

2018 DOE Hydrogen and Fuel Cells Program and Vehicle Technologies Office
Annual Merit Review and Peer Evaluation Meeting



Cryogenically Flexible, Low Permeability
H₂ Delivery Hose

P. I.: Dr. Jennifer Lalli, Chief Development Officer
NanoSonic, Inc.
6/14/2018

Project ID #
PD149



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
- NREL
- PNNL
- Cardinal Rubber & Seal
- LifeGuard Technologies
- Shell, Tatsuno, and WEH
- Giles County Government

Relevance:

Develop a H₂ Hose for Fuel Cell Vehicles

Objectives:

- Flexible: H₂ hose to enable delivery < \$2/gge
- 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 > 33,000 psi for H35 Service
- Goal: Metal Free Hose Design and Custom Fitting to Enable Long-term H70 Service

Current Approach to Hoses up to 20' Long: *Filament Wound Metal-free H₂ Hose*

NanoSonic Flexible Composite Hoses Exhibit Hydrostatic Burst Strength > 33,000 psi

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.



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

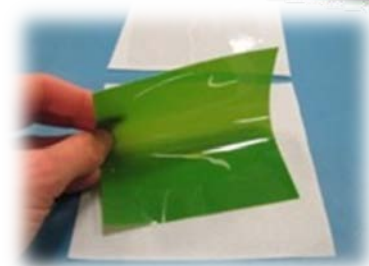


Critical Criteria

- 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

Accomplishments in Design

Evolution of the High Pressure Hose



Hydrogen Permeance by ASTM D 1434

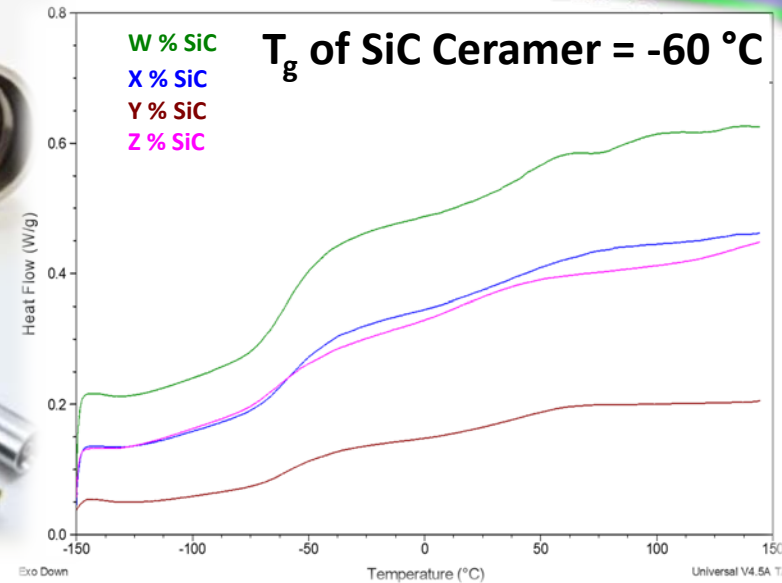
| | |
|--------------------------|------------------|
| Sample No. | 0.29 |
| | 0.22 |
| | 0.20 |
| Set 4 - Lot # LB199-119 | |
| Hydrogen - 10A, 10B, 10C | AV = 0.24 ± 0.04 |
| | |
| Hydrogen Cold - | 0.36 |
| 10A, 10B, 10C | 0.35 |
| | 0.20 |
| | AV = 0.31 ± 0.09 |



The ideal burst pressure was estimated for the single braid reinforced hoses to be ~ 2560 bar using Barlow's Formula: $P = 2 s t / (d_o SF)$
 Where, P = max. working pressure (psig), s = material strength (psi) = 10.2×10^6 (Aramid fiber); yield strength = 522,000 psi. t = wall thickness (in) = 0.02" (Braid thickness),
 d_o = outside diameter (in) = 0.563" (9/16"), SF = safety factor (in general 1.5 to 10) = 1; Calculated maximum working Pressure 37,129 psi (2560 Bar or 256 MPa) 6

