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Inexpensive Delivery of Cold Hydrogen in Glass Fiber Composite Pressure Vessels

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Abstract

We are proposing to minimize hydrogen delivery cost through utilization of glass fiber tube trailers at cold temperature (200 K) and high-pressure (70 MPa) to produce a synergistic combination of container characteristics with properties of hydrogen gas: (1) hydrogen cooled to 200 K is ~35% more compact for a small increase in theoretical storage energy (exergy); and (2) these cold temperatures (200 K) strengthen glass fibers by as much as 50%, expanding weight-limited trailer capacity without the use of much more costly carbon fiber composite vessels.

Analyses based on US Department of Energy H2A cost and efficiency parameters and economic methodology indicates the potential for hydrogen delivery costs below \$1/kg H₂. Dispensing cold hydrogen also allows rapid refueling without overtemperatures and overpressures which are typically as high as 25%, simplifying automotive vessel design and improving safety while potentially reducing vessel weight and cost.

We have conducted preliminary experiments confirming an increase in strength of glass fibers at cryogenic temperatures. Further experiments are necessary for more comprehensive evaluation of glass fiber strengthening as a function of temperature and environmental conditions.

Introduction

Today's hydrogen delivery options are energetically and economically expensive [1]:

- As a cryogenic liquid, hydrogen is compact, but evaporates very rapidly, requiring high performance insulation and care during transfers. In addition there are substantial electricity requirements for liquefaction and conversion to *parahydrogen*.
- As room temperature gas, hydrogen occupies substantial volume even at high pressures, leading to relatively heavy containers, and therefore low capacity delivery trailers.
- Absorbents and adsorbents (hydrides and carbon structures) would reduce the pressure and volume of a delivery trailer, but trades the weight of absorbent materials and thermal management (e.g. heat exchangers) for the weight of pressure vessel walls.
- Chemical storage media often require 2-way transportation by truck. Reprocessing is expensive and energy intensive; and chemical carriers are often toxic, polluting, heavy, or require high temperatures for dehydrogenation.

Substantial reduction in delivery cost and energy appears possible with development of advanced pressure vessels and a broadened range of thermodynamic conditions under which hydrogen is trucked and delivered (Figure 1). Here we report the results of analyzing these approaches using the Department of Energy's (DOE) H2A infrastructure analysis tool [2] applied to the cost of hydrogen truck delivery. These cost savings are based on the compounding of four factors relative to conventional tube trailers: volumetric efficiency, increased storage pressure, reduced temperature, and higher strength of glass fiber at low temperature. Based on these results, we suggest hydrogen delivery by truck with trailers carrying hydrogen gas at pressures as high as 70 MPa, cooled to approximately 200 Kelvin (-73° C) in glass fiber vessels.

Analysis

Our approach has been to use H2A [2] to estimate the costs of hydrogen delivery by truck. We feed H2A the scalars resulting from candidate designs for pressure vessels. These designs embody our analyses of the thermodynamic properties of hydrogen, choice of structural materials, optimization of operating pressure and temperature, and onboard storage implications. These candidates allowed us to find favorable synergies aimed at achieving substantial rather than incremental *overall* cost reductions. We developed a range of hydrogen storage and vessel design parameters, which form the technical basis for our cost estimates using H2A delivery cost models. Our general strategy has been to choose delivery and trailer storage parameters that *simultaneously* reduce cost components rather than optimize detailed tradeoffs between cost components, since the first approach is more likely to produce a robust result for a variety of delivery logistics scenarios.

The analysis is based on the following operational and economic assumptions:

- 50 km one-way (100 km round trip) delivery distance from production site to fueling station.
- Trailer drop-off time determined by trailer capacity and station scale (throughput in kg-H₂/day).
- All trailers sized to 1300 kg H₂ capacity (1000 kg deliverable), except for metallic compressed hydrogen trailers (300 kg deliverable).
- We use real hydrogen thermodynamic and Pressure-Volume-Temperature (PVT) properties from the NIST database [3].
- All trailers store hydrogen at 70 MPa, except for metallic compressed H₂ trailers (18 MPa).
- Trailers are designed for a safety factor of 2.25 (burst pressure 157.5 MPa in 70 MPa vessels).
- Hydrogen is delivered to stations at either 200 or 300 Kelvin.
- Analysis is consistent with H2A methodology [2]. H2A financial parameters are used for everything except trailer and refrigerator cost (not available in H2A database).
- Electricity cost at \$0.08/kWh for hydrogen compression and/or cooling.
- Analyze costs as a function of station demand from 70 kg H₂/day to 1000 kg H₂/day.

- Analysis includes the driver cost in addition to the capital and energy costs of hydrogen compression, hydrogen refrigeration (if necessary), delivery by truck, and dispensing from the truck at the fueling station.
- Analysis does not include other costs associated with the rest of the delivery infrastructure such as storage at the terminal and compression, cascade, and chilling at the refueling site that may be necessary for fast refueling.

Based on our economic assumptions, we obtain the results of Figure 2. The figure shows total cost in thick lines and driver labor and truck cost in thin lines. The difference between the thick and thin lines is the cost of compression and cooling, including capital and energy cost.

According to H2A, liquefaction costs over \$2/kg – too expensive to appear on the much smaller scale of the vertical axis in Figure 2. Large liquefaction plants or technological advances may considerably reduce liquefaction costs [4], and therefore liquid hydrogen delivery warrants further research due to its high storage density and ease of dispensing.

As Figure 2 shows, delivery cost quickly drops as the fueling station demand increases. High hydrogen demand permits fast cycling of the trucks (i.e., a 1000 kg tube trailer can be filled and emptied every day if the station throughput is 1000 kg/day), reducing the capital cost contribution to the total cost of H₂ delivery.

