**105 %**	1560 bar*	**97.5 %**

*following 2 pressure spikes to 1500 bar

Phase 1 go/no-go completed in Feb 2015

Responses to Reviewers' comments

Project not reviewed last year

Collaborations with Industry Leaders

- **Linde:** Very cooperative, sharing detailed information throughout pump development, construction, and installation. Interpreting and sharing data from multiple pumps. LH_2 handler's perspective on testing facility design. Will provide 50 LH_2 tanker deliveries over project duration.
- **BMW:** Automotive LH_2 experience. Extensive 350 bar system design and subscale cycling. Safety validation of commercial vessels. Guidance on storage geometry, use cases, cycling design. Monthly phone meetings discussing thin liner potential, vacuum stability, LH_2 pump operation and performance comparisons.
- **Spencer Composites (Sacramento, CA):** Long expertise in custom composite vessel development. Very close collaboration on component testing, first 700 bar thin liner vessel design & build. Will build 6 custom prototypes during the project.

Remaining Challenges and Barriers for FY16 milestones

- ***Cycling efficiency and logistics***
  - **Challenge**: Warm cycles dominate calendar time (due to warmup time to room temperature) and increase LH_2 pump boil-off substantially
  - **Solutions** : Shift mix of warm cycles toward empty (instead of full), 7 day/week operation, approval for alternating cycling of 2 prototypes in one containment, coordinate LH_2 delivery to support operation with 2 test vessels
- ***Test facility***
  - **Challenge**: Peak H_2 density at 700 bar estimated at 80 g/L (4.2 kg H_2 per cycle), from 350 bar data using short 10 ft vacuum jacketed hose. 100 ft high pressure line could increase cryogenic H_2 fill temperature
  - **Solutions** : Consider purging line before cold fill and/or vacuum jacket high pressure H_2 line (challenging due to high pressure ratings)

Challenges will be better understood once cycling has begun. Cycling procedure and objectives can then be refined.

Proposed Future Work

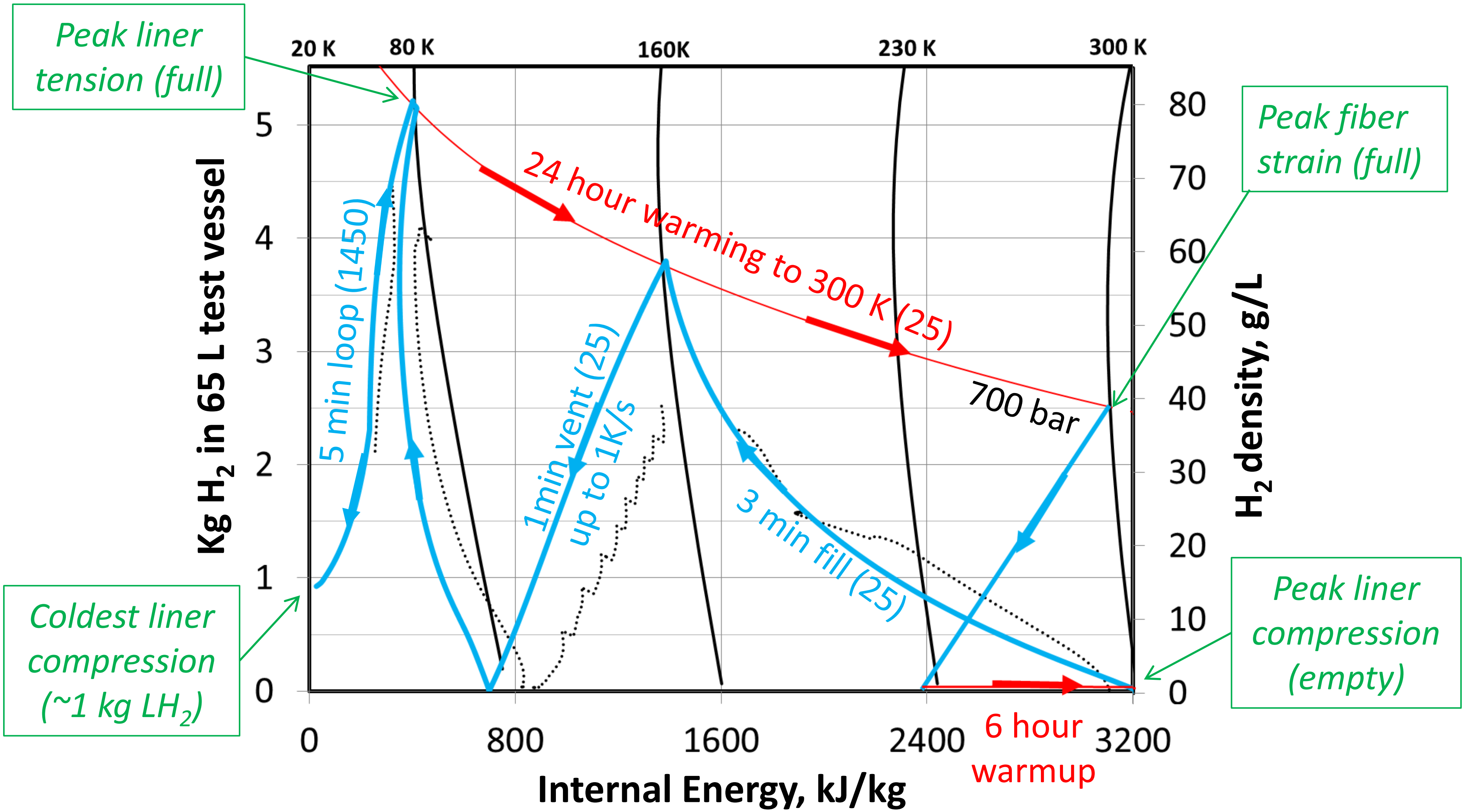
Future work includes:

