

# NEW, COMPOSITE POLYMERIC/METALLIC MATERIALS AND DESIGNS FOR HYDROGEN PIPELINES

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## 1. Introduction

Renewable energy resources in the U.S. could satisfy most of the nation's future energy needs. However, *distributed* sources of domestic renewable energy—particularly those east of the Mississippi River—cannot meet the concentrated energy demands of large cities and heavy industry. The richest *centralized* renewable energy resources in the U.S.—wind energy in the Great Plains States, and solar energy in the American Southwest—are largely stranded; i.e., located far from population centers, with no means for energy transmission or storage [1]. Long electric transmission lines could be built to tap these resources, but they are capital intensive, difficult to site and permit, and special financing may be required to recover transmission costs, and to earn a profit. In addition, if the transmitted electricity is produced entirely or mainly from wind or solar energy, overall system performance will be burdened by a low capacity factor (intermittency), and by the inability to store part of the energy to “smooth” or “firm” the delivery of power. *For these reasons, converting the produced electricity to hydrogen, and transmitting the gas through a network of pipelines, is a potentially viable alternative strategy for delivering the energy to distant markets.* Building new underground pipelines has historically been easier and faster than constructing regional electric infrastructure. Moreover, large-scale electric-transmission and hydrogen-pipeline systems are comparable in capital and O&M costs.

Thus, it has been suggested [1] that large-scale, on-site, electrolytic production of hydrogen, bulk storage of the produced gas, and long-distance pipeline hydrogen transmission, can provide “seasonally firmed” renewable energy to city-gate markets. To minimize greenhouse gas emissions, and to lower the costs of gas compression, the hydrogen could be formed from water (pumped from local aquifers, or delivered to the site by pipeline) using large electrolyzers that create gaseous hydrogen at pressures as high as 1,500 psi. The resulting pressurized gas is either directly injected into one or more pipes connected to a pipeline transmission system, or compressed to 2,000-2,500 psi for temporary storage.

Challenges for mass production of hydrogen in remote locations, and transmitting the gas to distant points of end-use, are daunting. One of the main difficulties—long recognized and extensively studied, but still largely unresolved—is safe, efficient, and cost-effective pipeline delivery of gaseous hydrogen at pressures  $\geq 500$  psi. Compressed to such levels, hydrogen is difficult to contain in two respects. First, due to the tiny size of its molecules, hydrogen will pass through the narrowest of passageways, which means that leakage is very difficult to prevent. Second, hydrogen readily dissolves in, and diffuses through, many of the solid materials that are commonly used to contain gases. This permeation, in some circumstances, can cause irreparable

physical damage to the wall(s) of the container, thereby enhancing the chances that it will rupture at some uncertain future time.

Thus, it is significant that new designs and methods of fabrication [2, 3] can greatly reduce the diffusive loss of hydrogen from containers of many shapes and sizes. For pipes, the techniques involve: (i) use of one or more layers of homogeneous or laminated polymeric material, and (optionally) solid metal(s)—e.g., copper (Cu), aluminum (Al), or stainless steel—and/or liquid(s), to create multiple equilibrium and kinetic barriers to hydrogen diffusion (e.g., Figs. 1 and 2); (ii) in special circumstances, physical separation of gaseous hydrogen from one or more static or flowing liquid interlayers; and (iii) when necessary, capture and recovery of escaping gaseous hydrogen at the points in a pipeline system where connections are made (Figs. 3-5).