According to Figure 2, the cost for hydrogen delivery in a metallic tube trailer cannot be reduced below ~\$1.50/kg H₂ due to the low capacity of the trailer, which magnifies the impact of labor cost. Trailer cost is \$165,000, and compression cost (to 18 MPa) is 41 cents/kg H₂ including capital as well as energy cost (from H2A [2]).

Increasing the delivery pressure to 70 MPa reduces labor cost per kg of H₂ while increasing the trailer capital cost. The overall balance in terms of delivery cost is positive. Carbon composite tanks (at \$430,000 per trailer) delivering hydrogen into a large (1000 kg H₂/day) fueling station have the potential to reduce delivery cost to less than \$1/kg (at \$0.54/kg for compression). However, fast refueling with ambient temperature gaseous hydrogen is challenging due to the compression heating and overpressurization (typically 25%) necessary for a complete fill [1]. Chilling at the station may be necessary, introducing additional costs (not included in this analysis).

We can look for reduction in delivery cost by considering the whole hydrogen phase diagram (Figure 1). Delivering hydrogen at 200 Kelvin can increase the density of hydrogen by ~35% over ambient temperature delivery for a small increase in theoretical storage energy (exergy) requirement (0.34 kWh/kg). Operating at this unusual condition may be expected to greatly increase trailer capital cost. However, we have identified a synergy between inexpensive glass fiber and low temperature operation that holds promise to deliver minimum cost high performance delivery trucks: legacy data [5] indicates that glass fiber strengthens ~50% when cooled from 300 Kelvin to 200 Kelvin, expanding weight limited trailer capacity and reducing capital expense. Inexpensive glass fiber (at \$6/kg compared to \$24/kg for stronger carbon fiber) has unequalled tensile strength per dollar when cold, resulting in a wound pressure vessel trailer that can be built for \$200,000. Savings in capital cost have to be balanced against the added cost

of refrigeration, estimated at 16 cents (8 cents for capital and 8 cents for electricity in a 30% Carnot efficient refrigerator).

Figure 2 shows the cost of hydrogen delivery in light of this new concept. The thin line marked “cold glass fiber” shows the cost of the truck and driver. The thick line shows total delivery cost including compression (54 cents/kg) and the additional cost of cooling hydrogen to 200 K in a central plant (16 cents/kg). The figure shows that cold glass fiber vessels deliver the minimum possible cost, below \$1/kg for delivery at stations that dispense 500 kg/day or more. Delivery cost is very insensitive to the hydrogen demand at the station due to the low capital cost of the trailer, enabling inexpensive hydrogen delivery even at small stations.

Very importantly, an extra synergy exists which has not been captured in the above figures: delivering 200 Kelvin compressed hydrogen avoids overheating and overpressurizing of automobile storage tanks, increasing the fill speed and potentially reducing the cost of automotive storage. Vessel designs can be simplified if pressure and temperature never exceed the nominal vessel rating.

Today’s automotive pressure vessels are designed for 2.25 safety factor – defined as the burst pressure divided by the maximum service pressure. However, these vessels are routinely filled at 125% of the service pressure to compensate for the density loss due to heating during fueling. The *net* safety factor is therefore $2.25/1.25 = 1.8$. Dispensing cold (200 K) hydrogen eliminates the need for overpressurizing, because the chilled gas ends up at near ambient temperature (at 326 K vs. 465 K for 300 K dispensing [3]) once it stagnates inside the tank. Vessels consistently filled with cold hydrogen may therefore be designed with 1.8 safety factor to provide the same pressure margin. A 70 MPa vessel would therefore be built for 126 MPa burst pressure (70×1.8) instead of 157.5 MPa (70×2.25). Considering that fiber is the most expensive pressure vessel component, reducing the burst pressure by 25% would reduce vessel cost by roughly 20% [6].

Considering an automotive pressure vessel that stores 5 kg H₂, costs \$2,800 (at \$27/kWh for 70 MPa vessel [6]), and is installed in an efficient 100 km/kg H₂ car driven 200,000 km during its life, the 20% savings in vessel cost (\$900) is spread over 2,000 kg H₂ consumed over the life of the vehicle. Therefore, the savings in vessel cost amounts to roughly 45 cents/kg H₂. Overpressurization avoidance and the consequent reduction in automotive vessel cost may therefore be the most significant advantage of low temperature compressed hydrogen delivery.

Concept Feasibility

Today’s fiber wound pressure vessels are not specifically designed for low temperature operation that may introduce thermal stresses and reduce cycle life. Warming of the vessel due to heat transfer from the environment is yet another issue, possibly leading to venting losses if the vessel exceeds its service pressure. Warming also weakens the vessel, because the concept depends on cold temperature for extra strength. However, the authors’ ongoing work on hydrogen storage in cryogenic capable pressure vessels [7-9] has demonstrated the many advantages of low temperature and high-pressure operation for automotive on-board storage. While the approach for hydrogen storage focuses on maximizing packaging density instead of

minimizing cost, the advantages of the approach apply to delivery vessels as well as to automotive storage:

