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PNNL-30887

High-Pressure, Low Temperature Composite Nozzles for Long-Term H2 Dispensing

CRADA 399 (Final Report)

January 2021

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U.S. DEPARTMENT OF ENERGY

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Cooperative Research and Development Agreement Final Report

Report Date: February 1, 2021

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Parties to the Agreement:

NanoSonic Inc.

CRADA number: 399

DE-FOA-0001941, CFDA Number: 81.049, Topic 10b

CRADA Title:

High-Pressure, Low Temperature Composite Nozzles for Long-Term H2 Dispensing

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DOE Program Office:

DOE HFTO

Joint Work Statement Funding Table showing DOE funding commitment (all $ in K):

Funding	Project Year 1	Project Year 2	Project Year 3	Project Year 4	Project Year 5	TOTALS
Government						
DOE	$30					$30
Other						
Total Govt.	$30					$30
Participant						
In-Kind						
Funds-In	$30					$30
FAC						
Total Participant	$30					$30
TOTAL CRADA Value	$60					$60

Executive Summary of CRADA Work:

NanoSonic was awarded a DOE Phase IIB SBIR program for the commercialization of an innovative metal-free polymer based H2 dispensing hose to make H2 an economically viable fuel alternative to gasoline. During the Phase I and Phase II base programs, NanoSonic's H2 hose demonstrated ultra-high hydrostatic burst strength values > 31,000 psi where failure occurred due to fitting slippage, rather than hose burst. Additionally, NanoSonic hoses survived >51,000 pressure impulse cycles at 12,000 psi over a thermal cycle of -40 °C to 85 °C. These hoses also failed due to fitting slippage. Thus, their Phase IIB program was centralized on the addition of a new fitting, polymer hose refinement, and validation of a complete hose and fitting system certified for use with H2. While NanoSonic has partnered with the National Renewable Energy Laboratory (NREL) to test their hose on their robotic H2 dispensing system, PNNL was seeking durability of material performance and testing with the H2 environment at the molecular level. The PNNL CRADA utilized their expertise in H2 polymer materials compatibility. PNNL tested NanoSonic noses and materials to understand material performance changes when subjected to hydrogen and extreme temperature environments. PNNL provided subambient hose burst testing with digital image correlation to provide composite strain and crimped hose connector analysis to help NanoSonic better understand how to improve the performance of the material and the hoses. PNNL also provided NanoSonic with in situ tribology of NanoSonic hose materials to determine if there was increased wear in their materials from frequent hose bending.

Importantly, the work conducted under the NanoSonic CRADA helped to increase the safety and reliability of NanoSonic H2 hoses while expanding their market for use of NanoSonic's low H2 permeation polymers and durable cryogenic composites to realize the H2@Scale objectives to reduce the cost of H2.

Summary of Research Results:

DMA Testing of Hose Materials

To get the mechanical aspect of the hose components, DMA TTS master curves were established for NanoSonic's H2 hose component A and B in air using a commercial DMA system (TA Instruments Q800). The results are shown in Figure 1. DMA can provide useful information on materials mechanical performance and its variation in response to environmental changes (e.g., temperature, force). The master curves obtained here can be used to set baseline for future testing under H2 environments.

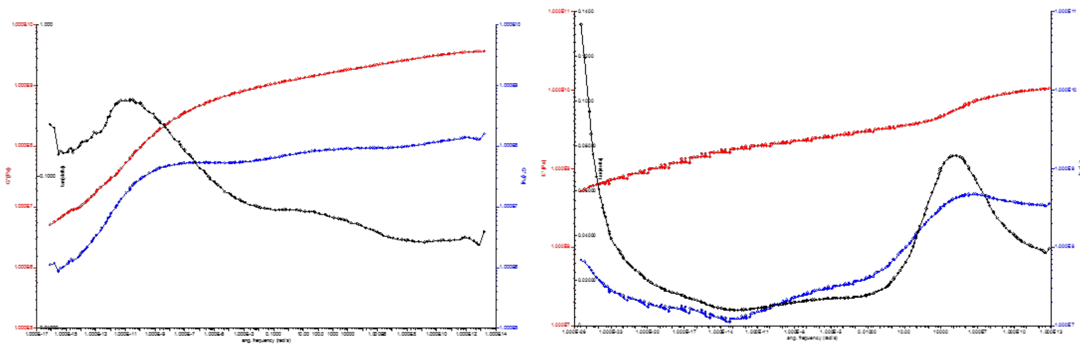

Figure 1. Time-temperature superposition master curves for NanoSonic's H2 hose component A (left) and B (right) in air.

