

NanoSonic, Inc.

**2019 DOE Hydrogen and Fuel Cells Program
Annual Merit Review and Peer Evaluation Meeting (AMR)**

PI: Dr. Jennifer Lalli

5/1/2019

**Cryogenically Flexible, Low Permeability
H₂ Delivery Hose**



Overview

Timeline:

- **Project Start Date:**
7/31/2017
- **Project End Date:**
7/30/2019

Budget:

- **FY13 DOE Ph I Funding:**
\$150,000
- **FY14 DOE Ph II Funding:**
\$1,000,000
- **FY17 DOE Ph IIB Funding:**
- **Total DOE Project Value:**
\$2,150,000

Barriers:

- **Reliability and Cost of Gaseous H₂ Compression**
- **Reliability and Cost of Liquid H₂ Pumping**
- **Eliminate H₂ Embrittlement, Increase Durability**
- **Lack of Fittings for New High Pressure Hoses**

Partners:

- **CSA Group**
- **PNNL**
- **NREL**
- **Techsburg Machining**
- **Cardinal Rubber & Seal**
- **LifeGuard Technologies**
- **Shell, Tatsuno, WEH and Air Liquide**
- **Giles County Government**

Relevance:

Develop a H₂ Hose for Fuel Cell Vehicles

Objectives:

- Flexible: H₂ hose to enable affordable delivery
- Rugged: -50 °C and 875 bar for H70 service
- Reliable and safe: 70 fills/day, > 2 years



Impact in April 2017 – April 2018:

- Produced: Low T_g, Low H₂ Permeability, Fiber Reinforced Hose with High Pressure Fittings
- Demonstrated: Hydrostatic Burst Strength > 36,000 psi for H35 Service
- Goal: Metal Free Hose Design and Custom Fitting to Enable Long-term H70 Service

Approach:

Project Phases and Selected Milestones



Evaluate FY17 Q4
critical performance metrics, fittings,
partners for deployable design



Qualify FY18 Q3-Q4
H70 hose with OEM's dispenser /
nozzle to assess service life (~2 years)



Test FY18 Q1-Q2
hose/fittings with H₂ via TTS (NS),
robotic fill (NREL), and DMA /
tribology (PNNL)

Deploy FY19 Q2
H70/H35 hoses at
H₂ stations



Critical Criteria

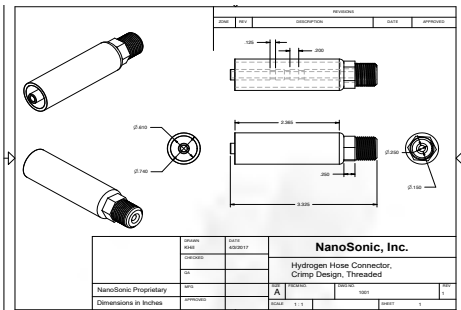
- Surpass 3500 Bar hydrostatic burst strength (> 50,763 psi) held for 1 min
- Survive 875 Bar pressure cycle at (50,000x at -50F and 50,000x at 85F)
- No contaminant leaching, Competitive cost, mechanical durability, and environmental lifetime

Phase IIB Commercialization Challenge

Lack of Commercial Fittings for New Hoses

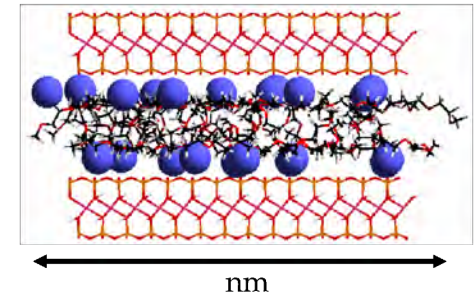
1. Design New Fittings

- For current cores
- Address leakage
- Custom OD and ID



2. Design New Cores

- For new fittings
- Advanced materials
- Custom ID



PRESSURE RATING

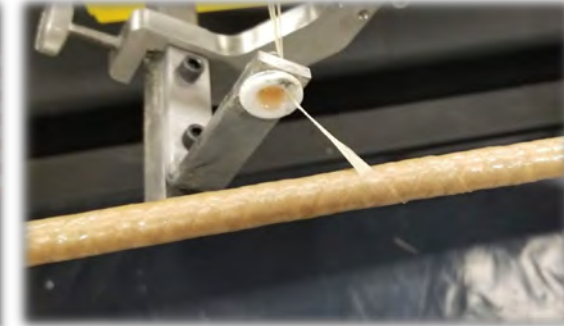
Hose Assembly
(hose + fitting)

4:1 safety factor

Working Pressure (1)
Burst Pressure (4x)



Technical Approach to Flexible Metal-Free H₂ Hose *Filament Winding*



**2 (3m) hoses per run
16 hoses per day**



Tested at RTP at CSA Group per Hydrostatic Strength (section 2.4) of ANSI/CSA HGV 4.2-2013 *Standard for hoses for compressed hydrogen fuel stations, dispensers and vehicle fuel systems*. Requires a 1 min hold without burst or visible loss of fluid at a hydrostatic pressure of four (4) times the manufacturers specified maximum allowable working pressure (MAWP). Up to a 10,000 PSI MAWP hose assembly.