1. *Conventional pressure vessels can operate at cold temperature with no structural damage.* We have cycle tested pressure vessels of different types and materials at cold temperature (down to 20 K) and high pressure multiple times (thousands) without any damage to the vessel. Finite element analysis indicates that no significant plastic deformation occurs after the first few cycles, and therefore vessels can be expected to have a long life even at cryogenic conditions [7].
2. *Cryogenic capable pressure vessels cool down when hydrogen is extracted.* The first law of thermodynamics indicates that a vessel cools down when hydrogen is extracted. This cooling effect is equal to the difference between enthalpy and internal energy, and at 200 K and 10,000 psi is equal to 0.4 kWh per kg of hydrogen extracted [3]. Therefore, dispensing just a few kilograms of hydrogen per hour is enough to compensate for heat transfer into the vessel, greatly reducing the need for high performance insulation. Consequently, delivery vessels in regular service are likely to remain cold *forever* after the initial fill cycle, simplifying the fill process and reducing energy consumption.
3. *Cryogenic capable pressure vessels have low sensitivity to heat transfer.* Automotive cryogenic compatible pressure vessels have ~10 times less sensitivity to heat transfer from the environment than conventional low-pressure liquid hydrogen tanks [7]. Large tanks for hydrogen delivery are even less sensitive to heat transfer due to their low area to volume ratio, simplifying the insulation problem even further and permitting the use of inexpensive and/or thin insulation. Insensitivity to heat transfer protects from venting losses or vessel weakening due to warming during long periods of inactivity (e.g., when the delivery truck is stranded due to mechanical failure or accident).

Experiments

Costs in Figure 2 assume that glass fiber strengthens by 50% when cooled down to 200 K. This assumption is supported by legacy research [5]. Whether minimum-cost, cold, glass fiber composite vessels are optimal or not could depend on the actual tensile strength versus temperature curve of the best composite materials, as well as on the capital and operating costs of refrigeration. We do not know if the legacy data apply to our currently available composite materials. Our current front-running candidate glass fiber for delivery truck applications – a uniform grade of volcanic basalt glass that might result in the best hydrogen delivery economics – was not available in the twentieth century.

The legacy research data on cold glass strength is characterized by its lack of repeatability. Hypotheses used to explain plate glass strength phenomena in the 1960s attribute a variety of detrimental effects on strength to very low levels of humidity. Other polar gas environments (e.g. ammonia) produced even stronger detrimental effects on glass strength. This leaves open the question of the presence of ambient contaminants in composites resulting from incomplete matrix polymerization. There is no shortage of possible explanations for why the legacy data do not agree, and every reason to avoid trying to repeat their unrepeatability.

Testing ambient-dependent cold glass strength demands a comprehensive experimental plan to evaluate fiber tensile strength at a wide range of reduced temperatures and atmospheric compositions. Capability for baking out samples is also necessary to remove the moisture (and possible contaminants from manufacture) already bound in microscopic defects of the fiber. While we are building such an apparatus capable of conducting extensive cold temperature experiments under controlled environments, we have conducted preliminary proof-of-concept tension tests on glass fiber samples immersed in liquid nitrogen (LN). These LN-immersed tests were intended to suffice for a preliminary confirmation of the strength gains afforded by cold temperature operation.

Figure 3 shows the experimental apparatus and the main steps in the LN-immersed testing process. Strength measuring experiments are conducted in a tensile test machine (upper left). Fiber samples (pultruded rectangular rods of basalt fiber composite) are carefully prepared for the tension tests by gluing them into metallic fixtures (upper center) that can be instrumented with a thermocouple (upper right), then mounted inside an LN Dewar that has been incorporated into the load path of the tensile test machine (bottom left). The Dewar is then filled with LN (bottom center). The test starts when the fiber sample and fixture reach thermal equilibrium with the liquid nitrogen. While environmental conditions were not deliberately controlled, room temperatures were stable within $\sim 1^\circ\text{C}$.

Preparing suitable test fixtures (Figure 4) is a technical challenge due to the high strength of the basalt composite rods relative to shear strength in the epoxy. The best structural epoxy was used to bond the rods, but remains roughly two orders of magnitude weaker than their composite. In the first sequence of experiments the epoxy bonds failed at lower tensile loads than those that could fail the fiber. To improve these preliminary fixture designs, we conducted finite element analysis of the alternative specimen geometries, and identified the virtues of a conical fixture cross section (Figure 5, top) that considerably reduces stress concentration at the tip (Figure 5, middle) with respect to straight cross section designs.

Subsequent design of experiments determined that thickening the epoxy layer (increasing the clearance between the fiber and the fixture) would contribute most to strengthening the bond between composite rod and fixture. Fixture optimization based on finite element analysis and design of experiments succeeded in strengthening the bond sufficiently for basalt composite rods to fail in tension somewhere between the fixtures (Figure 5, bottom). This is our most desirable failure location, allowing us to measure tensile failure in the composite (rather than the fixtures).

Figure 6 shows the experimental results for tensile testing of fixtured specimens of pultruded basalt fiber composite. The figure shows tension in kilograms plotted as a function of elongation in millimeters for four specimens: two at ambient temperature and two at liquid nitrogen temperature (77 K). The figure reveals a sawtooth pattern for all the fixtures, caused by the composites' failure mode. As the basalt rod is stretched, it reaches a point where the weakest fibers in the cross sectional area fail. The tension then drops suddenly because only a smaller fraction of the original total cross sectional area remains available to hold the force. Elongation does not change at the instant that some fibers break. With further elongation the force increases

again until the next weakest fibers fail. This progressive failure process continues until finally the strongest fibers fail, reducing the force to zero at some ultimate elongation.

The data plotted in Figure 6 can be further analyzed to produce the robust tensile strength information shown in Figure 7. Analysis begins with the first sawtooth profile in Figure 6. We calculate the tensile stress that broke the weakest fiber (by dividing the failure tension by the cross sectional area) and the fraction of the fiber that fails at this stress (by dividing the tension after failure by the tension before failure). Subsequent sawteeth can then be analyzed to determine the strength of progressively stronger fibers and the fraction of the area that fails at any particular stress. This analysis procedure concludes with the results plotted in Figure 7, where tensile stress at individual fiber failure replaces tensile load force on the entire rod and percentage of fibers remaining in the load path replaces elongation. The plot of Figure 7 (known as a Weibull Cumulative Failure plot) allows direct comparison of the tensile strength of different specimens of fiber when tested at ambient and LN temperatures.