Accomplishments in Manufacturing

Fittings Crimped In-House with Polymer Derived Ceramer Coupling Agent



| Test ID | Hose | Length (inch) | Fitting Manufacturer | Fitting OD | Ceramer in Hose | Ceramer at Fitting | Burst Pressure (psi) |
|----------|------|---------------|----------------------|-------------|-----------------|--------------------|----------------------|
| WH208-8C | A | 17 | 1 | proprietary | x | | 6446 |
| WH208-8D | A | 17 | 1 | proprietary | x | x | 8334 |
| WH208-8E | A | 17 | 2 | proprietary | x | | 4304 |
| WH208-8F | A | 17 | 2 | proprietary | x | x | 7016 |
| WH208-9A | B | 17 | 1 | proprietary | | | 52959 |
| WH208-9B | B | 17 | 1 | proprietary | | x | 58440 |
| WH208-9C | B | 17 | 2 | proprietary | | | 9635 |
| WH208-9D | B | 17 | 2 | proprietary | | x | 26136 |

Ceramer increases burst strength by ~ 25% as a coupling agent

Accomplishments in Burst Strength

NanoSonic Carbon Fiber Hose = 31,421 psi

| HOSE MFG | HOSE ID | Fitting Mfg | ACTUAL NIPPLE OD (mm) | NIPPLE | Hose O.D (mm) | Ceramer | Wrap Thickness (mm) | Number o Wraps | Wrap Angle | Burst Strength (psi) | average weight (g) |
|-----------|--------------|-------------|-----------------------|--------|---------------|---------|---------------------|----------------|---------------------|----------------------|--------------------|
| NanoSonic | 7-1 | Swagelok | 14.9 | 2.4 | 12.5 | yes | 3.0 | 3 | 54 | 19,237 | 200 |
| | 7-2 | Swagelok | 15.3 | 3.2 | 12.1 | yes | 2.6 | 3 | 54 | 26,614 | |
| | 7-3 | Swagelok | 15.8 | 3.8 | 12.0 | yes | 2.5 | 3 | 54 | 25380 | |
| | 8A-1 | Swagelok | 16.1 | 2.5 | 13.6 | yes | 4.1 | 4 | 54 | 31,421 | |
| | 8A-2 | Swagelok | 16.5 | 3.4 | 13.1 | yes | 3.6 | 4 | 54 | 30,475 | |
| | 8A-3 | Swagelok | 17.0 | 4.1 | 12.9 | yes | 3.4 | 4 | 54 | 16357 | |
| | 8B-1 | Swagelok | 15.2 | 2.0 | 13.2 | yes | 3.7 | 4 | 54 | 22,591 | |
| | 8B-2 | Swagelok | 15.2 | 2.3 | 12.9 | yes | 3.4 | 4 | 54 | 30,576 | |
| | 8B-3 | Swagelok | 16.1 | 3.4 | 12.7 | yes | 3.2 | 4 | 54 | 28063 | |
| | 13-1 | Swagelok | 15.6 | 2.4 | 13.2 | yes | 3.7 | 4 total | 3 at 54 - 1 at hoop | 24,916 | |
| | 13-2 | Swagelok | 16.0 | 3.3 | 12.7 | yes | 3.2 | 4 total | 3 at 54 - 1 at hoop | 24,273 | |
| | 13-3 | Swagelok | 16.4 | 3.5 | 12.9 | yes | 3.4 | 4 total | | 23522 | |
| | 41A | Swagelok | 16.1 | 3.2 | 12.9 | yes | NA | NA | NA | 21,191 | |
| 41B | Swagelok | 16.1 | 3.2 | 12.9 | yes | NA | NA | NA | 10,096 | | |
| SpirStar | WH208-9A2089 | Swagelok | 15.9 | 3.0 | 12.9 | yes | NA | NA | NA | 52,959 | 250 |
| | WH208-9B2089 | DHH | 15.9 | 3.0 | 12.9 | no | NA | NA | NA | 9,635 | |
| | WH208-9C2089 | Swagelok | 15.9 | 3.0 | 12.9 | yes | NA | NA | NA | 58,449 | |
| | WH208-9D2089 | DHH | 15.9 | 3.0 | 12.9 | no | NA | NA | NA | 26136 | |

