



**2020 DOE Hydrogen and Fuel Cells Program
Annual Merit Review (AMR) and Peer Evaluation Meeting**

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6/1/2020

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



Overview

Timeline Ph IIB:

- Project Start Date:
7/31/2017
- Project End Date:
7/29/2021

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 (CRADA #399)
- NREL
- Techsburg Machining
- Cardinal Rubber & Seal
- LifeGuard Technologies
- Shell, Tatsuno, WEH and Air Liquide
- Giles County Government

Approach:

Project Phases and Selected Milestones



Evaluate
critical performance metrics, fittings,
partners for deployable design

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



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

Deploy FY20 Q4
H70/H35 hoses and modified
nozzles at Ture Zero H₂ stations

Critical Criteria

- Surpass 3500 Bar hydrostatic burst strength (> 43,300 psi) held for 1 min
- Survive 875 Bar pressure cycle at (50,000x at -50 C and 50,000x at 85 C)
- No contaminant leaching, Competitive cost, mechanical durability, and environmental lifetime

STATION OWNER NEED

True Zero's Network

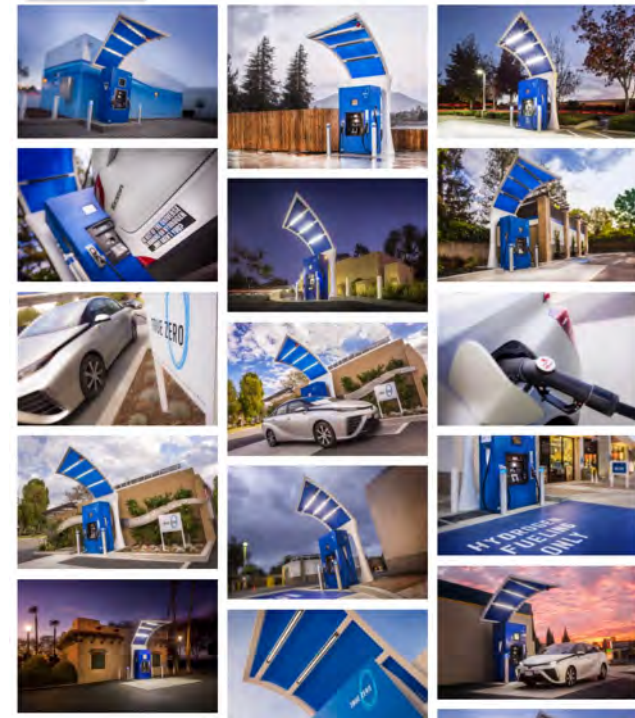


Meeting with First Element Fuel:

- Met at Headquarters, UC Irvine
- Restricted to H70 qualified products
- Limited to a handful a companies
 - Hoses: Spir Star and Parker
 - Nozzles: WEH and Walther
- Hoses - inflexible and leak
- Nozzles - freeze, leak, crack & lose communication

Station Owner Requests:

- NEED longer lifetime and lower cost materials
- WANT products that customers enjoy using
- ENJOYMENT = WORD of MOUTH = MORE FUEL CELL VEHICLES = MORE STATIONS
- Extremely interested in new materials
- Willing to test new components that have gone through baseline testing
- NanoSonic is currently producing prototypes for testing in H2 Environment at NREL through SBIR and PNNL through CRADA and SBIR

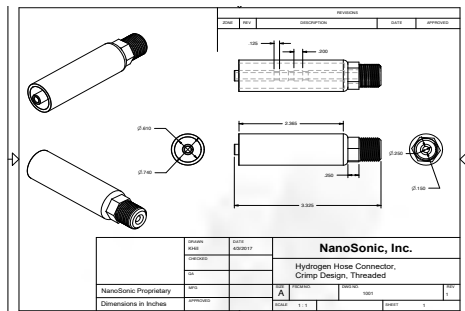


Phase IIB Commercialization Challenge

Lack of Commercial Fittings for New Hoses

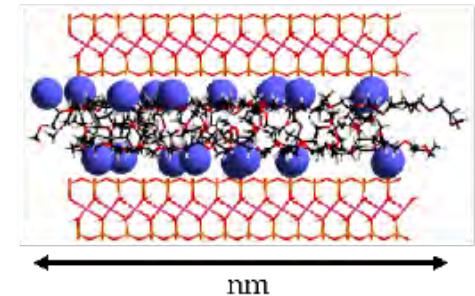
1. Design New Fittings

- 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



**7 meter
horizontal
carriage**



**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

Accomplishments in Burst Strength with Non-crimp Fitting



FITTING INSTALLED AT PARTNER SITE

With proprietary fitting

Achieved Burst Strength of >43,300 psi (very near new spec)

**Requirement may change from 4x (875 bar)
to 3x (1000 bar) = 43,511 psi**

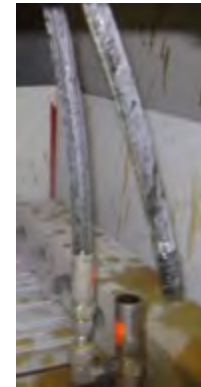
Burst Strength Values > 43,300 psi, and failure consistently at edge