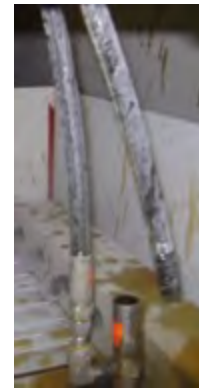
Demonstrated Hoses with Varied Fibers with Hydrostatic Burst Strengths > 36,000 psi

Major Accomplishment in Filament Wound Hose

Pressure Cycling: Leak before Burst ~52,000x

Pressure Cycle Test (section 2.17) of ANSI/CSA HGV 4.2-2013

- 50,000 cycles at 12,000 psi (827 Bar) at -40°C (-40°F) and
- 50,000 cycles at 12,000 psi (827 Bar) at 85°C (185 °F)



Down-selected filament wound composite survived:

- 50,000 cycles at 12,000 psi (827 Bar) at -40 °C and
- 1,988 cycles at 12,000 psi (827 Bar) at 85 °C

**NanoSonic Hose Survived 51,988 cycles at 12,000 psi over -40 °C to 85 °C
prior to leak rather than burst**

Accomplishments with Partners

Cardinal Rubber & Seal Swaged Fittings



Test Results

High Pressure Test Results

Customer:	NanoSonic Inc	Mfg. Part #:	
Part Number:	Test Hose	Description:	
Order Quantity:	1	Measured Intervals:	1
Date:	4/11/2017	Time:	10:48:17 AM
Operator:	SF	Test Pressure:	18000
Test Time:	1	Minimum Pressure:	14000

	1	2	3	4	5	6	7	8
1	19463	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0	0
3	0	0	0	0	0	0	0	0
4	0	0	0	0	0	0	0	0
5	0	0	0	0	0	0	0	0
6	0	0	0	0	0	0	0	0
7	0	0	0	0	0	0	0	0
8	0	0	0	0	0	0	0	0
9	0	0	0	0	0	0	0	0
10	0	0	0	0	0	0	0	0
11	0	0	0	0	0	0	0	0
12	0	0	0	0	0	0	0	0
13	0	0	0	0	0	0	0	0
14	0	0	0	0	0	0	0	0
15	0	0	0	0	0	0	0	0
16	0	0	0	0	0	0	0	0
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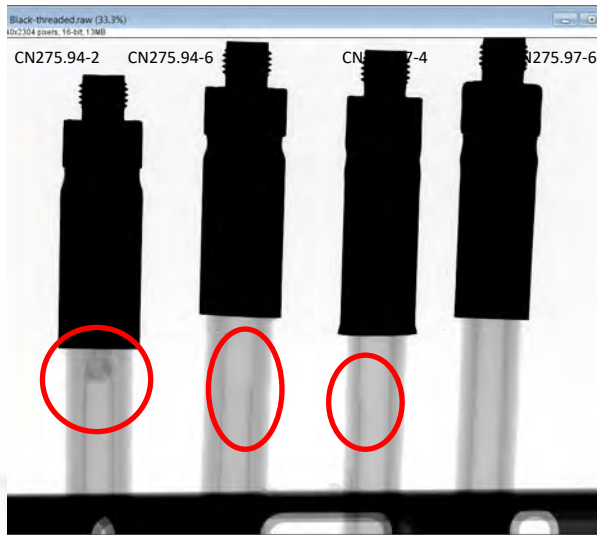
Fittings Swaged at Cardinal Passed High Pressure Testing (max is 20,000 psi)

Accomplishments in New Fittings

X-ray analysis – core defect characterization



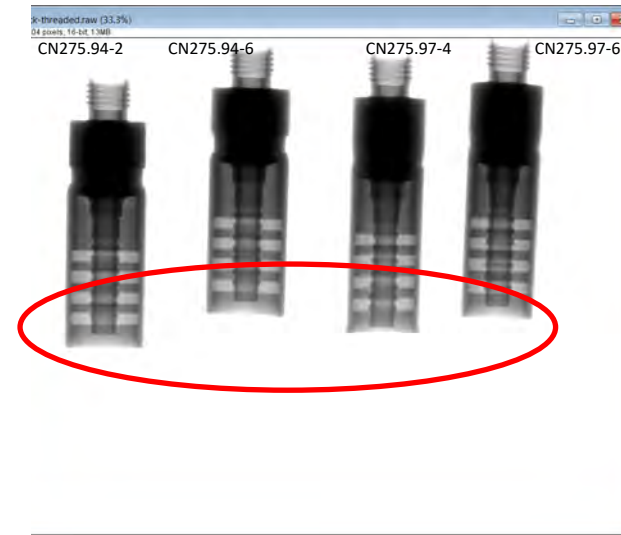
X-ray analysis at PNNL for defect characterization