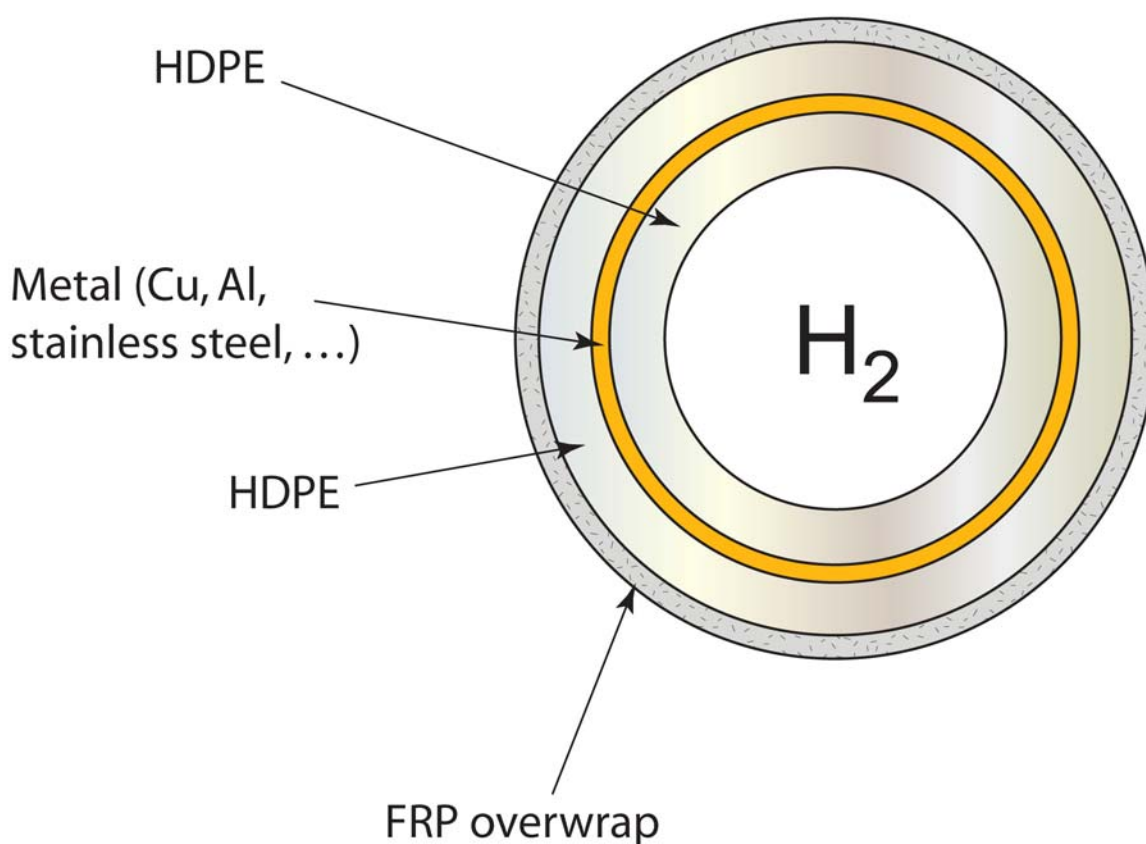


Figure 1. Transverse cross-section of a multi-layered polymer/metal pipe. HDPE = high-density polyethylene, FRP = fiber-reinforced polymer. For purposes of illustration, the thicknesses of the individual interlayers are greatly exaggerated.