There is no need to plot more than roughly half of the fibers failing, since the accuracy of the analysis drops due to lack of stress uniformity when the remaining cross sectional area shrinks, and only the first major portions of fiber failing matter. Figure 7 indicates that operation at liquid nitrogen temperature does indeed strengthen the basalt fiber composite we tested by ~40% compared to operation at ambient temperature. This conclusion is based on the average strength of two specimens of the same composite broken at each temperature, where each of the specimens tested at each temperature was fixtured in a different metal. The strength results from the different fixture metals (4140 with steel-like thermal expansion, and Invar 36 with almost no thermal expansion) agreed with each other within ~5 percent, which is roughly the error we anticipated due to fixturing and composite manufacturing variations.

Strength results can be seen in Figure 7 as the 'plateau' stresses each curve achieves just below the 'cliff' on the right of each curve, whereat a significant fraction of the fiber failed. Failures to the left of this plateau are artifacts of fixturing, whereas small fractions of fiber fail due to stress concentrations as particular fiber bundles on the surface of the rod emerge from the epoxy bonding them into their fixtures. Other small area fractions to the left of the plateau fail at the periphery of the reduced area section shown at the bottom of Figure 5. Stresses as large as or larger than these plateau stress values are available to provide tensile strength from every fiber in well-built pressure vessel designs, which avoid localized stress concentrations implicit in fixtures or reduced area sections. The consistent strength values analyzed from these recent basalt composite experiments therefore provide preliminary confirmation of glass fiber strengthening with cold temperature operation.

Further strengthening may occur from baking the fiber or composite in vacuum to eliminate moisture effects, and/or from leaving specimens in liquid nitrogen for a longer time sufficient to equilibrate the concentration of water molecules at defects within the fiber to the vapor pressure of water at a particular temperature. Little thought was given to mass diffusion equilibrium when thermal equilibrium was declared based on thermocouple measurements in these proof-of-concept experiments. More thorough experiments based on solid-state refrigerators are under construction to enable exact and stable long duration control at the cold temperatures of interest (from ~140 to 300 K). Extensive testing is necessary to perform a

comprehensive evaluation of fiber strengthening as a function of temperature and environmental conditions for several of the best candidate glass fiber materials.

Conclusions

Glass fiber pressure vessels operating at low temperature (200 K) and high pressure (70 MPa) minimize delivery cost through a synergistic optimization of hydrogen properties and fiber characteristics:

1. *Optimization of operating pressure and temperature:* Today's hydrogen delivery technologies (compressed and liquid) are restricted to single points at extremes of the hydrogen phase diagram. Hydrogen delivery cost can be minimized by exploring the entire phase diagram and finding pressures and temperatures that result in high storage density without the heavy thermodynamic penalty of hydrogen liquefaction. Cold high-pressure hydrogen (~200 K and up to 10,000 psi) appears most promising. Pressure vessel operation at low temperature high-pressure regimes has recently been demonstrated in two LLNL experimental hydrogen vehicles.
2. *Use of inexpensive glass fiber:* Glass fiber is typically considered an inexpensive low performance alternative to carbon fiber. However, glass fiber is synergistic with low temperature operation, strengthening ~50% as it is cooled down from 300 K to 200 K [1]. Cold glass fiber delivers unequalled performance per unit of cost, increasing weight-limited trailer capacity and reducing capital expense.

Cold, compressed hydrogen vessels deliver high-density hydrogen (~50 kg/m³) and high truck capacity (~1100 kg) without the high liquefaction energy, ortho-para conversion or evaporative losses of liquid hydrogen. Insulation performance is a much simpler problem to solve when delivering 200 K high-pressure hydrogen. This approach enables high speed refueling and high capacity delivery in trucks, enabling affordable delivery. The approach is environmentally benign, with low energy input and no need for expensive 2-way transportation or reprocessing.

Delivering 200 K hydrogen increases the density by 35% at a low energy cost and enables fast refueling without vessel overheating and overpressurization. Avoiding the need to exceed the rated pressure and temperature of the vessel during fast fill is an important advantage of this approach that may be more valuable to the driver than its reduction in delivery cost.

Proof of concept experiments show promise for engineering the strengthening of glass fiber composites operating at low temperature. Further experimentation at low temperature will determine the extent of the strengthening as a function of temperature, ambient humidity, and pre-testing bakeout.