Composite: ~ 10,000 psi

Core Alone: ~ 3,000 psi

CryoHose: > 30,000 psi

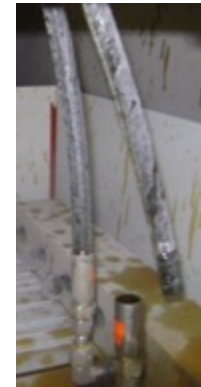
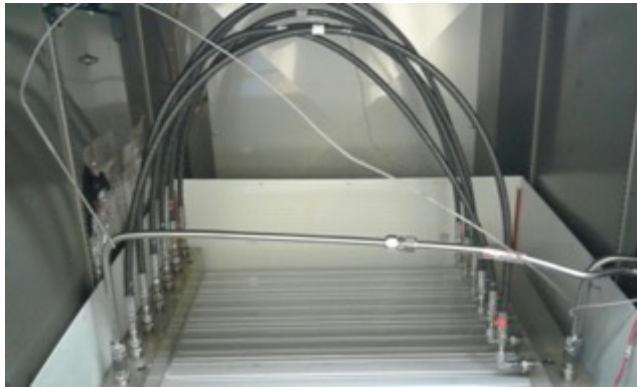


Failure Mode for NanoSonic Hose is at the Fitting – Slippage
 Failure Mode for Metal Wrapped Hose is – Hose Burst

Accomplishments in Pressure Cycle Testing: *52,000x at 12,000 psi (-40 °C to 85 °C)*

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



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

Failure Mode for NanoSonic Hose is at the Fitting – Slippage

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 |
| 17 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 18 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 19 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 20 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

Fittings Swaged at Cardinal Passed High Pressure Testing

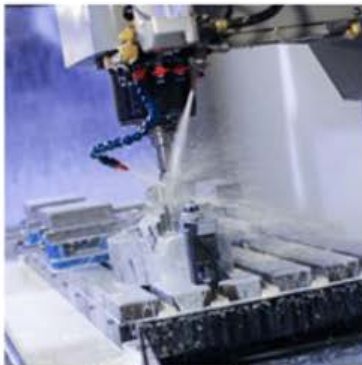
Accomplishments with Techsburg Manufacturing