Core defects



Socket symmetry



Stem depth

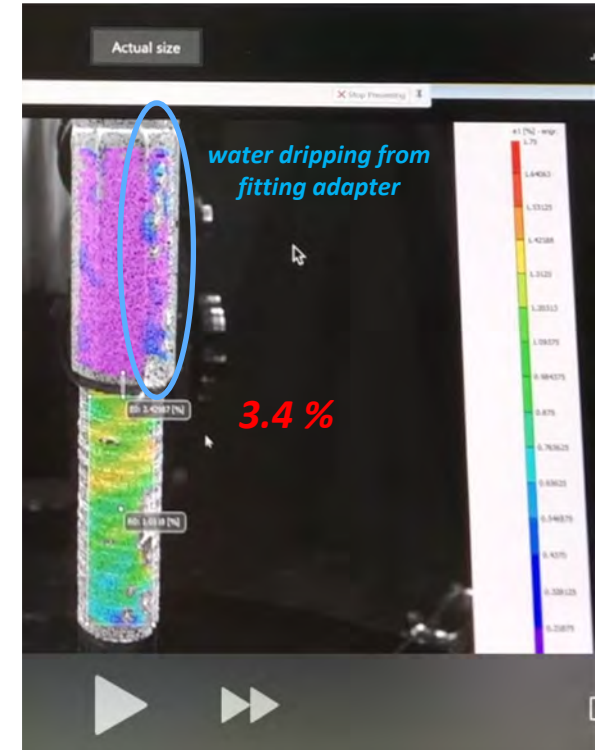
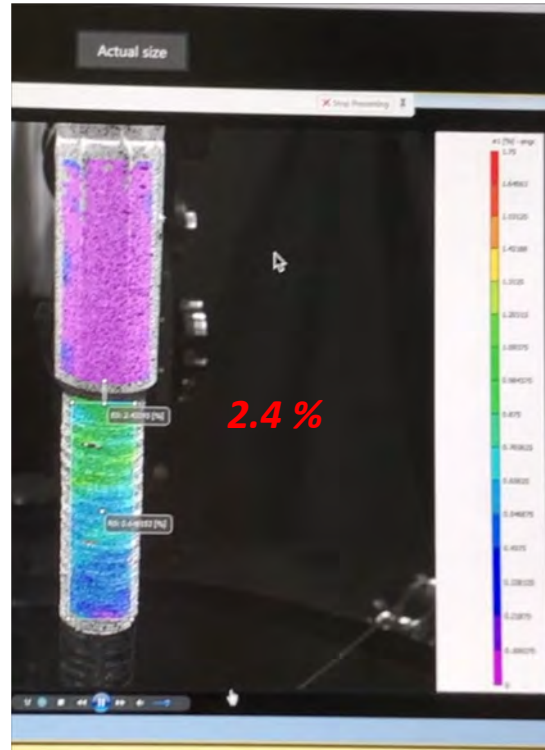
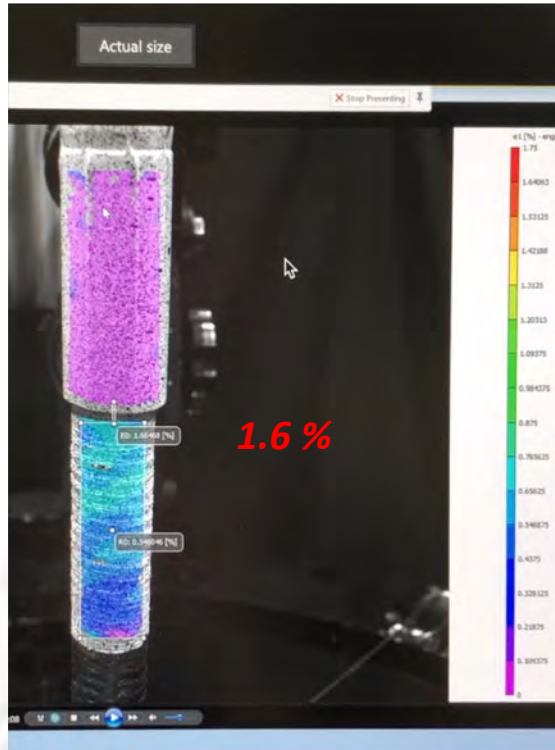
**Method of Fitting Installation (physical drill, physical pressure, and thermal viscoelastic)
Clearly Identifies Extent of Core Damage Prior to Burst Testing**

Accomplishments in New Fittings

In-situ pressurization – leak and strain characterization

















Digital Image Correlation with in-situ 25,000 psi Strain Characterization at PNNL



DIC Verified Leakage in Fitting Coupling and 3% strain at Hose/Fitting Interface at 25k psi