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)



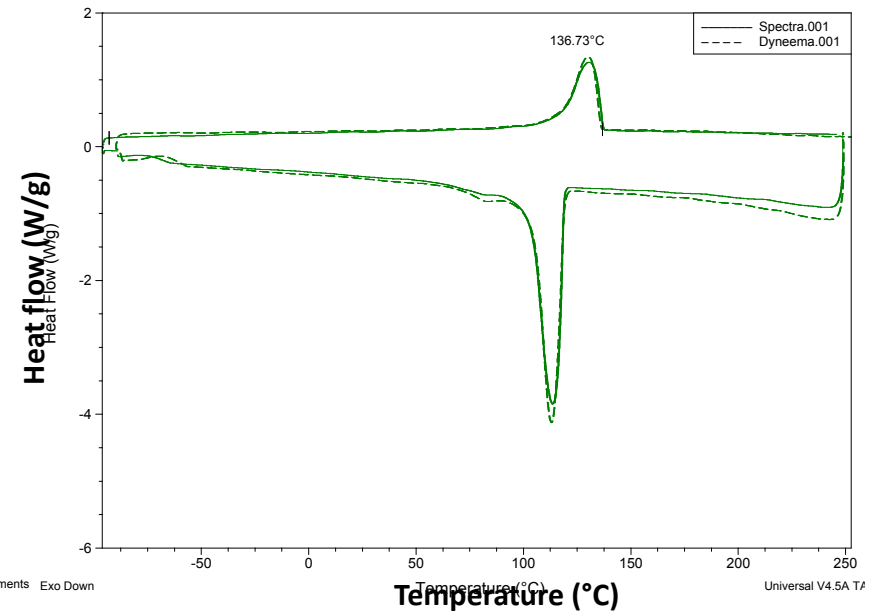
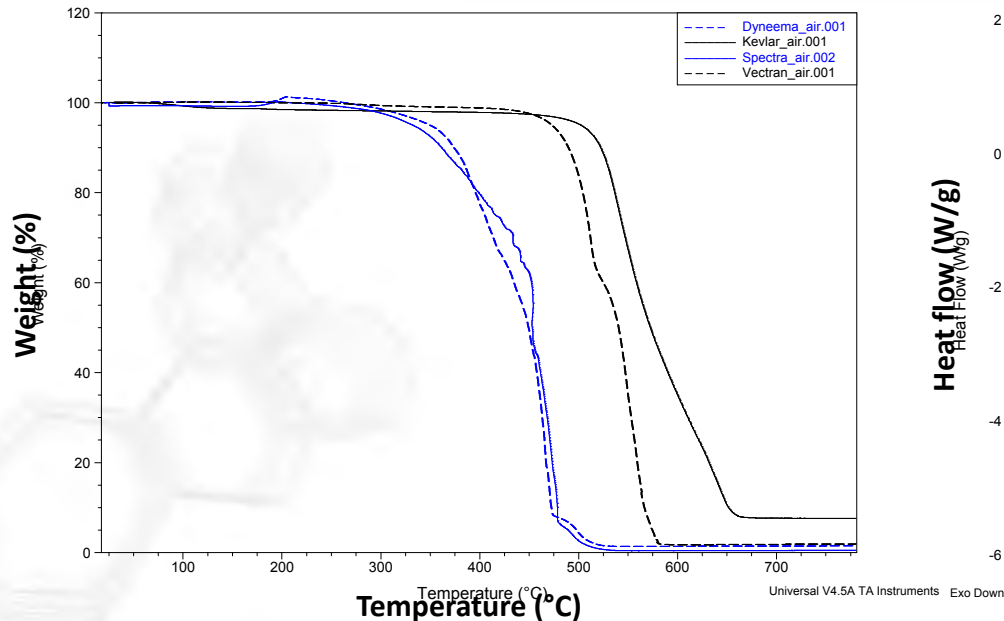
Down-selected filament wound composite survived:

- 50,000 cycles at 12,000 psi (827 Bar) at -40 °F and
- 1,988 cycles at 12,000 psi (827 Bar) at 185 °F