- Installation of containment, vent stack, and heat exchanger
- Manufacture and cycle testing of 4 thin-lined full scale 700 bar vessels
- Test LH_2 pump degradation over 6,000 refuelings (~24 tonnes LH_2)

Date	Description
03 /2015 **Postponed**	Determine pump performance at 700 bar using 163 L conventional vessel (40 gH_2/L_{sys}, 6 wt% H_2) **Postponed**
07/2015	First operation of LH_2 pump to 700 bar with one 65 L prototype *Pursue LLNL safety approval for 2 test vessels operation*
10/2015 ***Go/No-Go***	Complete 1,500 thermomechanical cycles on one 65 L 700 bar prototype with > 80% volumetric efficiency. Pass 1300 bar H_2 cryogenic strength test.
01/2016	Complete 1,500 thermomechanical cycles on second 65 L 700 bar prototype. *May be done earlier if alternate cycling approved & 2 LH_2 deliveries/week.*
02/2016	Begin accelerated cycling of 3rd 65 L prototype vessel using heat exchanger

Second Go/No-Go (winter 2015): Successful cryogenic 1300 bar strength test of *at least one* prototype vessel after 1,500 thermomechanical cycles

Future Work : Cryogenic H_2 cycling will cover the full pressure & temperature range, emphasizing maximum thermomechanical stress



1500 pressure cycles using 7000 kg H_2 per vessel (12 LH_2 deliveries) with 50 excursions to 300 K (25 from 230 K, empty)

Future Work : cryogenic cycling and strength testing of 4 prototypes will be conducted in 2 phases to determine final demo vessel design

Design Risk

	1/2016		*6/2016*
Liner : *Conventional* (1.3 mm) **Resin :** Low viscosity **Stiffness :** *Minimum* **Weight :** ~ 27 kg **Volumetric Efficiency :** 85 % A	**1300 bar cryo strength test**	**Liner :** *Conventional* (1.3 mm) **Resin :** Low viscosity **Stiffness :** *Minimum* **Weight :** ~ 27 kg **Volumetric Efficiency :** 85 % A	**1300 bar cryo strength test**
Liner : *Alternate* (1.5 mm) **Resin :** Low viscosity **Stiffness :** *Maximum* **Weight :** ~ 34 kg **Volumetric Efficiency :** 80 % B		**Liner :** *Alternate* (1.5 mm) **Resin :** Low viscosity **Stiffness :** *Minimum* **Weight :** ~ 30 kg **Volumetric Efficiency :** 85 % C	

Temperature Variation and Non-Uniformity

Passive (external) Warming

1500 cryogenic fills
(25 from 300 K)

Active (internal) Warming

1100 cryogenic fills
(25 from 300 K)
(175 from 270 K)

400 warm fills
(200 from 20 K)
(200 from 80 K)

Goal: 50 days @ 700 bar (25 days <200 K)

2015: most aggressive and conservative designs tested early
2016: diversify heat transfer and temperature of pressure cycles

In the event the most aggressive design fails, we will then test an alternate (thicker) liner with conventional cryogenic epoxy

1/2016 — *6/2016*

Design Risk ↑

A (crossed out)
Liner : *Conventional* (1.3 mm)
Resin : Low viscosity
Stiffness : *Minimum*
Weight : ~ 27 kg
Volumetric Efficiency : 85 %

B
Liner : *Alternate* (1.5 mm)
Resin : Low viscosity
Stiffness : *Maximum*
Weight : ~ 34 kg
Volumetric Efficiency : 80 %

1300 bar cryo strength test

D
Liner : *Alternate* (1.5 mm)
Resin : Cryogenic epoxy
Stiffness : *Minimum*
Weight : ~ 32 kg
Volumetric Efficiency : 80 %

C
Liner : *Alternate* (1.5 mm)
Resin : Low viscosity
Stiffness : *Minimum*
Weight : ~ 30 kg
Volumetric Efficiency : 85 %

1300 bar cryo strength test

Temperature Variation and Non-Uniformity →

Passive (external) Warming

1500 cryogenic fills
(25 from 300 K)

Active (internal) Warming

1100 cryogenic fills
(25 from 300 K)
(175 from 270 K)

400 warm fills
(200 from 20 K)
(200 from 80 K)