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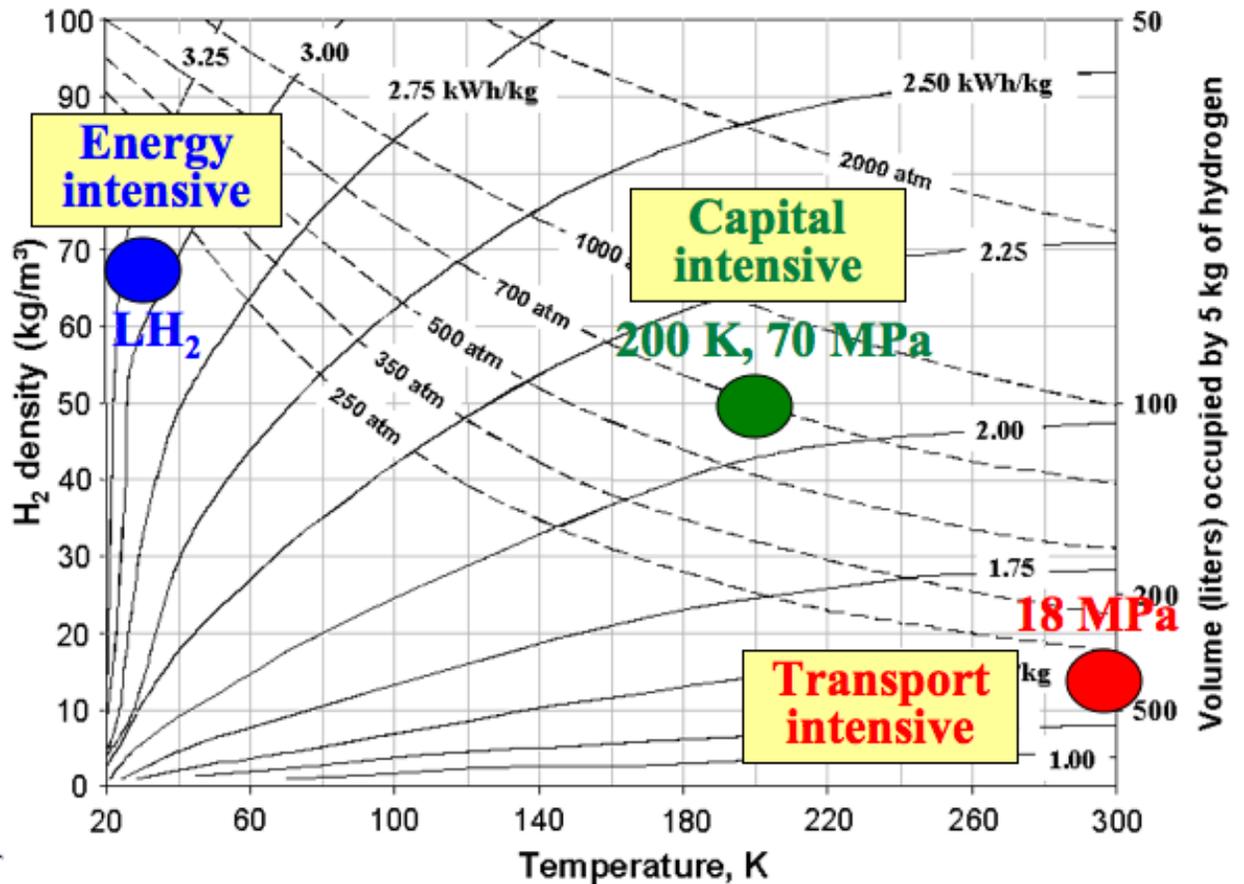


Figure 1. Commercial hydrogen delivery technologies occupy the extremes of this phase diagram. Hydrogen is often delivered as a compressed gas (red dot) at ambient temperature (horizontal axis), high pressure (dotted lines), and relatively low density (vertical axis). Hydrogen is delivered at much higher density as a cryogenic liquid (blue dot) with higher energetic cost (solid lines indicate the theoretical minimum work, also known as thermomechanical exergy necessary to densify hydrogen). Analyzing the entire phase diagram offers the possibility of finding operating conditions (such as 200 K and 10,000 psi) that may offer a favorable trade-off between the high transport cost of compressed hydrogen and the high-energy cost of hydrogen liquefaction. The challenge is to operate in this region while keeping capital costs under control.