Hose Material and Crimped Fitting Testing

To determine the failure mode of NanoSonic's hose samples, PNNL utilized their x-ray and digital image correlation (DIC) capabilities shown in Figure 2 and 3 respectively. X-ray analysis was also used to examine hose structures prior to testing (Figure 2). The x-ray imaging allowed NanoSonic to inspect the effects of crimping on the materials of construction. In Figure 2, the image on the right side with the highlighted circle, shows damage to the hose liner prior to testing. The hose liners also show different internal diameters due to the use of an insertion tool to expand the end of the hose and liner for inserting the internal fitting for crimping down onto the material shown on the interior of the images on the left side of Figure 2.

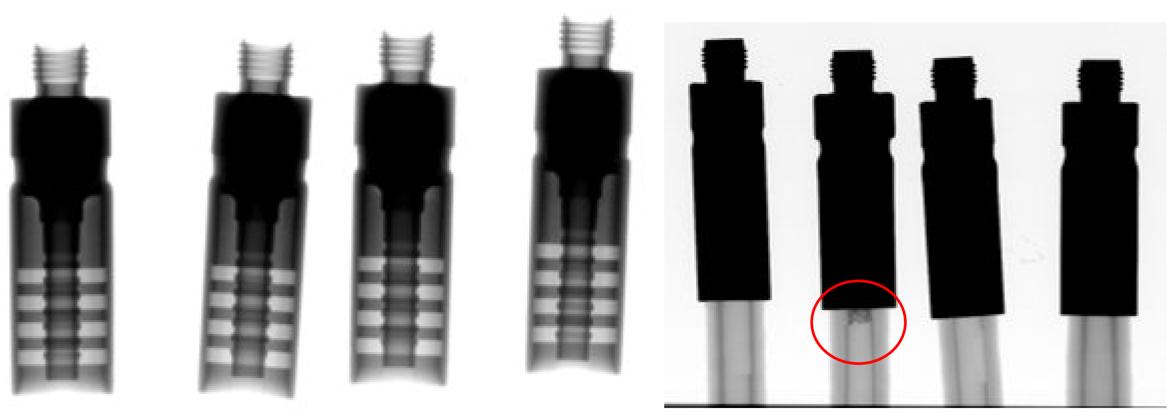

Figure 2. Pre-test X-ray analysis of Nanosonic's hose samples.

The DIC system was used to visualize strain movement in the hose during burst testing under extremely high pressure. The DIC is used to measure multi-axis strain at where structural failures were found to occur. NanoSonic fabricated hoses were pressure tested at room temperature and at -130°C. The room temperature hose tests burst at ~7,800 psi. The rupture was successfully captured as shown in Figure 4. During pressurization, a helical pattern of straining was observed across the specimen tube and a concentration of straining was found around the crimps where the catastrophic failure occurred. Further investigation of strain patterns from the winding profiles need to be better understood and how they relate to the failure mechanism in the context of how the hoses were fabricated or designed. The second room temperature hose didn't burst until ~14,200 psi. The different in materials and crimp stresses were the contributing differences between their burst pressures. Crimp hose ends that slide off the ends were shear failures in the material or reduction in crimp force as shown in the far-right image in Figure 4.