Engineering or Manufacturing,
Techsburg is Your Partner in **Creation**



Engineering Services Division



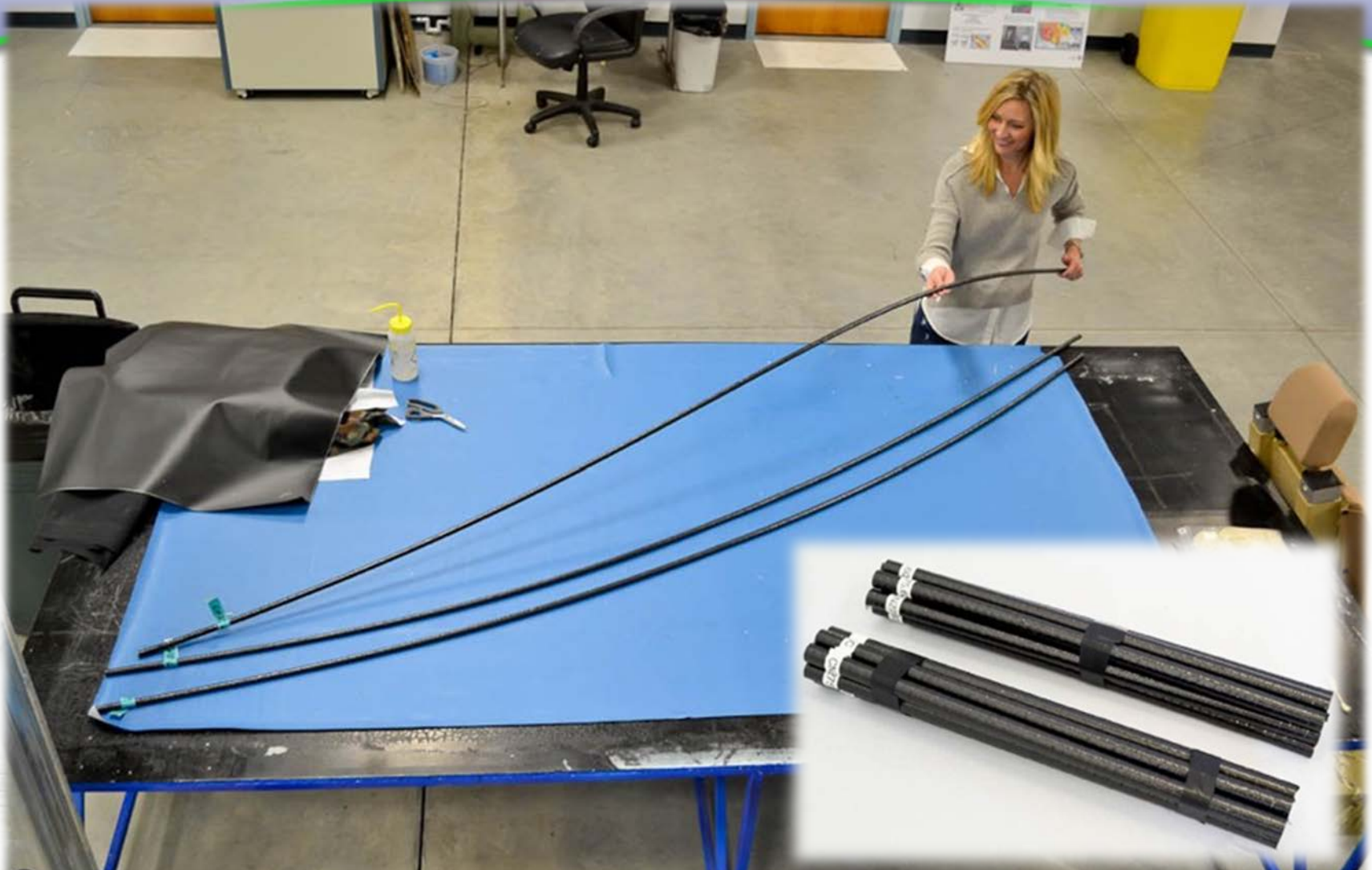
Manufacturing Division



| | | | |
|------------------------|----------|---|------|
| DESIGN | DATE | NanoSonic, Inc. | |
| NAME | PROJECT | Hydrogen Hose Connector, Crimp Design, Threaded | |
| CHECKED | | | |
| BY | | | |
| MATERIAL | QUANTITY | REV | DATE |
| NanoSonic: Proprietary | | A | 1001 |
| Dimensions in inches | APPROVED | SCALE | 1:1 |



















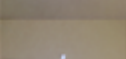
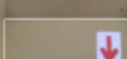
2-piece fitting any metal, starting at \$4/part