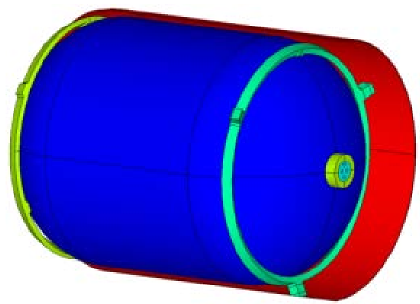
Goal: 50 days @ 700 bar (25 days <200 K)

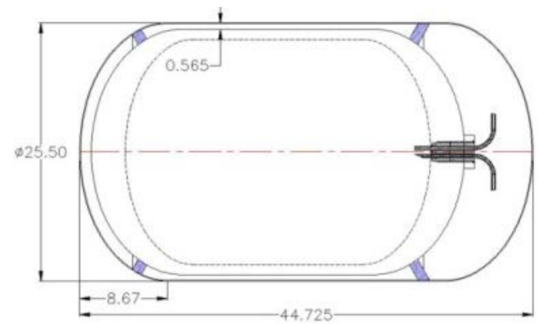
If all vessels succeed, 2 liner materials will be proven.
Otherwise, alternate liner will have been cycled using 2 different resins.

A 700 bar large (163 L, 23 inch diameter) commercial pressure vessel will be vacuum jacketed to test compact support & insulation designs

We plan to test compact supports and insulation for a minimum vacuum gap design using an existing 700 bar commercial vessel. The large capacity (163 L) and DOT rating for manned operation will enable precise heat transfer measurements for mechanism determination.

Long lead time components have been acquired but we are considering delaying this task until the phase 2 go/no milestone is successfully completed. This will still provide adequate time for results before the final demonstration system design is complete.





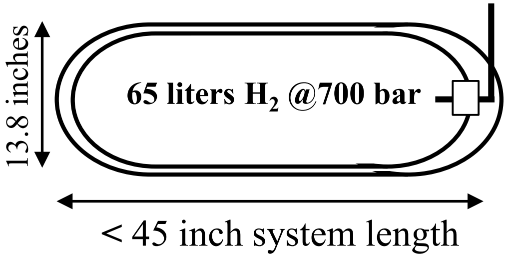
Once complete, this system will also be ideal for high resolution LH_2 pump performance characterization, particularly in the two-phase region with continuous weight measurements and external temperature control as future options.

This task was originally planned in phase 1 but was reduced in priority relative to safety plan and 1st prototype fabrication. We are now considering fabrication early in phase 3.

A 5 kg H_2 700 bar demonstration system will be built based on the best prototype vessel design & fully characterized in the LH_2 cycling facility

- Comprehensive refueling map (capacity vs. initial refuel condition) with 20-30 refuelings
- Determine system mass and external volume, with some allowance for instrumentation
- Continuous vessel and jacket temperature measurements
- Dormancy measurements and heat transfer estimates for 5 capacities and 3 temperatures
- Continuous vacuum monitoring over ~ 2 months

System volume < 100 L



Projected demo system specifications (65 L)	
Outer Diameter: < 14”	Weight (full): 40 to 60 kg
Length : < 45 “	Dormancy: 4-8 days at 90% full
Jacket volume : < 100 L	

Our projected system density is based on LH_2 pump refuel densities extrapolated from 350 bar to 700 bar (80 gH_2/L, 80 Kelvin)

Technology transfer activities: technology jointly developed with BMW and Spencer composites Corporation

- **BMW CRADA signed July 2014:** Includes $1M cost share

- **Two recent patents:**
  - Weisberg AH. **Methods for tape fabrication of continuous filament composite parts and articles of manufacture thereof**. United States Patent US 8545657 B2, November 2013.
  - Espinosa-Loza, F, Ross, TO, Switzer, V., Aceves, SM, Killingsworth, NJ, Ledesma-Orozco, E, **Threaded Insert for Compact Cryogenic Capable Pressure Vessels**, Granted March 12, 2015.

- **A provisional patent and two records of invention**

Summary: LLNL will demonstrate cryogenic durability of 12" thin liner vessels over 1500 refuelings, achieving 50 gH_2/L_{sys} & 9 wt% H_2

Relevance	LH_2 pump can rapidly and consistently refuel cryogenic H_2 onboard storage to 700 bar, with potential to exceed *weight* & *volume* DOE targets with substantial dormancy improvement for modest cost, with ideal scalability.
Approach	Determine cryogenic durability of 4 full scale 65 L thin liner 700 bar composite prototypes with maximum volumetric efficiency at 12" diameter. Demonstrate system volume, weight, dormancy and vacuum stability at 5 kg H_2 capacity.
FY15 Progress	Civil construction for cryogenic H_2 cycling facility completed Conducted safety analysis, obtained LLNL approval Manufactured 700 bar thin-lined vessel with 81% vol. efficiency Demonstrated 1560 bar cryogenic strength of prototype vessel
Future work	Complete H_2 cycling facility (1300 bar cryogenic burst containment) Cycle 4 vessels: 2 with external warming, 2 using heat exchanger Demonstrate minimum 1.85 EOL safety factor on up to 4 vessels Build & evaluate performance of 5 kg H_2 demo system Measure pump degradation over life of project