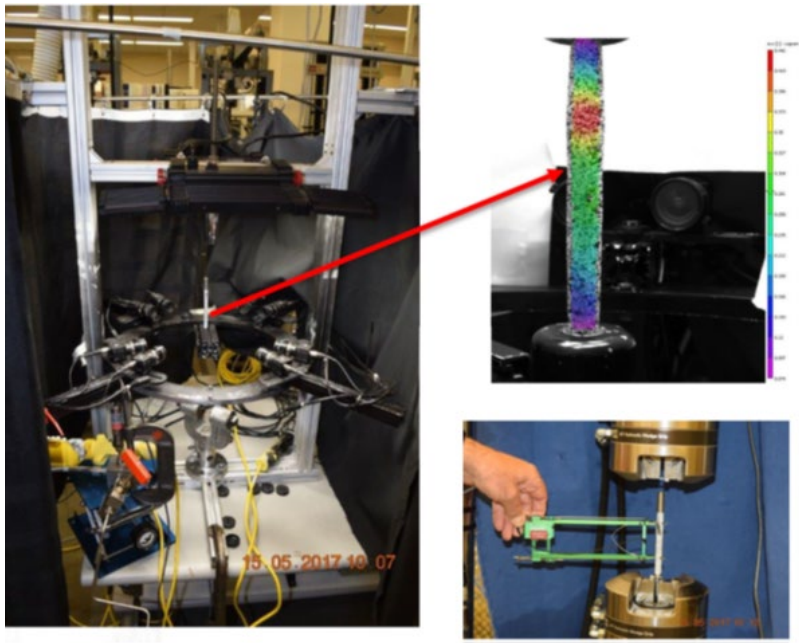

Figure 3. PNNL's digital image correlation capabilities for multi-axis strain testing.

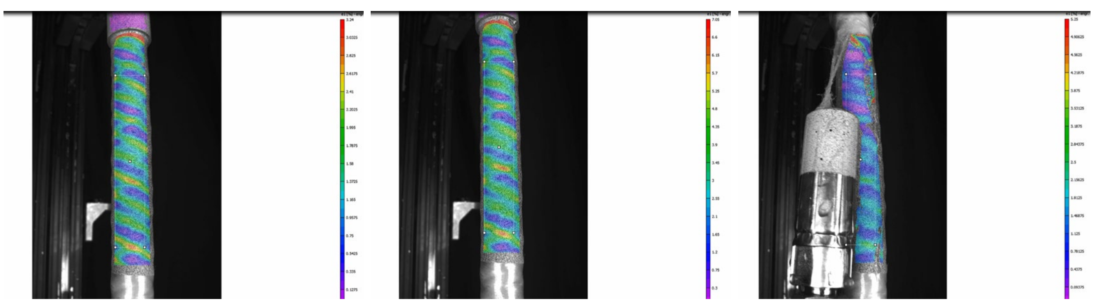

Figure 4. Pressure test of Nanosonic's tubes with DIC technique. Strain contour of NanoSonics hose at ~7,000 psi (left), near to burst pressure (middle), and immediately after burst (right).

A third hose design and material set was burst (water) tested at room temperature with both ends video recorded by our DIC system simultaneously. The separation between the fitting and tube happened above 16,000 psi. Water was observed seeping out from the tube near the crimp edges and between the fiber winding pattern before final failure. The crimp did not fail on these tested hose series

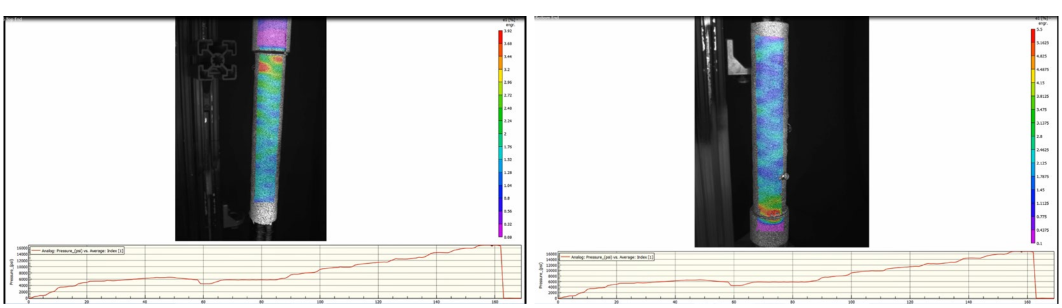

Figure 5. Burst test of NanoSonic hose: top end (left) and bottom end (right) based on the alignment in the test system.