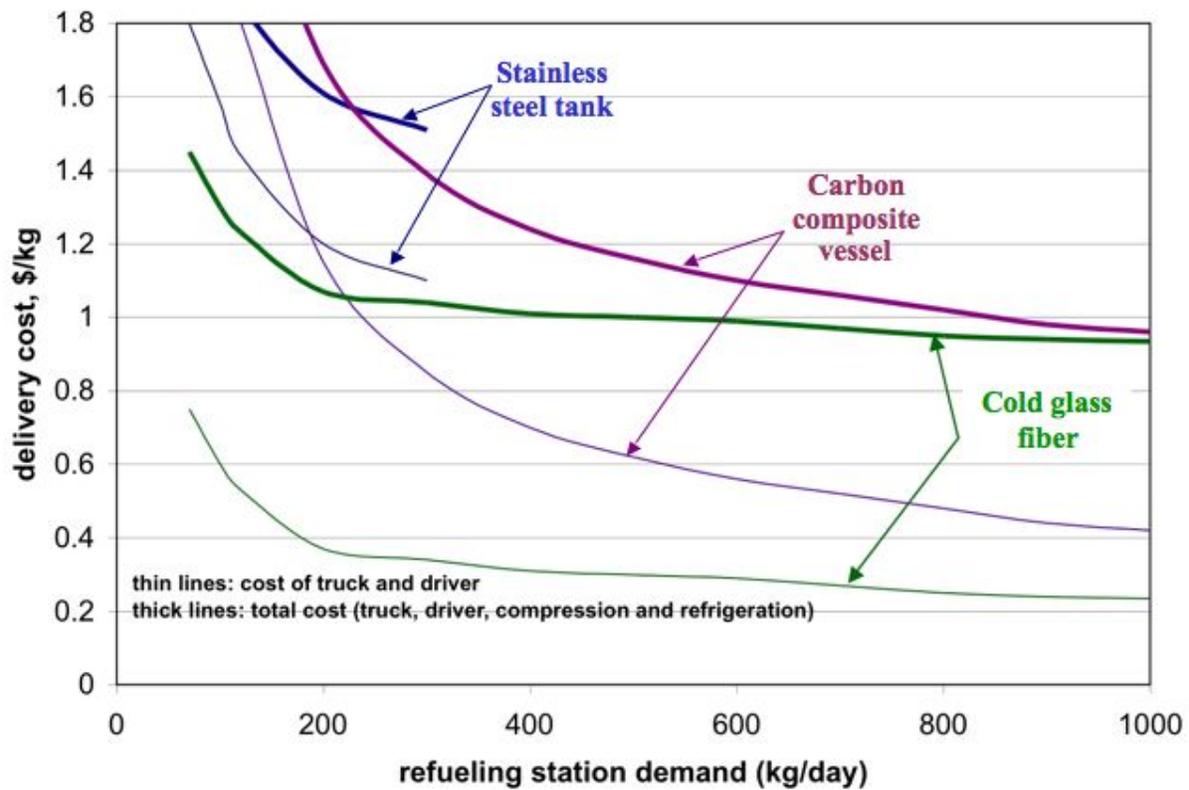


Figure 2. Cost of hydrogen delivery for metallic tube trailers, for carbon composite tanks, and for cold glass fiber tanks (200 K and 10,000 psi), as a function of refueling station demand. Thin lines show truck and driver cost, and thick lines show total cost, which also includes the cost of compression and (possibly) refrigeration (both energy and capital). Costs in Figure 2 do not include in-station compression, cascading and cooling that may be necessary for practical dispensing.



Figure 3. Experimental apparatus and the main steps in the experimental process. The experiment is conducted in a tensile test machine (upper left). Fiber samples (basalt rods) are carefully prepared for the tension tests by gluing them into metallic fixtures (upper center) that can be instrumented with a thermocouple (upper right) and mounted inside a Dewar into the tensile test machine (bottom left). The Dewar is then filled with liquid nitrogen (bottom center). The test starts a few minutes after the liquid nitrogen stops boiling (bottom right), indicating thermal equilibrium within the fiber sample and fixture.

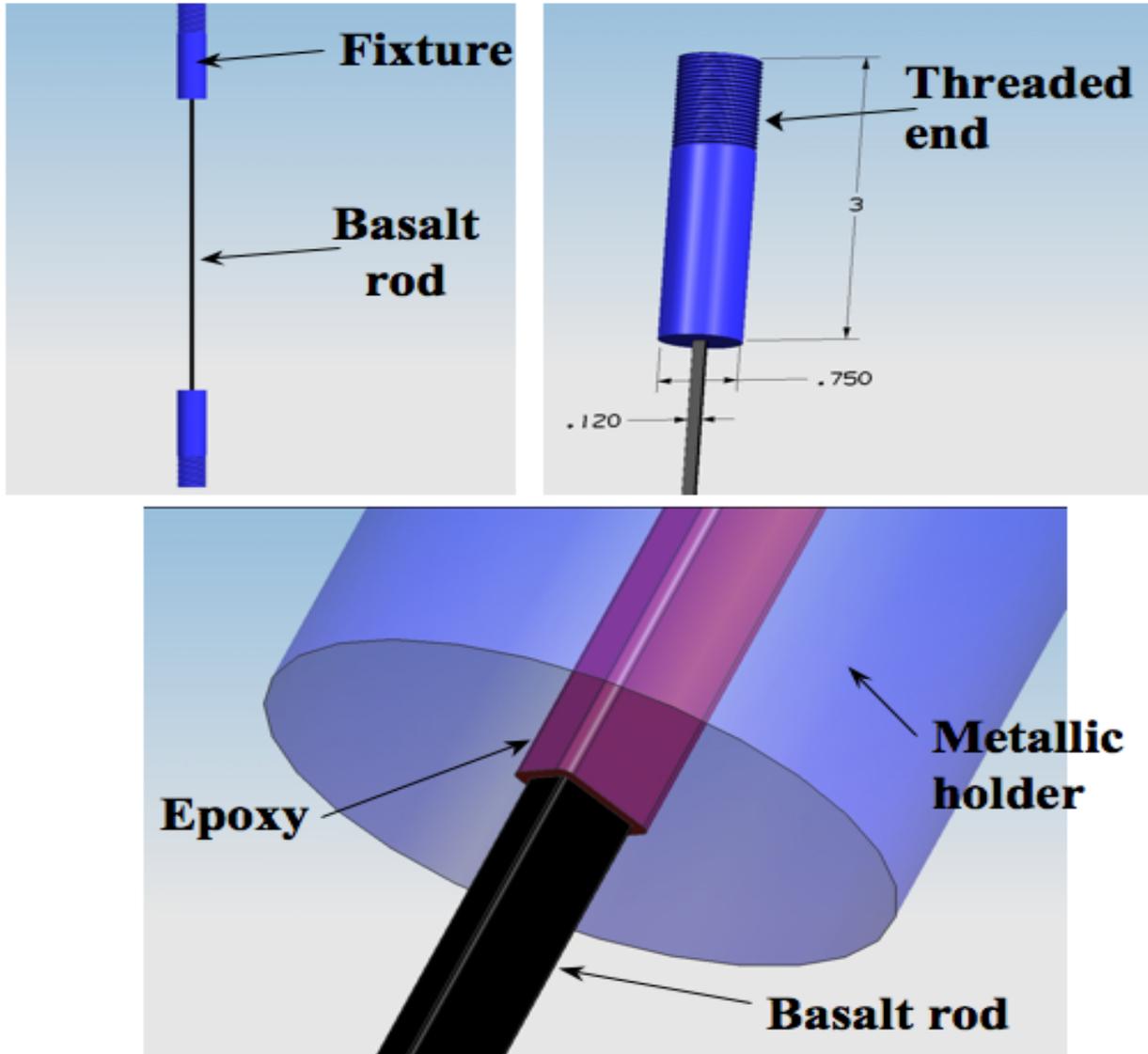


Figure 4. Testing fixtures are made by gluing basalt rods to metallic holders for subsequent insertion in the tensile test machine. The figure shows the overall fixture geometry (upper left), a detail of the metallic holder (upper right) and a detail of the bonding between the holder and the basalt rod.