**NanoSonic Hose Survived 51,988 cycles at 12,000 psi over -40 °F to 185 °F
prior to leak rather than burst**

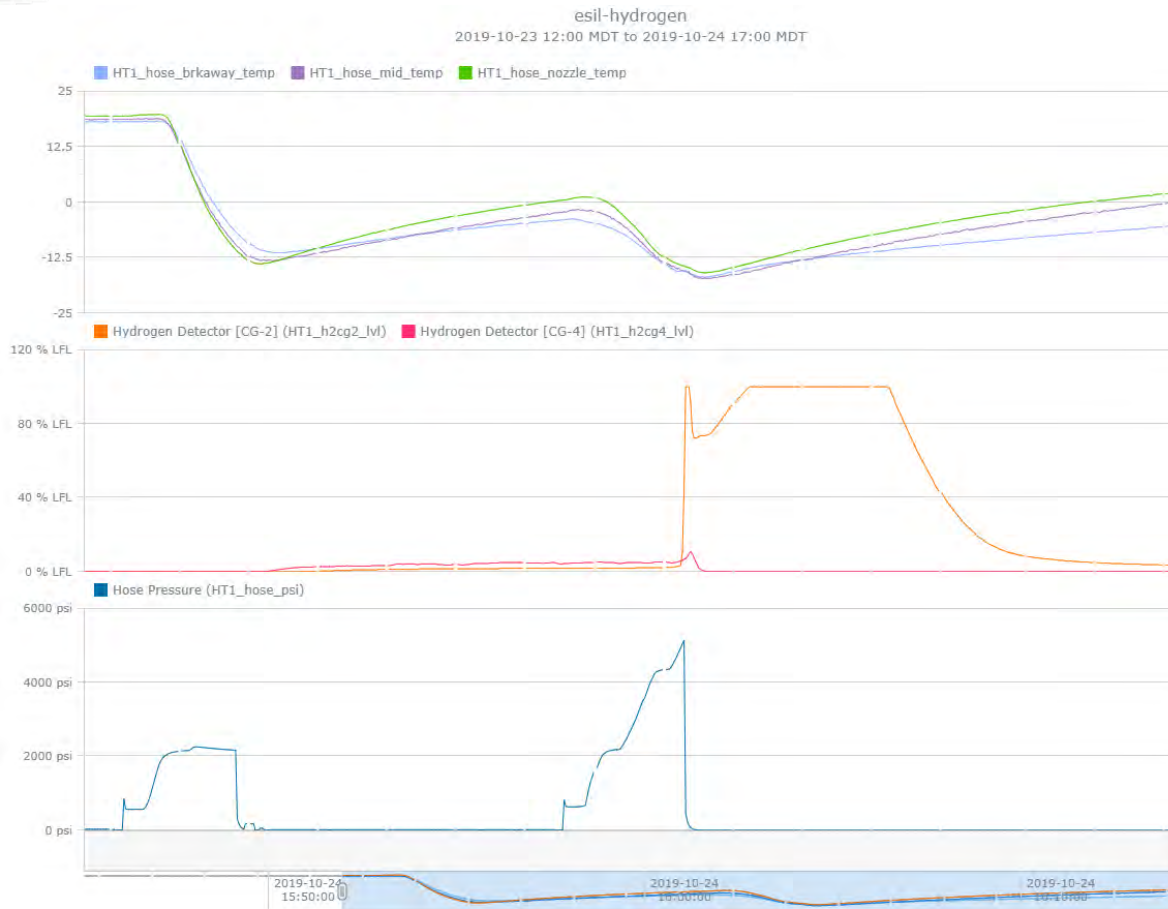
Accomplishments in New Fibers

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 H₂ Fills at NREL



No obvious failure mechanism

Could not visually see any issues around the crimp fittings

Utilized hydrogen detection tape to aid in hydrogen leak detection, slight discoloration was found around the crimps indicating a leak (nothing significant)

**H35 Fills Conducted at NREL with Crimped Fitting rated for 350 bar
New Fitting (non-crimp) in progress**

Accomplishments in H₂ Fills at NREL



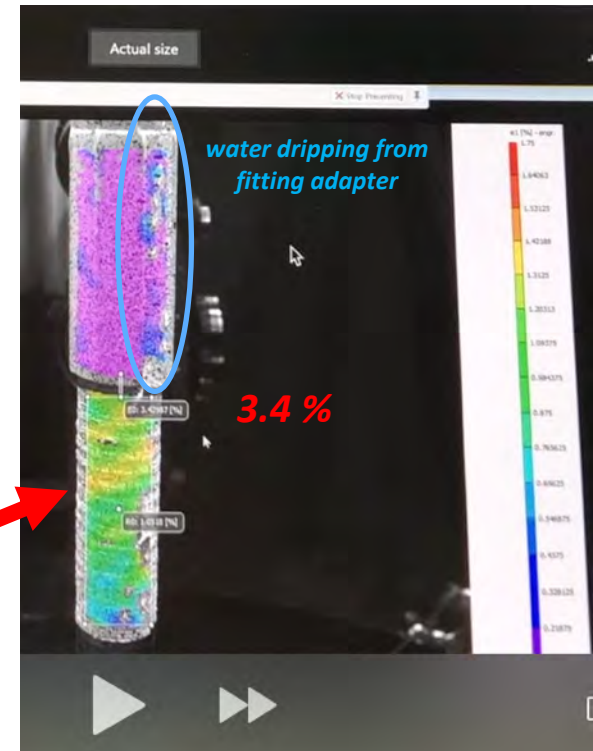
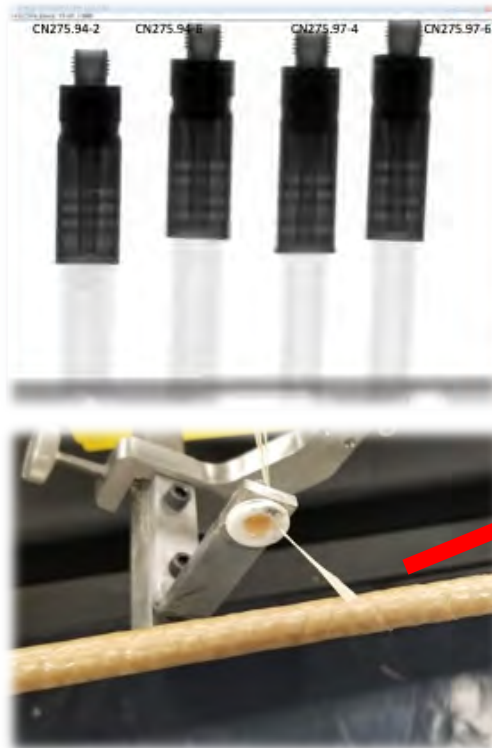
**H35 Fills Conducted at NREL with Crimped Fitting rated for 350 bar
H₂ Sensing Tape Indicates Leakage at Crimped Areas**