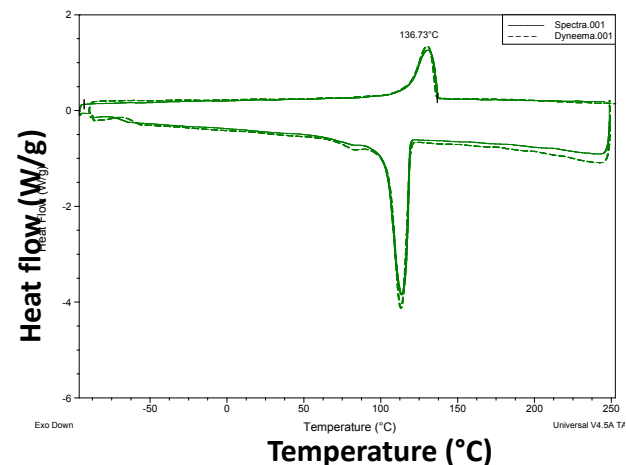
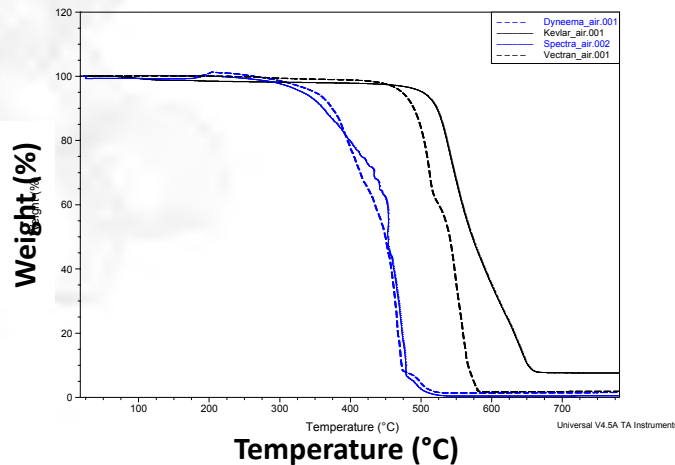
Accomplishments in Burst Strength

Sample ID	Sample Length (in)	Hose ID (mm)	Fitting Type	Burst	Leaked	Before	After
11 - 2/8/18	14"	1/4"	Swagelok	21,681			
12 - 2/8/18	14"	1/4"	Swagelok	17,995			
13 - 2/8/18	14"	1/4"	Swagelok	20,485			
14 - 2/8/18	14"	1/4"	Swagelok	18,236			
15 - 2/8/18	14"	1/4"	Swagelok	33,134			
16 - 2/8/18	14"	1/4"	Swagelok	33,055			
17 - 2/8/18	14"	1/4"	Swagelok	31,535			

Burst Strength Values > 33,000 psi, and failure consistently at edge

Accomplishments in New Fibers

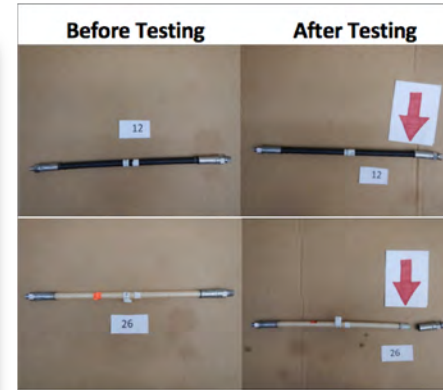
Thermal Properties



New Liquid Crystalline Polymer Fiber Reinforced Hoses with Enhanced Flexibility meet both High (T_m 137 °C) and Low Temperature Resilience Requirements (-196 °C)

Accomplishments in New Fibers

Burst Strength

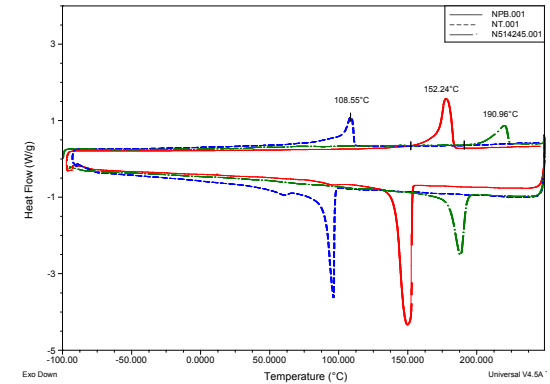
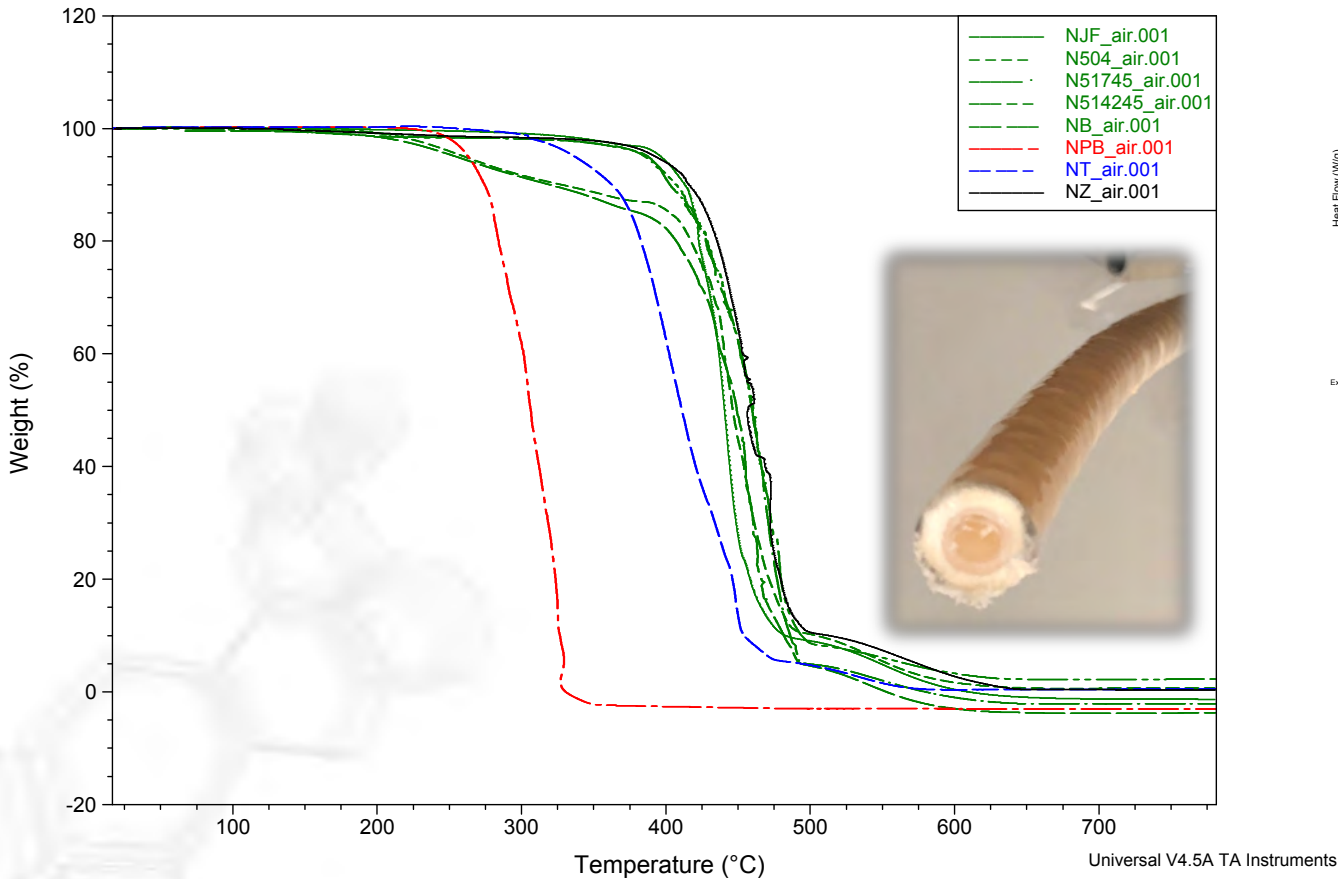