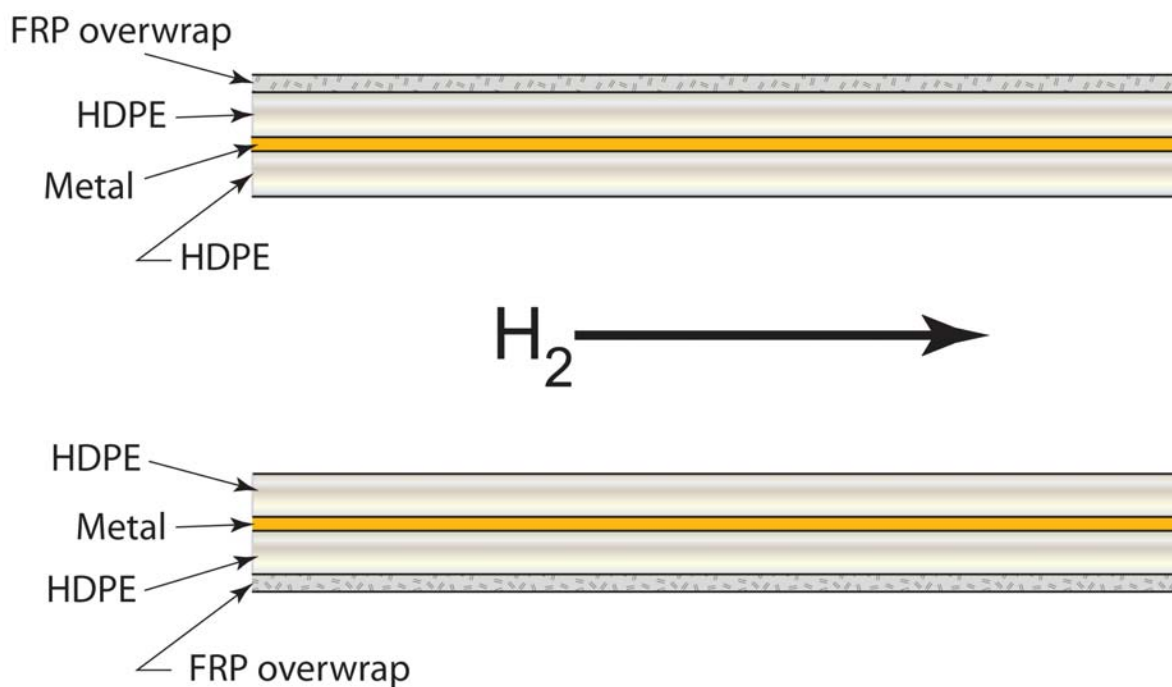


Figure 2. Longitudinal cross-section of the same multi-layered polymer/metal pipe shown in Figure 1.

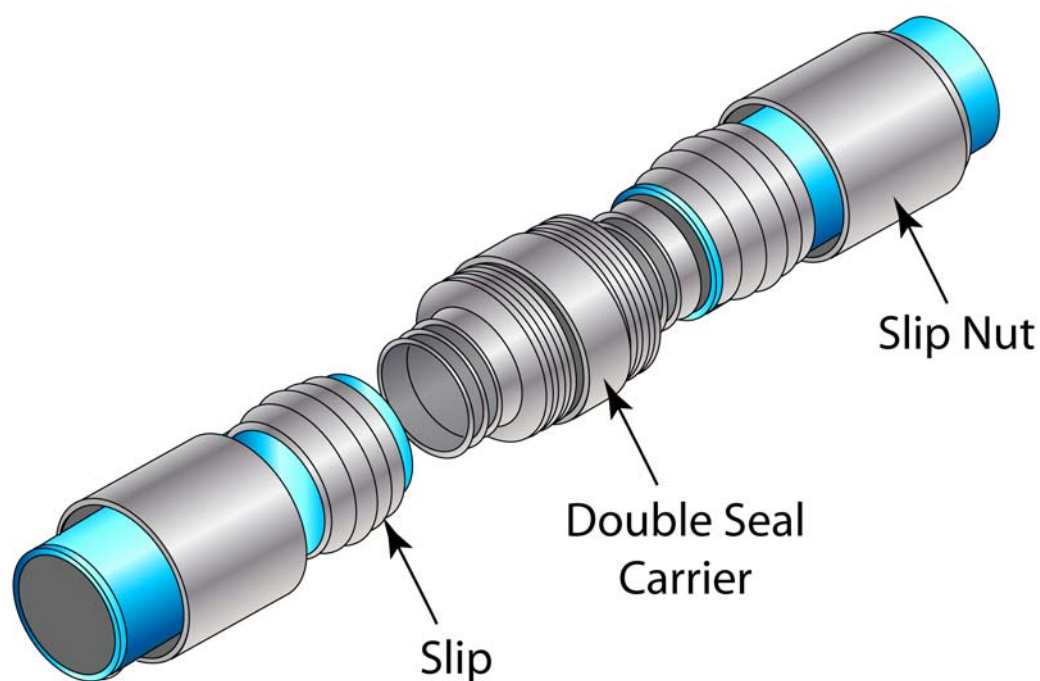


Figure 3. Schematic illustration of a pipe-to-pipe connector used by Fiberspar ([www.fiberspar.com](http://www.fiberspar.com)).