PNNL

Tribology, In-situ Pressurization, Leak and Strain DIC



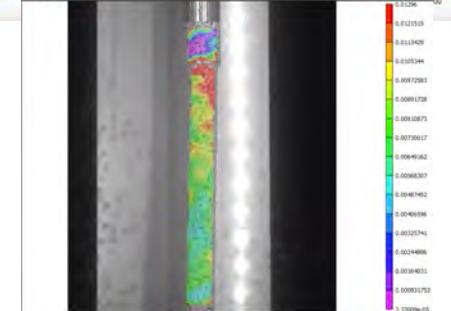
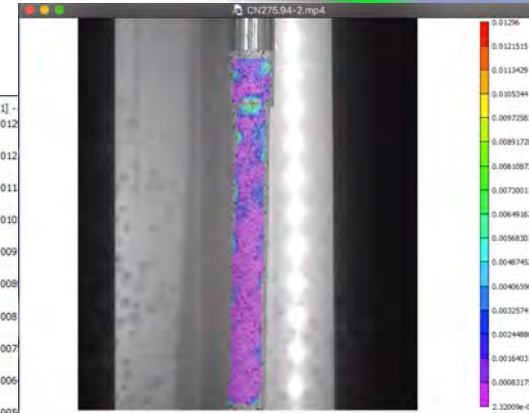
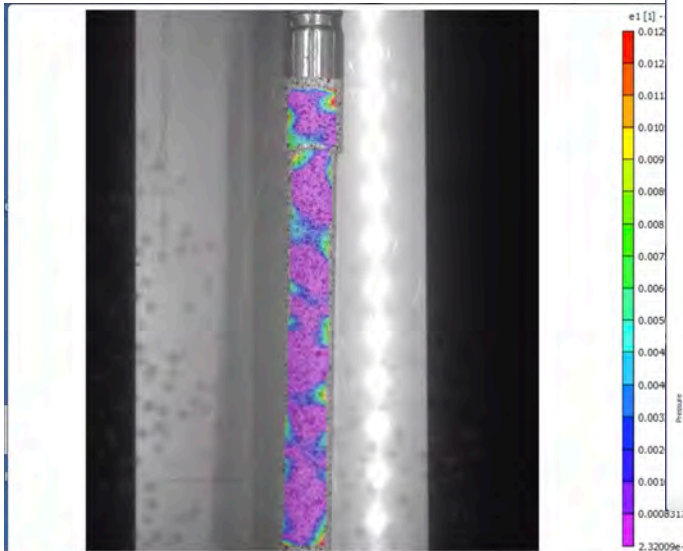
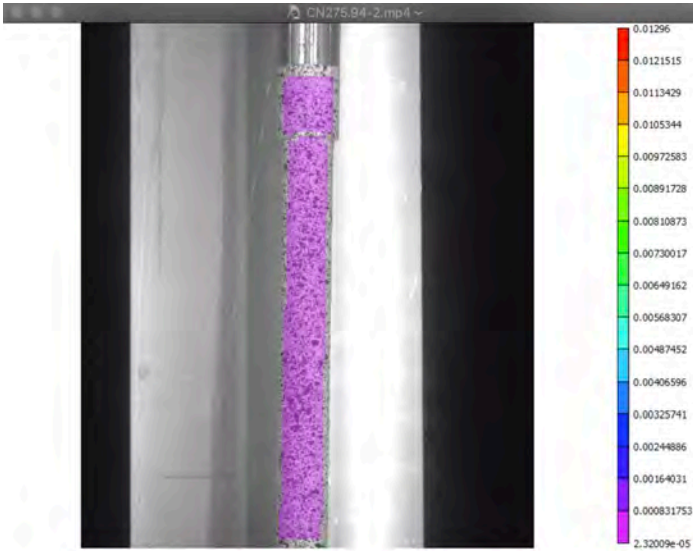
Digital Image Correlation with in-situ 25,000 psi Strain Characterization at PNNL