Hose Core	Average Filament Wound Burst Strengths		
	Carbon Burst (psi)	New Fiber 1 Burst (psi)	New Fiber 2 Burst (psi)
A	32,000	16,000	15,000
B	36,000	32,000	26,000
C	36,000	32,000	15,000

New Fiber Reinforced Hoses and More Flexible Cores Offer Burst Strength > 32,000 psi and Failure Remains Due to Fitting Slippage

Accomplishments in Cores

Thermal Properties and H₂ Permeation Underway



NanoSonic Shall Modify Cores With Exfoliated Clays, Tailor ID, and Evaluate H₂ Permeation

Accomplishments in New Modified Cores

Thermal Properties



5% wt loss

Benchmark: 368 °C

Clay 1: 358 °C

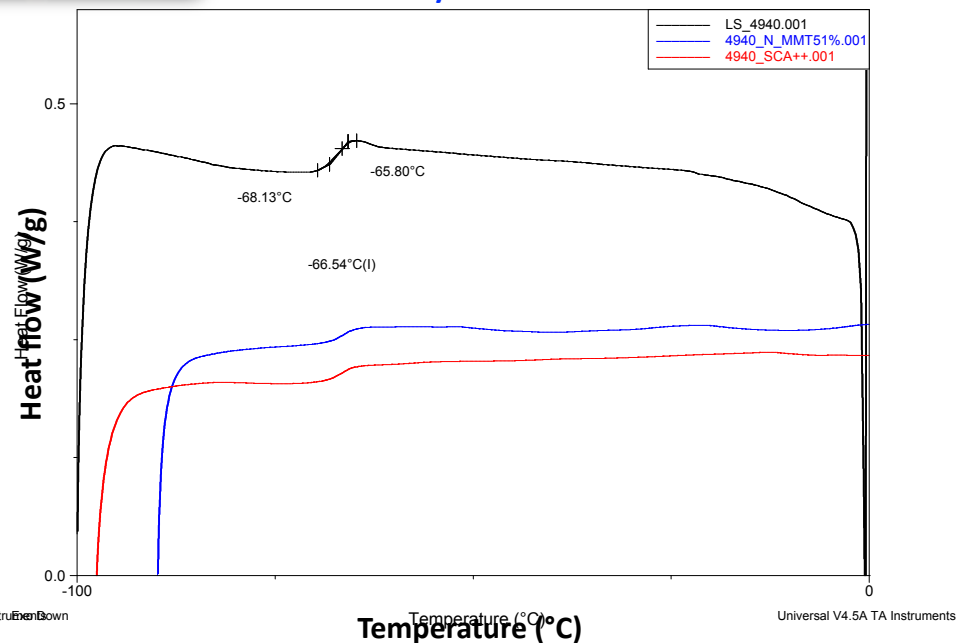
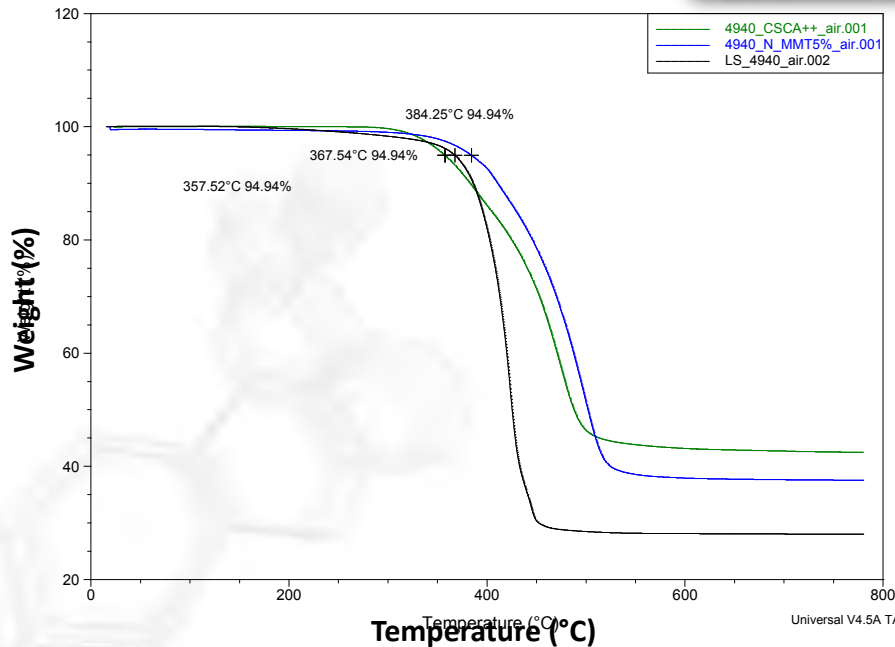
Clay 2: 385 °C