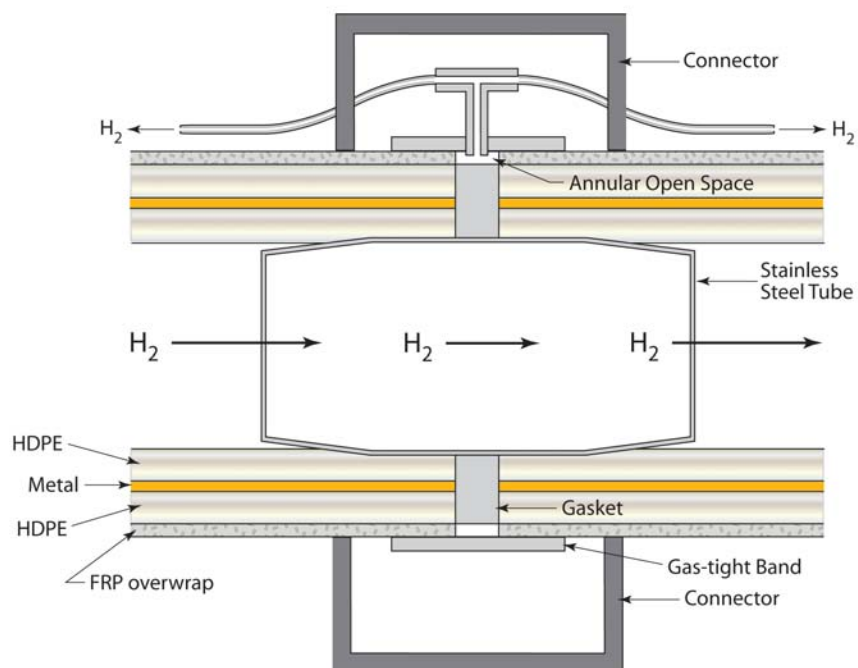


Figure 4. Schematic longitudinal cross-section/projection of a hydrogen-capture system invented by Hydrogen Discoveries, Inc. (cf. Fig. 5).

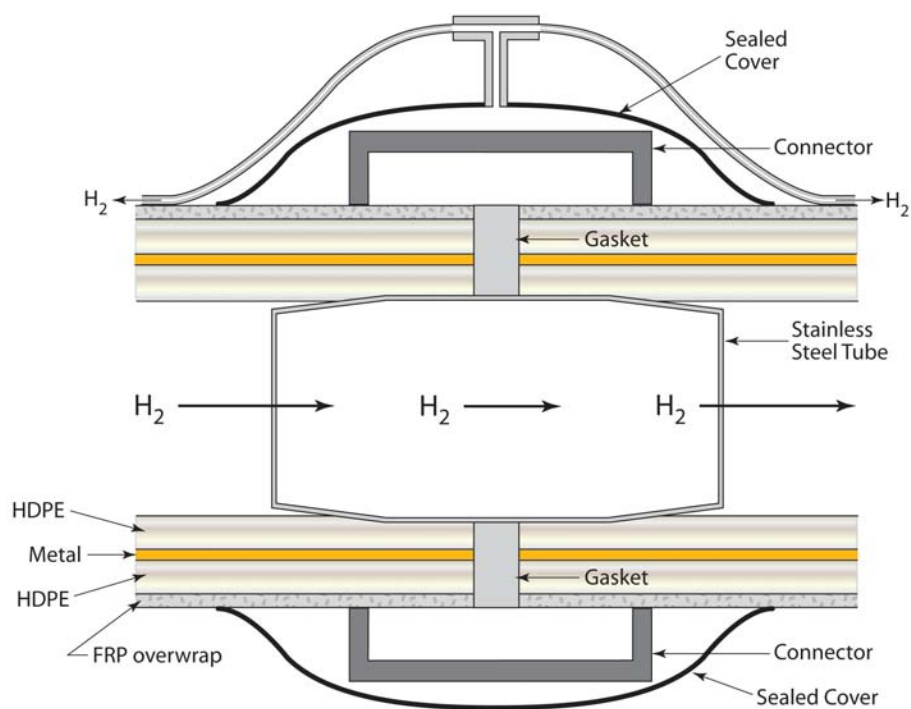


Figure 5. Schematic longitudinal cross-section/projection of a hydrogen-capture system invented by Hydrogen Discoveries, Inc. (cf. Fig. 4).

## 2. Materials of Construction for Hydrogen Pipelines

Most of the hydrogen produced today for commercial use is transferred short distances through relatively narrow-diameter pipes at pressures of just a few hundred psi. For this purpose, carbon steel has been the principal material of choice for pipeline construction; however, cast iron, copper, various plastics—e.g., polyvinyl chloride (PVC) and high-density polyethylene (HDPE)—have also been used, particularly to transfer the gas over short distances.

A major concern for future, high-capacity hydrogen pipelines is long-term durability at internal gas pressures  $\geq 500$  psi. It is well known that, at these pressures, carbon steels are susceptible to hydrogen embrittlement and cracking [4-6], and while the effects of high-pressure hydrogen on plastics are not well known, significant long-term negative impacts on these materials are also a real possibility. Hydrogen embrittlement of metals is generally manifested by surface cracking, crack propagation, decreases in tensile strength, loss of pipeline ductility, and reduced burst-pressure rating. This degradation can lead to premature failure of one or more segments of a pipeline, resulting in leakage of gas—or in extreme circumstances, bursting of a pipe. In view of these risks, it is not surprising that qualification of pipeline materials for hydrogen service at high gas pressures is currently an area of active research and development.