DIC Verified Leakage in Fitting Coupling and 3% strain at Hose/Fitting Interface at 36k psi

PNNL

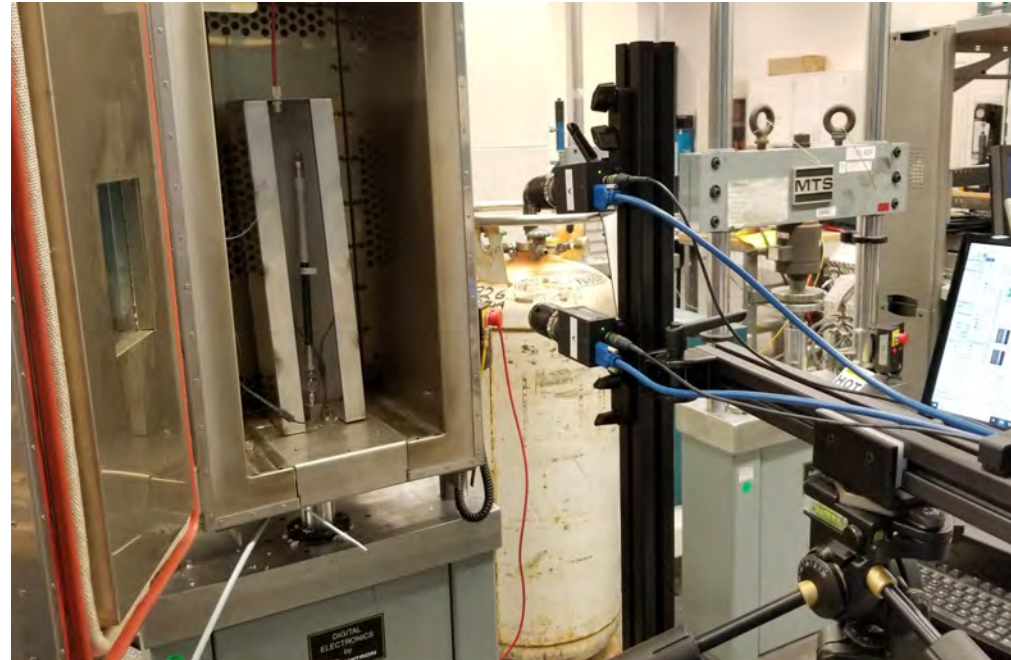
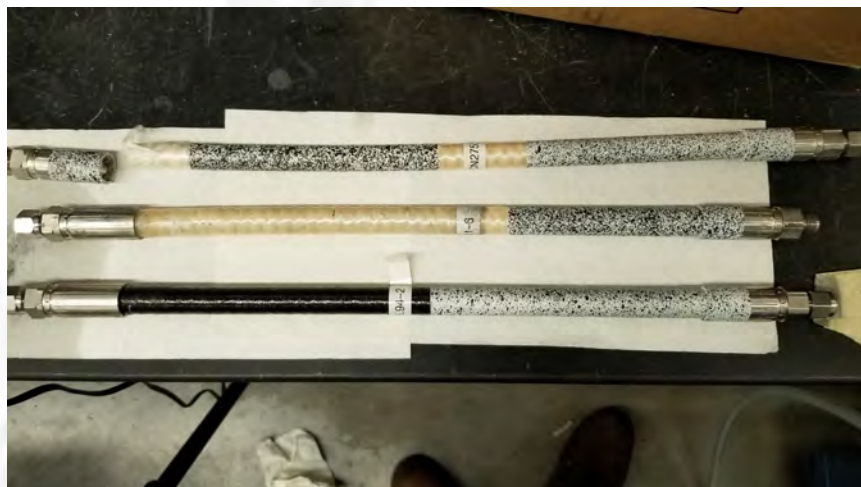
Cryo Burst Test