T_g

Benchmark: -67 °C

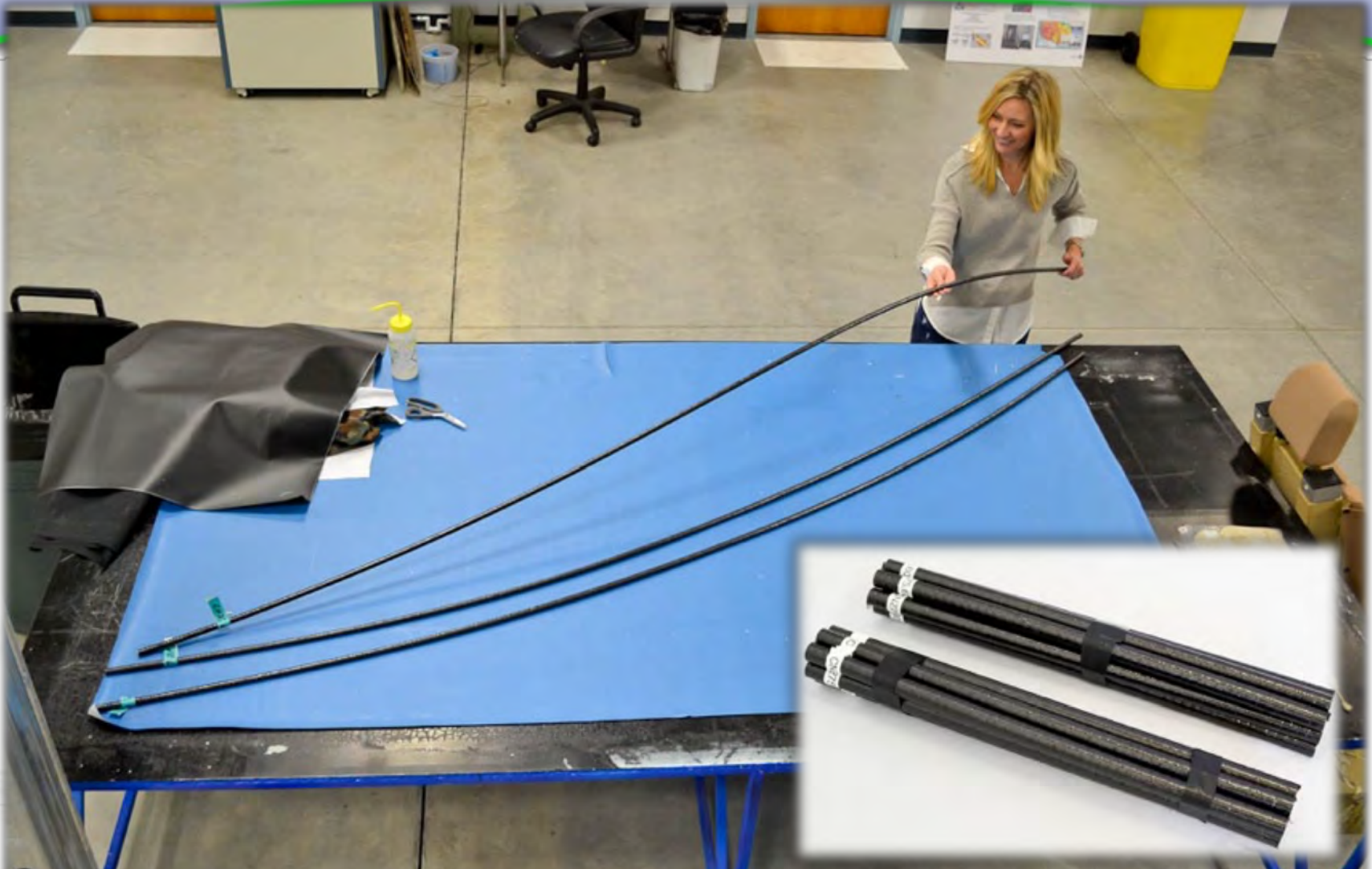
Clay 1: -67 °C

Clay 2: -67 °C



Exfoliated clays offer potential for significantly less H₂ effusion, enhanced high temperature stability and with no change in low temperature flexibility