Lastly, in order to determine the performance of the hoses at cryogenic temperatures, a temperature-controlled burst test system was employed with DIC set up to measure multi-axis strain. Selected hoses were tested at -130 °C. Some of the hose connections were found leaking around the threaded fitting and required additional work to stop the leakage. There were two hoses that were tested and were able to hold pressures up to 30,000 psi with a small amount of strain in those specimens. In general, the strain in the tube portion was around 1% with spots up to 2%. After pressure was released from the tube, there was approximately 0.1% residual strains in the tube. As a result, the major strains in the tube are almost entirely in the axial direction. These low temperature tested hoses did not burst or fail in the fittings. This indicates a higher modulus and strength resin system could improve the hose crimp performance and increase the burst pressures at room temperature.

Tribology of hose liner material

As previous findings showed fitting slippage that causes the hoses to fail, it becomes quite important to understand the change in tribological performance of the hose materials under high-pressure hydrogen and cyclic behavior. NanoSonic provided PNNL 18 different tube materials which were subsequently tested under ambient air and 27.6 MPa high-pressure hydrogen (4000 psi) using PNNL's unique in-situ tribology test system. Frictional force and penetration depth were recorded to estimate coefficient of friction and specific wear rate. The results shown in Figure 6 indicate that the Coefficient of Friction (CoF) increases in most materials when high pressure hydrogen is present with the exception of material 3. Materials 10, 3, C20, C31, and C36 showed varying degrees of reduced wear rate while other materials had an increase in wear rate. It is important to note that coefficient of friction and wear rate are not proportionally related in many cases. Among all 18 test materials, three materials were found to have the largest change in CoF due to the introduction of high-pressure hydrogen. One material showed a reduced CoF in the hydrogen environment which could result in fitting slippage. Further, the wear rate of the one material with the reduced CoF remains identical under ambient air and high-pressure hydrogen. This suggests high pressure hydrogen has minimal impact on the tribological performance of the reduced CoF material

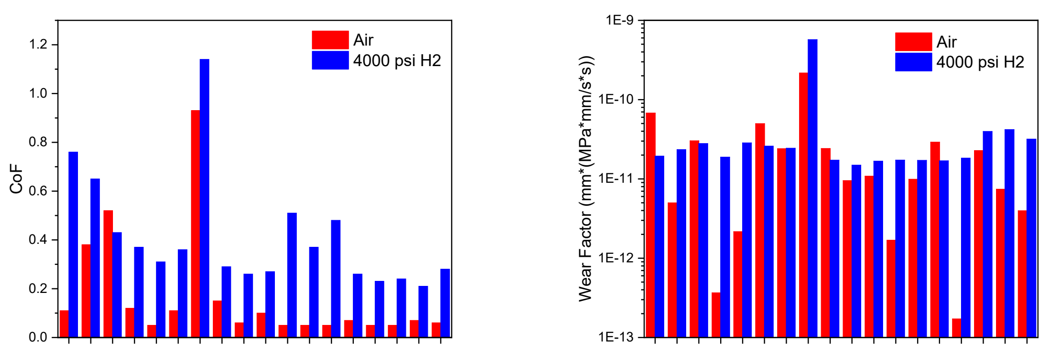

Figure 6. Coefficient of friction (left) and specific wear factor (right) of Nanosonic's tube materials under ambient air and high-pressure hydrogen.

Future Work:

- Perform DMA in hydrogen to compare with the master curves attained in air for the hoses.

- Use advanced imaging to examine the failure spots in the hoses. This can help gain knowledge about what causes the failure from a structural standpoint.
- Develop better design to mitigate strain concentration at the crimp in a tube.
- Change material parameters for increased shear performance at the fittings
- Crimp fitting test pull out
- Address thermal expansion difference between the fitting and the tube.
- Create models to corelate with experimental results and predict performance at different running parameters.

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