Accomplishments in Scaling



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















Accomplishments in Burst Strength

| Sample ID | Sample Length (in) | Hose ID (mm) | Fitting Type | Burst | Leaked | Before | After |
|-------------|--------------------|--------------|--------------|--------|--------|---|---|
| 1 - 2/8/18 | ~14" | 1/2" | Swaged | 18,462 | |  |  |
| 2 - 2/8/18 | ~14" | 1/2" | Swaged | 20,971 | |  |  |
| 3 - 2/8/18 | ~14" | 1/2" | Swaged | | 11,526 |  |  |
| 4 - 2/8/18 | ~14" | 1/4" | Swaged | 28,192 | |  |  |
| 5 - 2/8/18 | ~14" | 1/4" | Swaged | 28,809 | |  |  |
| 6 - 2/8/18 | ~14" | 1/4" | Swaged | 28,989 | |  |  |
| 7 - 2/8/18 | 14" | 1/4" | Swaged | | 12,365 |  |  |
| 8 - 2/8/18 | 14" | 1/4" | Swaged | 23,853 | |  |  |
| 9 - 2/8/18 | 14" | 1/4" | Swaged | 26,236 | |  |  |
| 10 - 2/8/18 | 14" | 1/4" | Swaged | | 12,739 |  |  |



Burst Strength Values > 28,000 psi, and failure through fitting failure or at edge

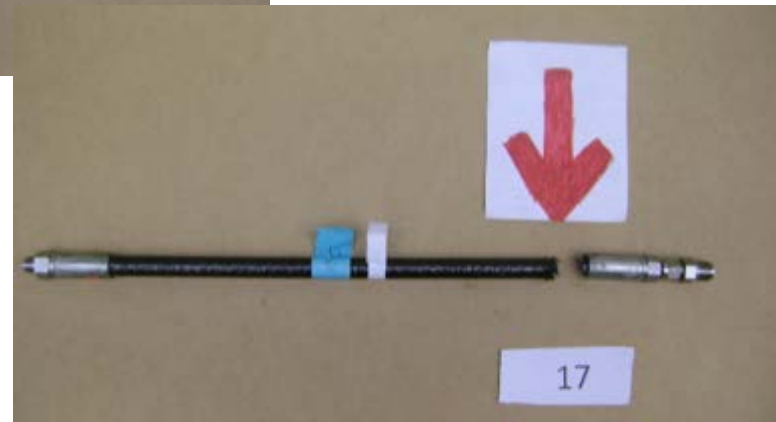
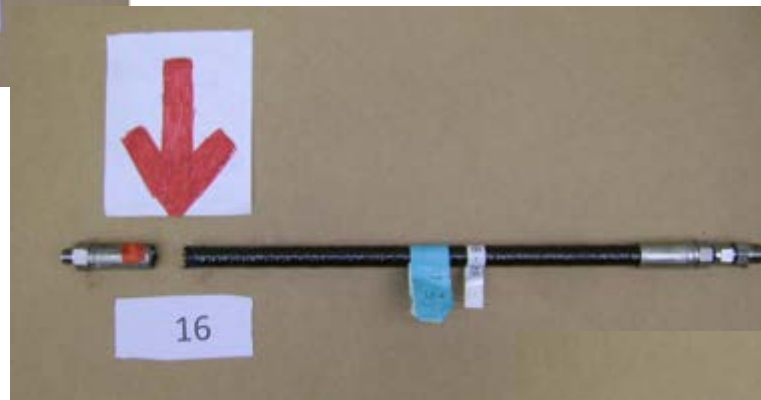
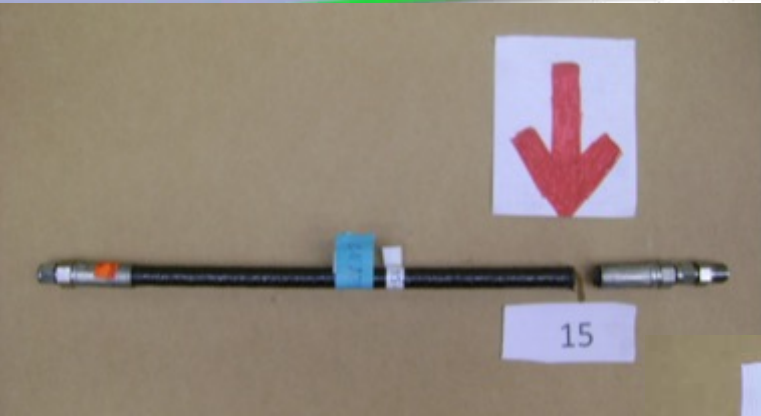
Accomplishments in Burst Strength