It has been suggested recently that many of the pipeline cost, weight, welding and joining, repair, and safety issues associated with carbon steel can be resolved by switching to fiber-reinforced polymer (FRP) materials [7, 8]. To have sufficient mechanical strength for high-capacity hydrogen delivery, a FRP pipeline segment would probably consist of an inner, low-permeability barrier tube that transmits the high-pressure ( $\geq 500$  psi) hydrogen gas, a protective layer placed over the barrier tube, an interface layer placed over the protective layer, multiple glass- and/or carbon-fiber composite layers, an outer barrier layer, and an outer protective layer. The issues and challenges for adapting existing FRP pipeline technology to hydrogen service at pressures above  $\sim 500$  psi are: evaluating polymeric materials for hydrogen containment, compatibility, and prolonged pressure-cycling; identifying methods for profitable manufacture of pipes with inside diameters  $> 4$  inches; weighing the options for on-site pipeline fabrication, joining, and repair; determining the availability of sensor technologies for measuring gas temperature, pressure, and flow rate in real time; and writing the necessary codes and standards to meet the requirements of local, state, and federal regulatory agencies. In this regard, it is noteworthy that the use of spoolable multi-layer FRP pipe (e.g., Fiberspar, [www.fiberspar.com](http://www.fiberspar.com))—or better yet, multi-layer FRP pipe continuously fabricated in the field (e.g., the Smart Pipe Company, [www.smart-pipe.com](http://www.smart-pipe.com))—would greatly simplify installation of long-distance hydrogen pipelines, thereby lowering *overall* costs of pipeline construction. FRP pipes can withstand large strains, which allows them to be “bent” easily and emplaced as a continuous, seamless monolith [8]. Finally, because FRP pipes can be manufactured with sensors embedded in their walls, it is likely that long-distance, large-diameter FRP pipelines built for hydrogen transmission could be operated as “smart structures.” This would enable lifetime performance-monitoring of the pipeline, which could result in substantial safety enhancements and long-term cost savings.

To bolster the hydrogen-containment capabilities of multi-layer structures of all shapes and sizes (but especially hollow cylinders), scientists at Hydrogen Discoveries, Inc. (HDI) have invented new methods for slowing and controlling hydrogen “loss” from the structures [2, 3]. The

inventions relate generally to the diffusion of hydrogen gas through solid and liquid materials, but focus particularly on diffusive hydrogen flux across the inner and outer surfaces of containers, or layers within those containers (“interlayers”), formed from one or more solid or liquid materials. As mentioned above, it is often observed that containers constructed from solid materials fail to prevent, or adequately control, release of stored hydrogen gas. In addition, permeation of hydrogen into a solid material can damage its microstructure and reduce its mechanical strength. The HDI inventions resolve these problems in two principal ways. First, one or more layers of polymeric, metallic, and/or liquid material(s) is used to create one or more supplementary, or enhanced, barriers to diffusion of hydrogen gas. Second, to augment creation of one or more supplementary or enhanced barriers to escape of hydrogen from a container, the released gas can be captured before it escapes to the surrounding environment (e.g., Figs. 4 and 5). The hydrogen containment and recovery practices inherent in the inventions can be applied to the construction of enclosures and passageways of many geometrical forms (planar, spherical, cylindrical, etc.).

The four main embodiments of the inventions are summarized below. Additional technical details are provided in the document entitled “Theoretical Analysis of Steady-State Diffusion through Multi-Layered Hollow Cylinders” [3].