DIC Verified Leakage in Fitting Coupling and Failure Beyond 25,000 psi

PNNL

Cryo Burst Test

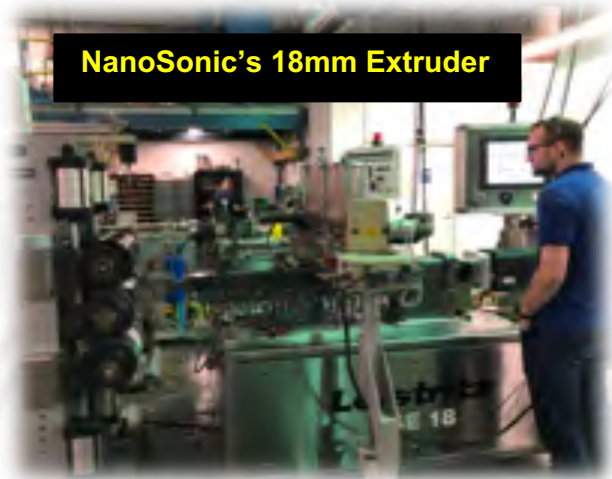
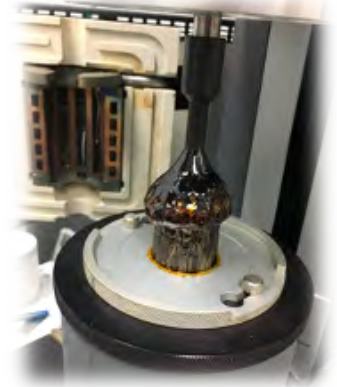


DIC Verified Leakage in Fitting Coupling and 3% strain at Hose/Fitting Interface at 36k psi

Accomplishments in Extrusion of New Filaments for 3D Printing of H₂ Hoses and Nozzles

New Materials and Manufacturing Approach:

- Expand upon NanoSonic's high pressure H₂ durable polymers into new formats
- Extrude the compounded materials as pellets or strands for (fused filament fabrication) FFF or injection molding within custom molds
- NanoSonic is building a custom 3D printer