| 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

Challenges with Fittings

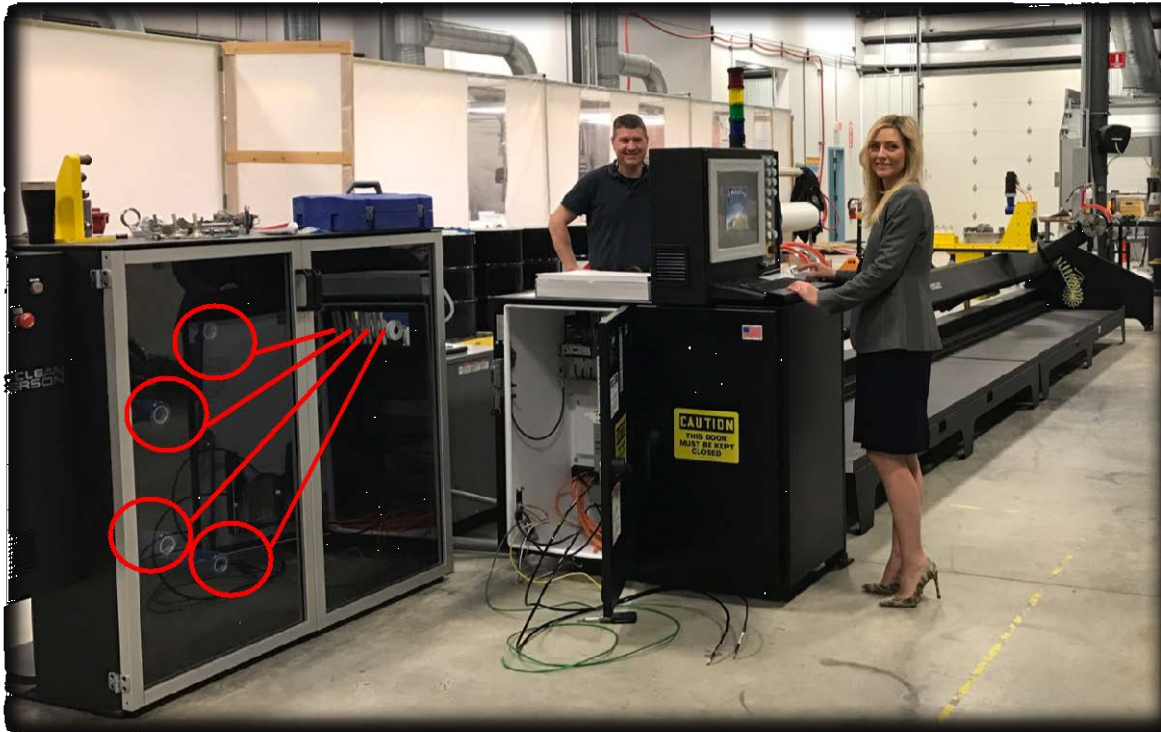
All Fittings Fail at Crimped Edge



Fittings Fail due to Under-Crimping (Slippage) or Over-Crimping (Core Defect) at Edges