Accomplishments in Scaling



Scaled Hoses to 3m lengths using filament winding process in-house

Econo-Technical Accomplishments

Cost



- NanoSonic can Produce 16 H₂ hoses / day, 3-m in length, at ~\$600 / Hose with Fittings
- ~40% reduction over current hose
- The 4 Spool Filament Winder Enables Multiple Fiber Functionality and Reinforcement within High Performance Custom Polymer Matrix Resins

Phase II B

Commercialization and Collaborations



Shell
Global

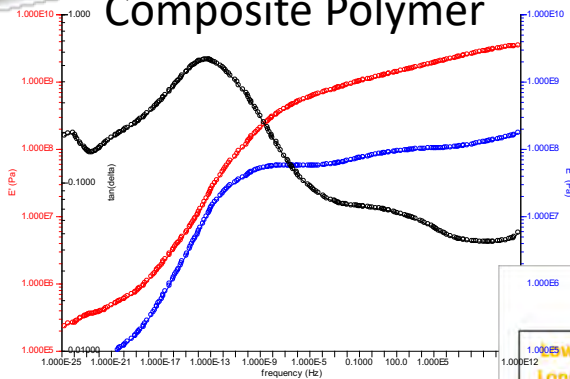


Ongoing Work

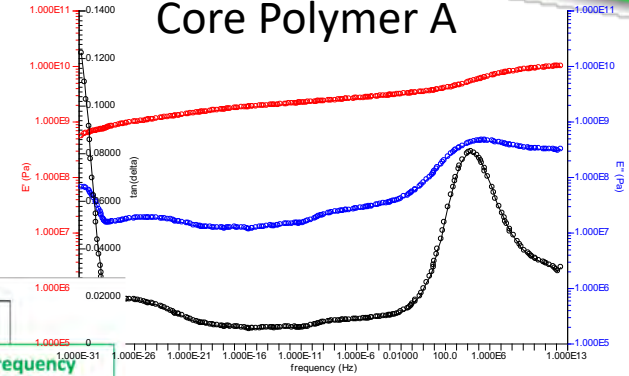
Compare DMA TTS in air vs. H_2 (at PNNL) to assess performance over 2 years

LIFETIME PREDICTION in HYDROGEN

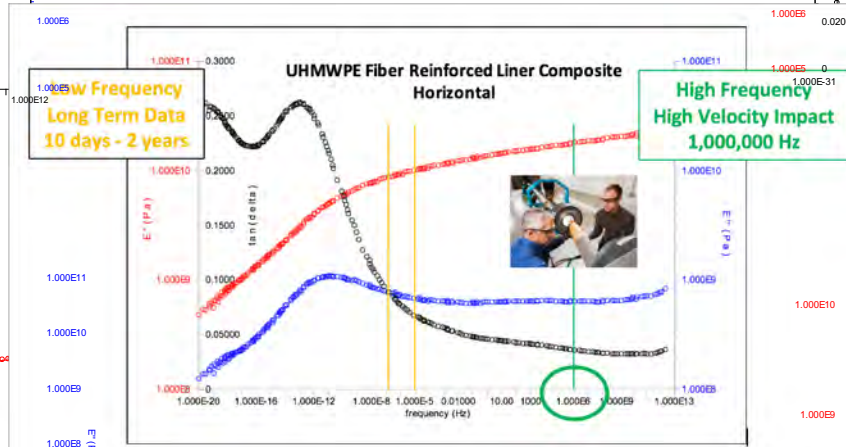
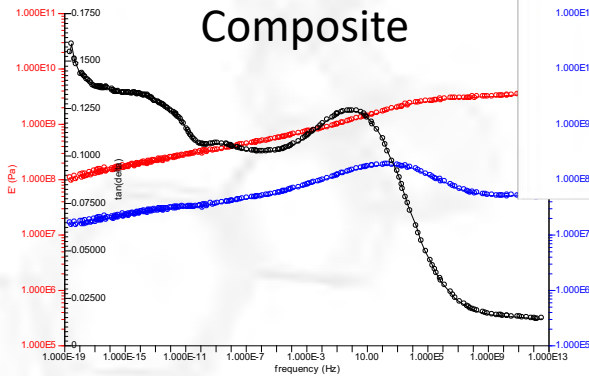
Composite Polymer