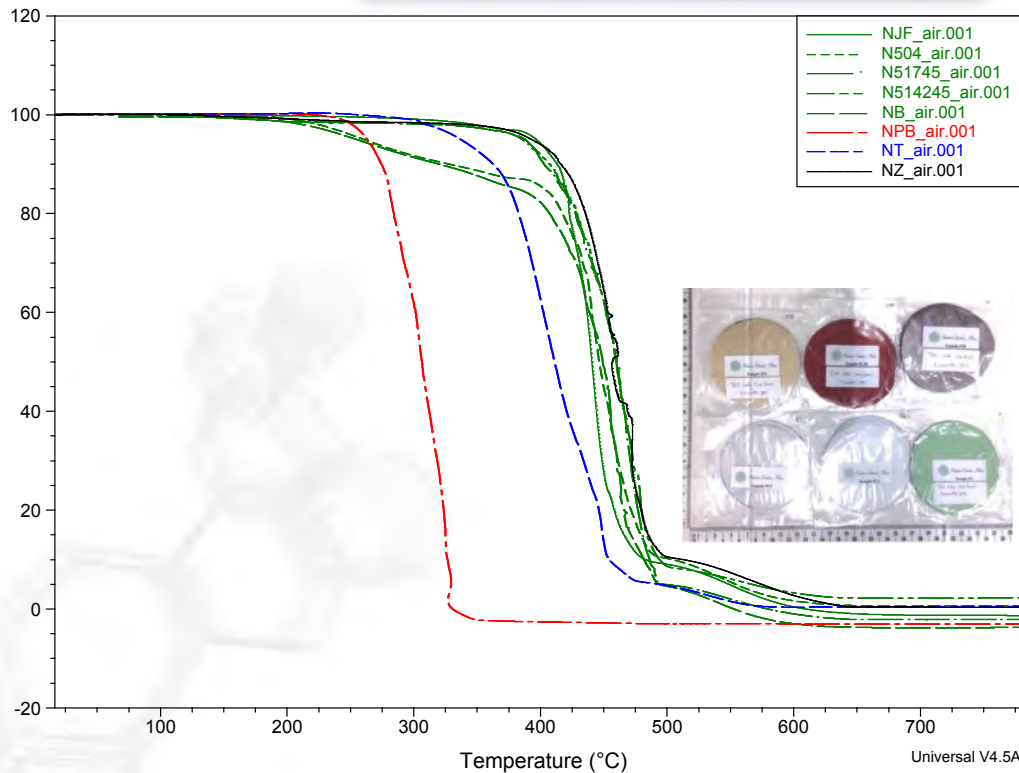
High Temperature Clay Nanocomposites

Thermal Properties and H₂ Permeation at ARDL per ASTM D D1434, Procedure V



GAS PERMEABILITY-ASTM D1434, PROCEDURE V

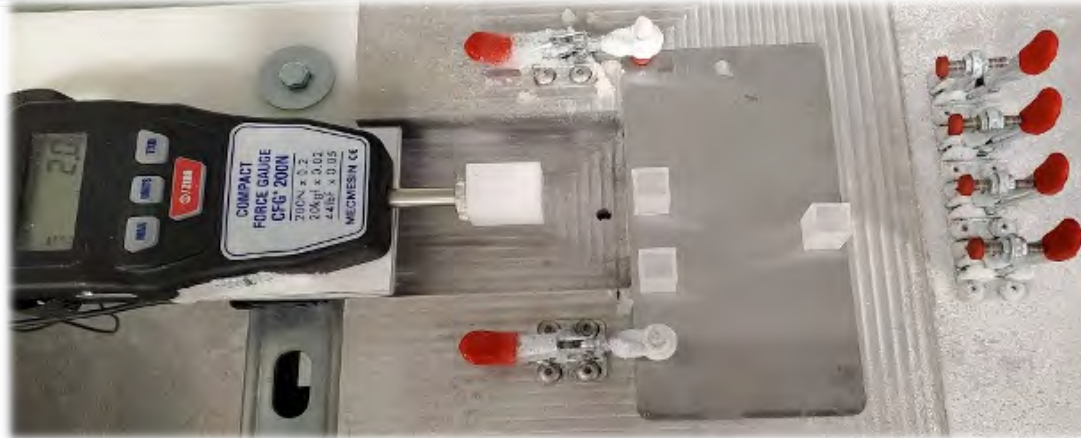
Apparatus: Custom Scientific Model CS-135
 Gas Used: Hydrogen
 Test Temperature: 23.0°C
 Test Gas Pressure: 15.0 psi
 Permeation area of the sample: 66.4 cm²
 Capillary Diameter: 0.1836 cm
 Conditioned: 48 hours



Sample #	Description	Permeance
3	NanoSonic 3	2725.02
4	NanoSonic 4	2705.09
10	NanoSonic 10	3385.50
C1	Delrin	199.20
C4	Tefzel	1410.88
C20	Silicone calendared kevlar	162852.00
C21	urethane vectran - 750 Denier	1627.39
30	V11 on It vectran control (vectran 2203)	9260.52
31	V11-10%BN/v11-20%BN-Green Vectran 2203	3994.73
32	V11-10%BN/v11-20%BN-Green/au veil Vectran 2203	4157.64
33	V11-10%BN/v11-20%BN-au veil Vectran 2203	3362.55
40	V11 on heavy vectran control (Vectran 1308/2)	873699.00
41	V11-10%BN/v11-20%BN-Green Vectran 1308/2	3287.19
42	V11-10%BN/v11-20%BN-Green/au veil Vectran 1308/2	3164.20
43	V11-10%BN/v11-20%BN-au veil Vectran 1308/2	2463.78
51	V11 neat control	4754.72
52	V11 5% CSCA++	1783.90
53	V11 5% MMT	887.76
C51	4940 F-SX neat; 30 min 250/ 3 hr 205	28.75
C52	4940 F-Sx 5% CSCA++; 30 min 250/ 3 hr 205	4712.82
C53	4940 F-Sx 5% MMT; 30 min 250/ 3 hr 205	277.00
60	Epoxy/urethane (winder) neat	2631.14
61	winder resin 5% CSCA++	2128.46
62	winder resin 5% MMT	1843.34

NanoSonic shall Produce New Extruded Compounds such as Exfoliated Clays for Hoses and Nozzles and Evaluate H₂ Performance with our Partners

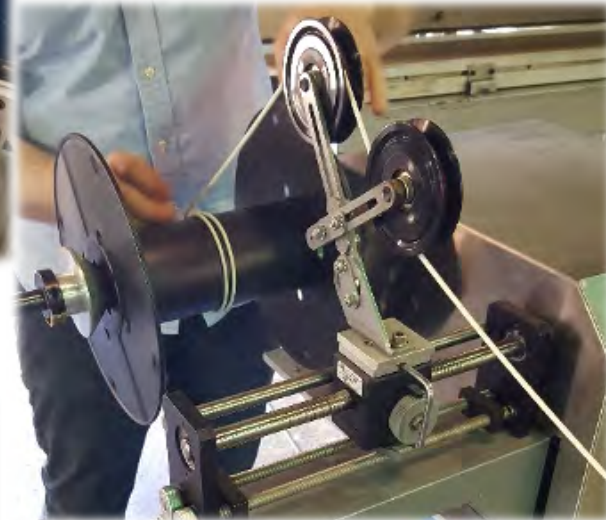
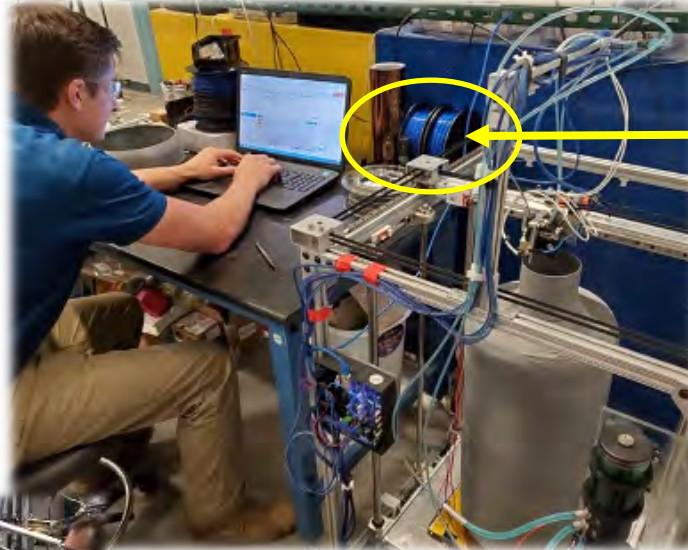
Ice Adhesion for New H₂ Materials Exposed to 27.6 MPa H₂ for 24 Hr at PNNL