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



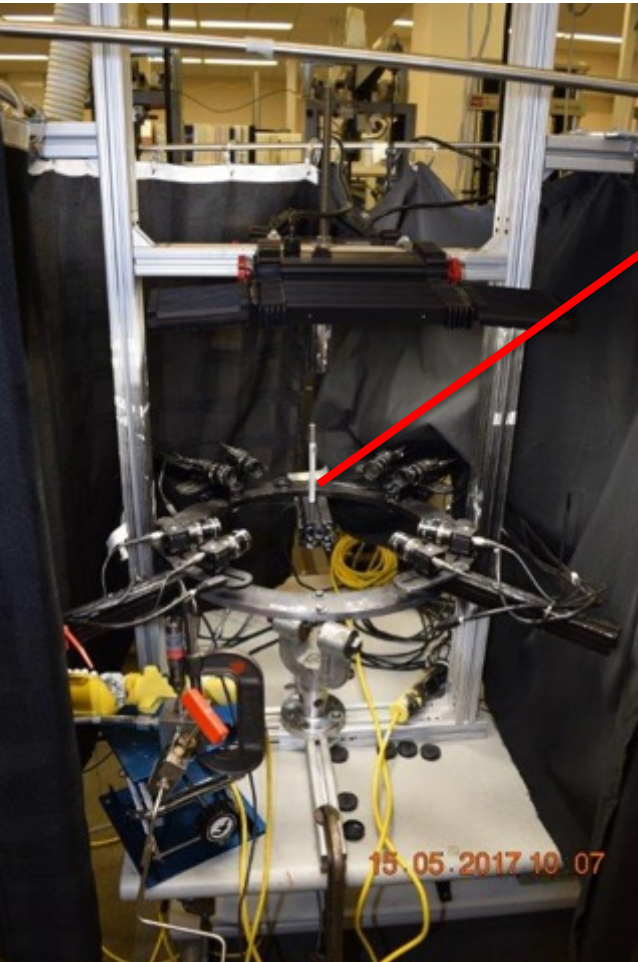
Future Work

H₂ Service and Durability Testing at PNNL and NREL

Any proposed future work is subject to change based on funding levels

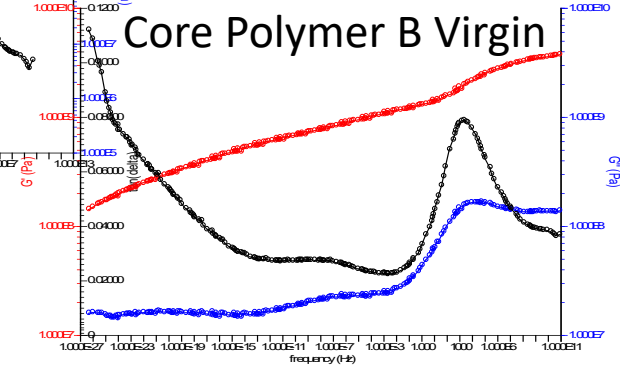
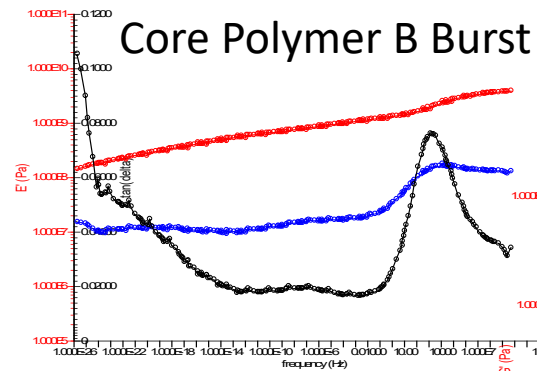
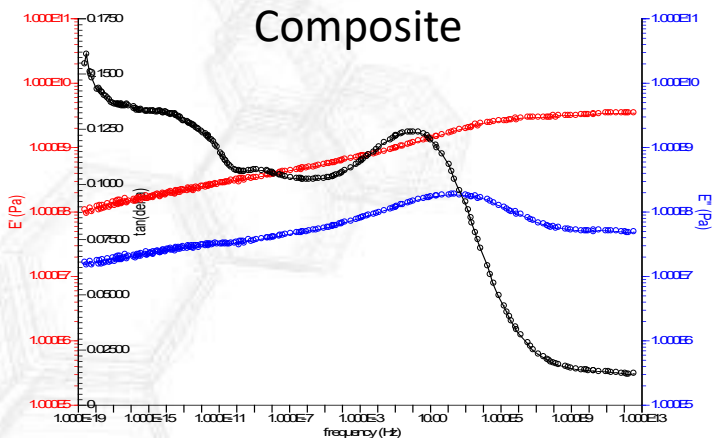
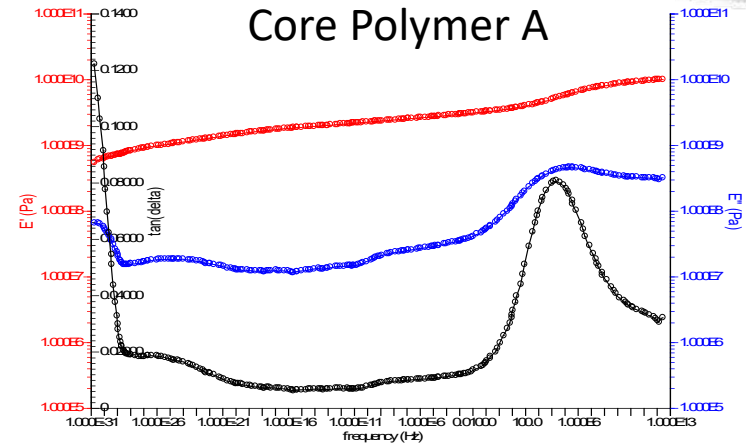
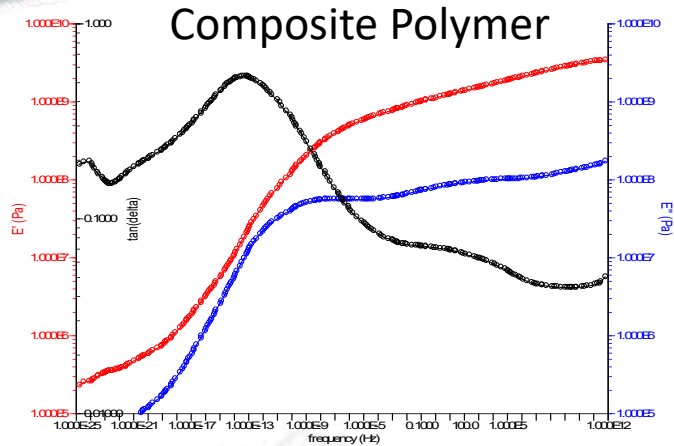
DIC System for multi-strain imaging during tube burst test

Pacific Northwest
NATIONAL LABORATORY
Fossilly Sponsored by **Battelle** Since 1962



Future Work

Compare DMA TTS in air vs. H₂ (at PNNL) to assess performance over 2 years

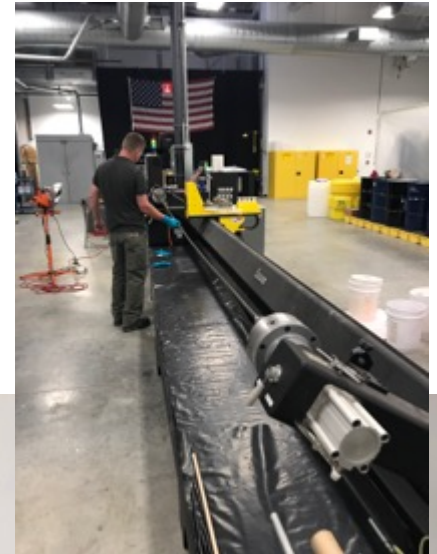


- NanoSonic DMA TTS data for polymer core, polymer composite, composite in air
- Hose Core Polymers analyzed before and after hydrostatic burst testing

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 > 33,000 psi
 - Demonstrated 50,000 cycles at -40F / 12,000 psi, and ~ 2,000 cycles at +85F / 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

This material is based upon work supported by the Department of Energy under Award No. DE-SC0010162

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

Phase II Integrators and Testing Facilities