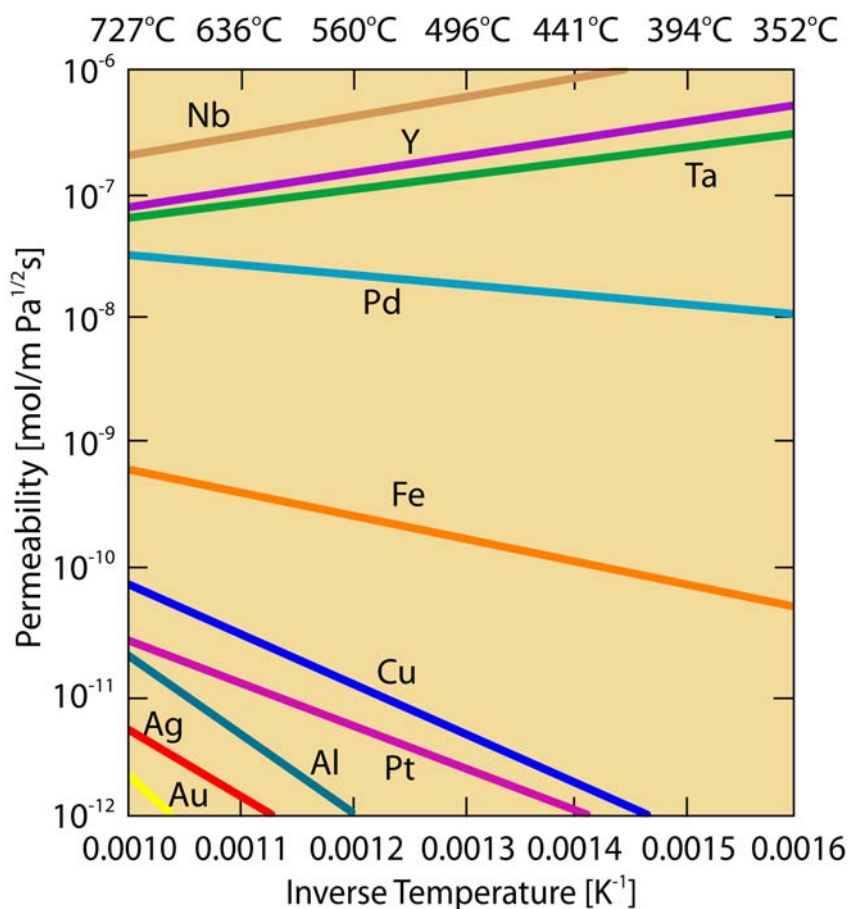
#### 1. One or more layers of laminated solid material

In this embodiment, diffusive flux of hydrogen gas is impeded by numerous thin layers of polymeric material, possibly containing one or more interlayers of metal foil. These layers are pressed together tightly to form one or more thicker, composite layers. Because mass transfer (diffusion) of hydrogen across the boundaries of the layers will proceed at finite rates, it is expected that gas concentration will be *discontinuous* at the boundaries between individual layers. The magnitudes of these discontinuities will depend on, first, the interfacial mass-transfer coefficients for the composite medium, and second, the equilibrium constants that represent the distribution of hydrogen between contiguous layers of contrasting composition. The former numbers undergird the concept of “contact resistance,” which refers to the degree to which diffusion of hydrogen gas is *impeded kinetically* by abrupt changes in microstructure at the boundaries of the individual layers in the multi-layer material. In this regard, a laminated polymer/metal composite is of particular interest because the modes of hydrogen dissolution in the two materials differ significantly. In polymers, hydrogen dissolves in the molecular (diatomic) state, whereas in metals, hydrogen molecules split into hydrogen atoms upon dissolution—reverting to the diatomic state only upon subsequent migration into a non-metallic material. If the latter substance is a polymer in which hydrogen is meagerly soluble, and if the polymer/metal interface is made sufficiently sharp by substantial compression, then a good possibility exists that hydrogen diffusion will be impeded due to the strongly nonlinear boundary conditions that are automatically created by this particular situation.

#### 2. Three-layer polymer/metal/polymer composites

For the reasons discussed above—and because metals such as Cu, Al, and stainless steel have very low “equilibrium” (steady-state) hydrogen permeabilities (Fig. 6)—a three-layer polymer/metal/polymer composite has high potential for being especially effective in deterring

hydrogen diffusion. [Note: When the wall of a polymer/metal pipe (e.g., Figs. 1 and 2) becomes saturated with hydrogen at a constant internal hydrogen pressure—i.e., reaches “equilibrium”/steady-state conditions—the thicknesses of the individual layers are no longer a factor in determining the *overall* rate of hydrogen flux. Thus, in this circumstance, a *thin* metal interlayer is as effective as a thick metal interlayer in slowing the overall rate of hydrogen escape through the wall of the pipe.] In addition, by virtue of its structural simplicity and ease of fabrication, a three-layer polymer/metal/polymer structure might prove to be a low-cost alternative to barriers consisting of finely-laminated polymers. A particularly attractive advantage of this embodiment is that the inner and outer (or upper and lower) layers of polymeric material will substantially protect the metal interlayer from mechanical abrasion and chemical attack. This can be important when the interior metallic layer is a foil formed from a metal that is relatively soft, or easily corroded (e.g., annealed, oxygen-free copper).