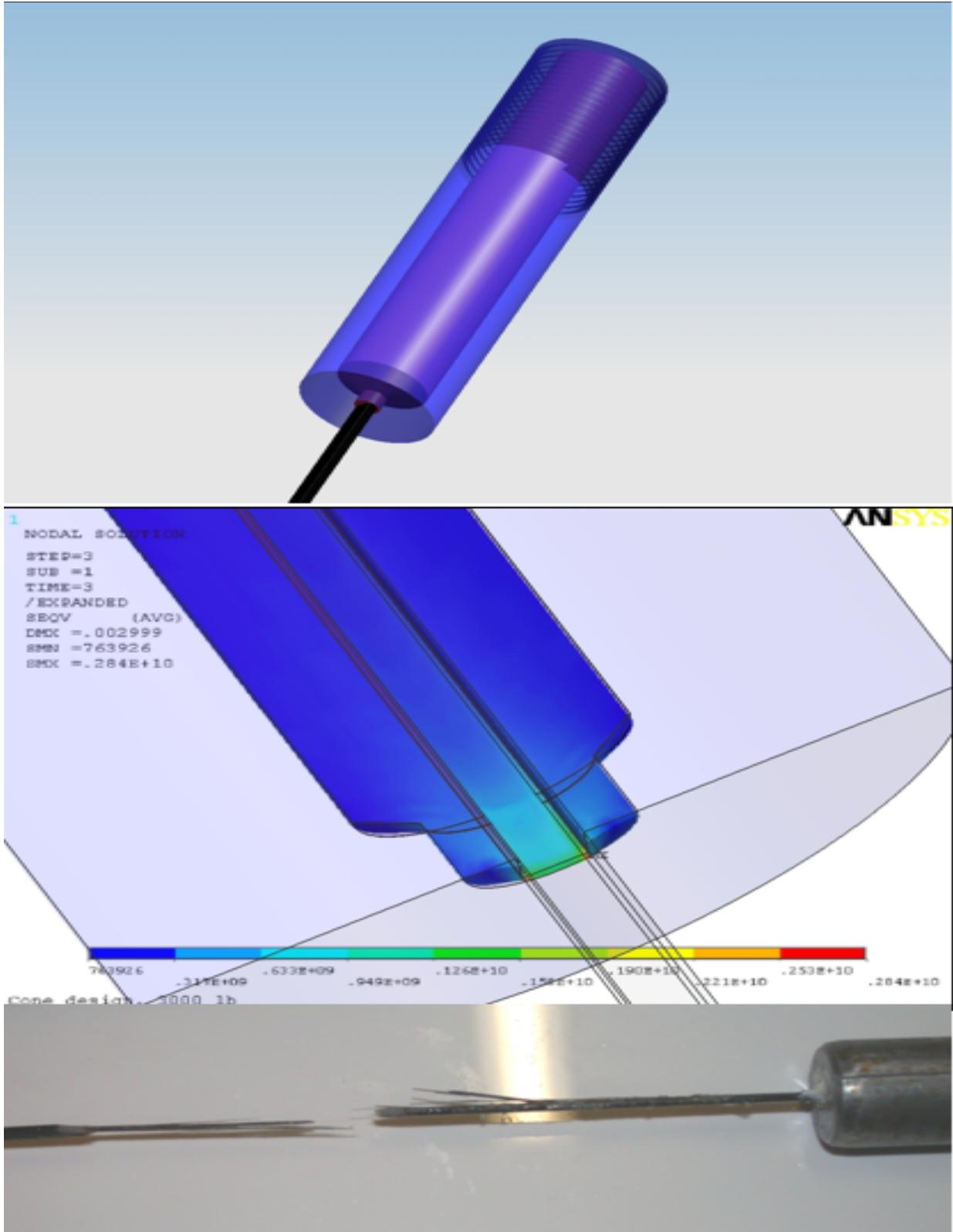


Figure 5. Improved metallic holder design that includes a reduced cross sectional area at the tip (top). This profile minimizes stress concentration at the tip (middle), therefore succeeding in holding the basalt rod during the tensile tests (bottom).