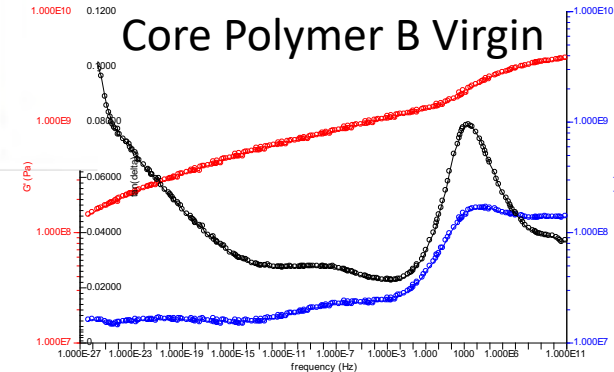
Core Polymer A



Composite



Core Polymer B Virgin



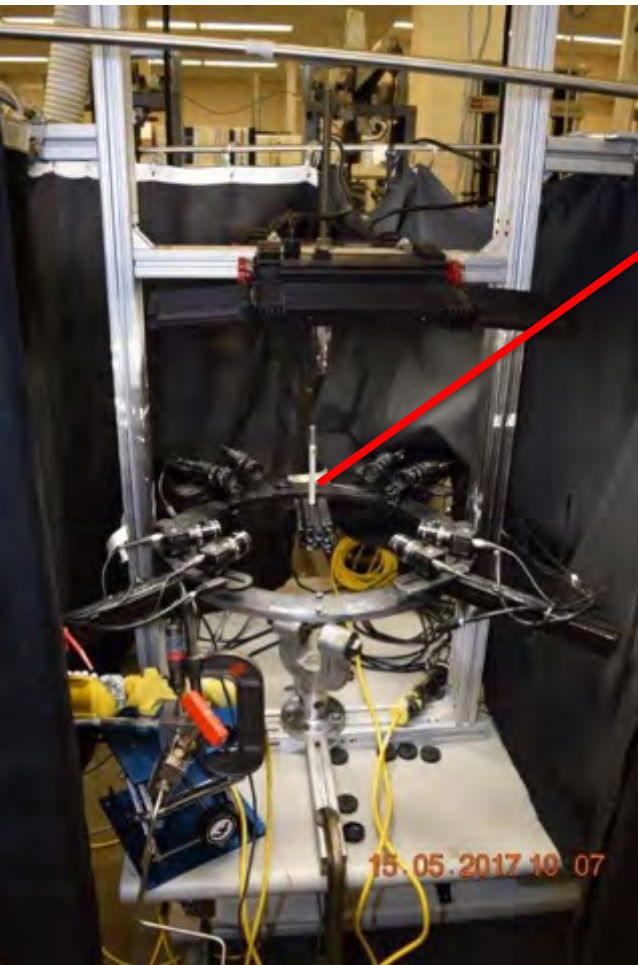
- NanoSonic DMA TTS data for polymer core, polymer composite, composite in air
- Core Polymers analyzed before and after hydrostatic burst testing

Future Work

H₂ Service and Durability Testing at PNNL and NREL

DIC System for multi-strain imaging during tube burst test

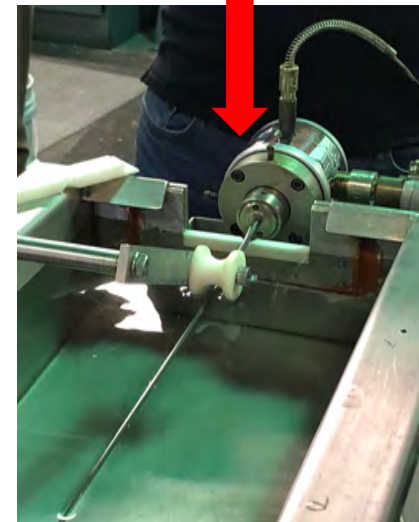
Pacific Northwest
NATIONAL LABORATORY
Funded by the Department of Energy



Remaining Challenges and Barriers:

Fitted Hose with Commercial H₂ Hose Safety Adapters Qualification and Deployment

- Challenge: Deployment date of FY19 Q1 because of fitting construction
- Resolution: Partnered with machinist to produce durable tailored high pressure fittings
- Benefit: Sell hose and fitting as a complete qualified set
- Benefit: Investigating new fitting materials and designs for the broader hose market



Project Summary

- **Relevance**: Durable and cost effective H₂ delivery hose that resists H₂ embrittlement, survives 25,550 fills/year for H70 service, cycled at pressures > than 875 bar over a range of -50 °C to 90 °C. A single qualified hose exists.
- **Approach**: NanoSonic's all polymer new class D hydrogen dispensing hose, for use on H70 station side applications, is chemically engineered to survive 51,240 fills, resist H₂ embrittlement, survive Joule-Thompson effect, and endure mechanical fatigue at the pump. Innovative SiC ceramer adhesive is under development to enhance fitting durability
- **Technical Accomplishments**:
 - Demonstrated hydrostatic burst strength > 36,000 psi
 - Demonstrated 50,000 cycles at -40C / 12,000 psi, and ~ 2,000 cycles at +85C / 12,000 psi
 - Failure for hydrostatic burst and pressure impulse each hose is at crimped fitting edge
 - Developing fitting with manufacturer and partnered with H₂ safety fitting expert
- **Proposed Future Research**: Evaluate hose under H₂ service conditions at NREL, PNNL, and at partner/distributor test facilities. Present H₂ hose partners (dispensing stations and fittings/breakaway/fueling nozzle OEMs) with integration and cost.

Questions & Acknowledgements

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Charles James, Grace Ordaz,
Laura Hill, and Erika Gupta

Phase II Integrators and Testing Facilities