Ice Adhesion Panel		CONTROLS		H2 27.6 MPa Soaked Samples	
		Avg. Ice Adhesion Strength (kPa)	Std. Dev.	Avg. Ice Adhesion Strength (kPa)	Std. Dev.
METALS	S-36-bare	108	19	106	15
	S-36-AI	38	7	17	13
	SS-36-bare	164	109	120	13
	SS-36-AI	43	4	40	14
	S-36-ICF-bare	125	23	174	79
	S-36-ICF-AI	54	11	39	15
POLYMERS	PEEK-36-bare	0	0	0	0
	N-PA66-36-bare	92	31	159	16
	B-PA66-36-bare	104	37	163	10
	PTFE-36-bare	4	5	9	1
	B-POM-36-bare	169	33	120	17

NanoSonic has Demonstrated Zero Ice Adhesion for 3D Print Polymers and a 67% Decrease in Ice Adhesion relative to Stainless Steel

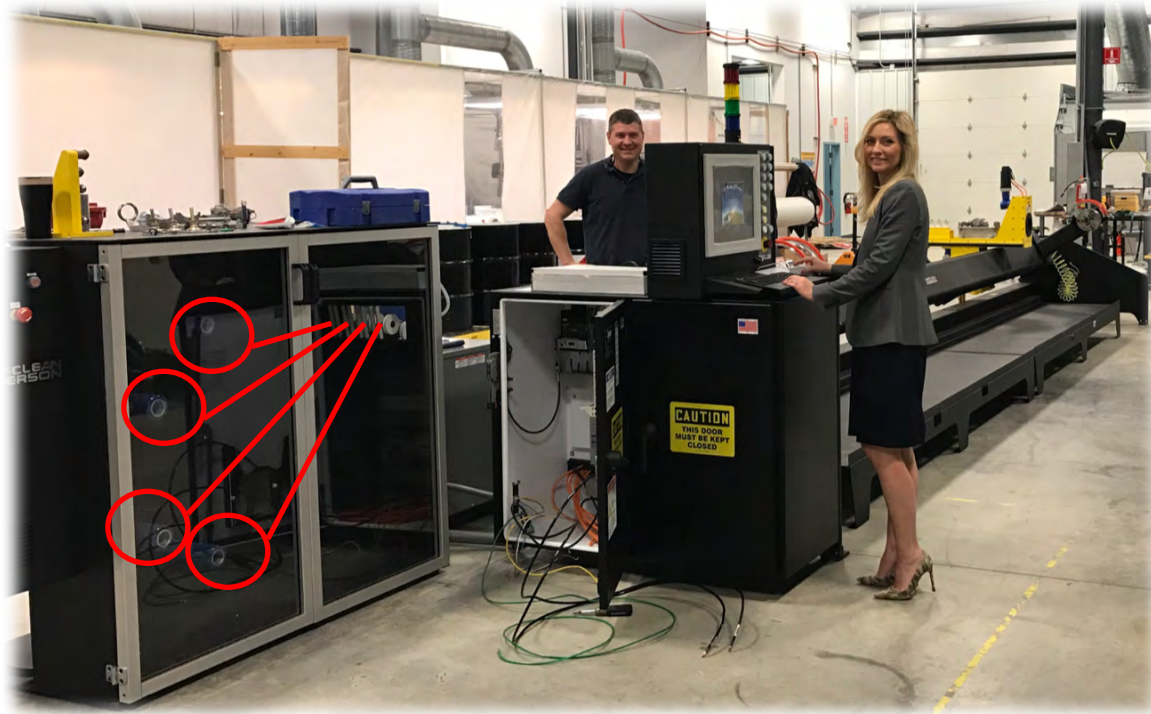
New 3D Printed H₂ Materials



NanoSonic has Demonstrated the Feasibility to Extrude New Low Ice Adhesion and Low H₂ Permeable Materials for 3D Printed Parts

Techno-economic 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 Testing at PNNL and NREL and by FCV Customers



Remaining Challenges and Barriers:

Fitted Hose with Commercial H₂ Hose Safety Adapters Qualification and Deployment



Current Cost for H70 Hose: \$600 with fittings

Projected Cost for Nozzle with IR Communication: ~ \$10k

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 -50 - 85 °C
- **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. 3D printed materials are under development with zero ice adhesion and low H₂ permeation for hose core and H₂ dispensing nozzles
- **Technical Accomplishments**:
 - Demonstrated hydrostatic burst strength > 43,300 psi
 - Demonstrated ZERO Ice Adhesion for new materials with abrasion resistance
 - 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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Phase II Integrators and Testing Facilities