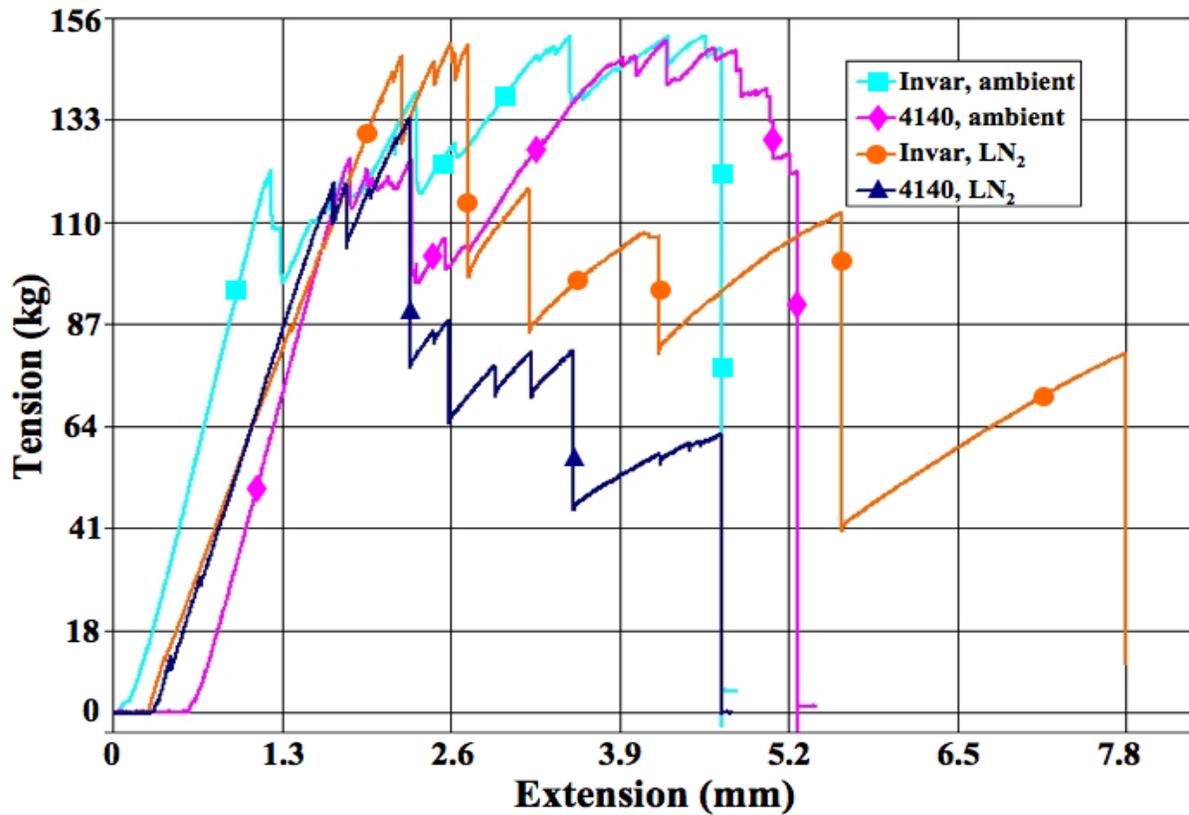


Figure 6. Experimental results for the tension test experiments. The figure shows tension in kilograms and elongation in millimeters for four fixtures: two at ambient temperature and two at liquid nitrogen temperature. The figure reveals a sawtooth pattern for all the fixtures caused by the failure mode. As the basalt rod is stretched, it reaches a point where the weakest fibers in the cross sectional area fail. The tension then drops suddenly because only a fraction of the total cross sectional area is now available to support tension.

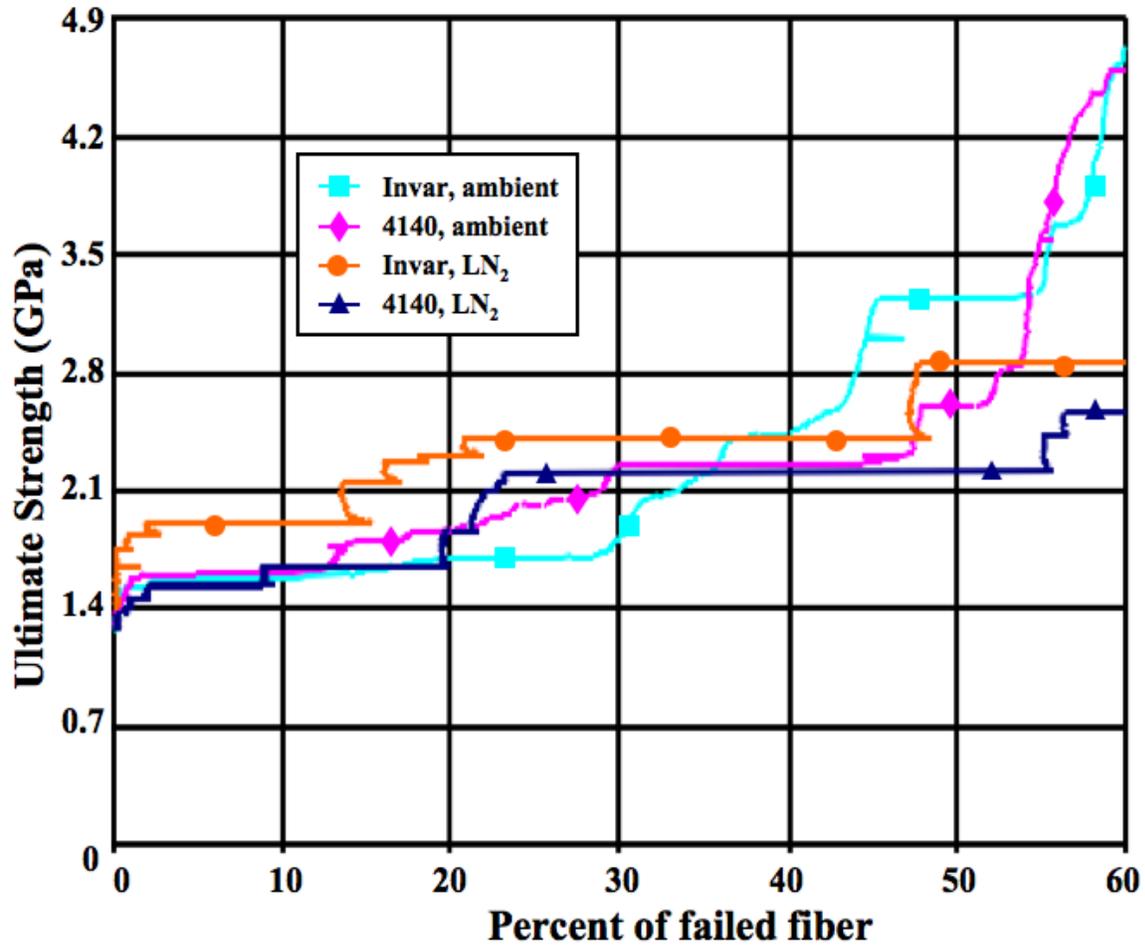


Figure 7. Analysis of experimental results in Figure 6 to produce results for ultimate strength as a function of the percent of failed fiber, for two probes at ambient temperature and two probes at liquid nitrogen temperature.