Source: REB Research & Consulting, 1996

Figure 6. An x-y plot of the hydrogen permeabilities of important metals plotted against inverse temperature. The curve for iron (Fe) is broadly representative of measured hydrogen permeabilities for carbon and stainless steels.

### 3. A composite material with one or more interlayers containing a static or flowing liquid

This embodiment has multiple forms that follow from three distinctly different objectives, which are, first, to achieve an enhanced ability to prevent hydrogen loss, second, to separate and capture escaping hydrogen gas by diverting its solid/liquid-state diffusion, or its buoyant ascent, toward one or more “points” of egress; and third, in the case of pipeline transfer of hydrogen gas from sites of electrolytic generation to remote destinations where it is used as a fuel, to permit reverse flow of either high-purity water or an aqueous electrolyte (e.g., Fig. 7).

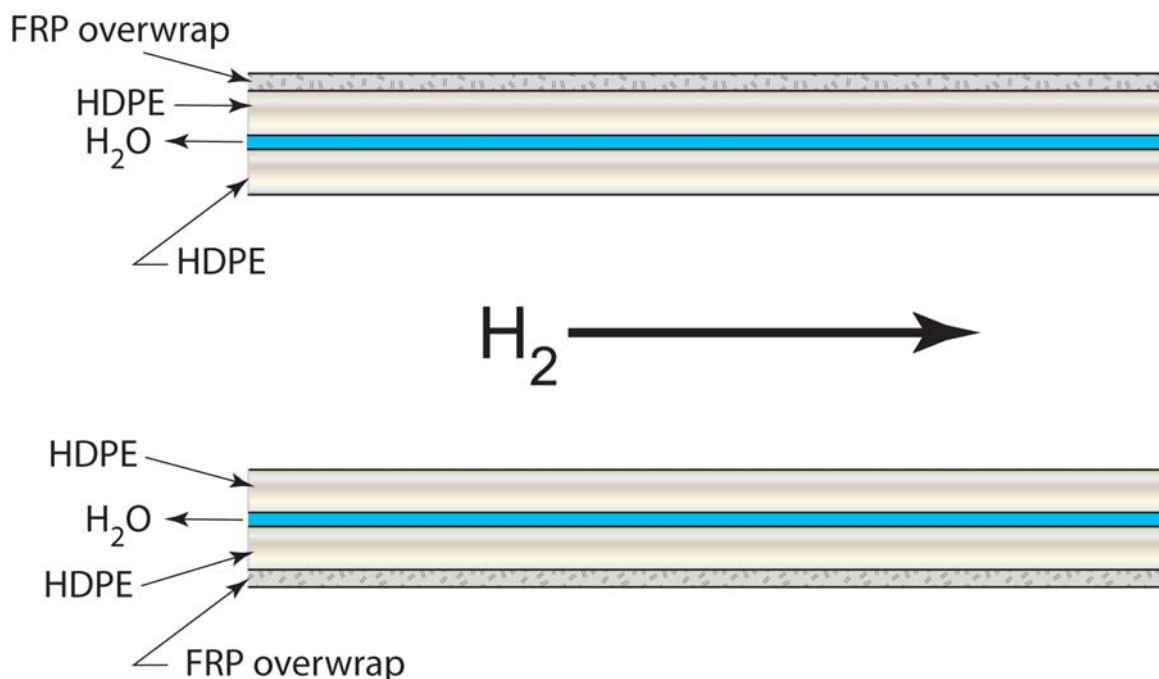


Figure 7. Longitudinal cross-section of a multi-layered, polymer/liquid interlayered pipe in which hydrogen gas and liquid water flow in opposite directions. See text for explanation.

In the first manifestation, one or more layers of nearly stagnant liquid, in which hydrogen is sparingly soluble, is used to decrease the overall rate at which gaseous hydrogen escapes from the container. There is little or no net flow of liquid into or out of the interlayer(s). In addition, no attempt is made to separate and capture the hydrogen gas that diffuses through the liquid(s), or which exsolves temporarily, forming a separate “free-vapor phase” due to cycling of temperature and/or pressure. The liquid(s) used might be, for example, one or more aqueous solutions that contain ordinary table salt, NaCl, or CaCl<sub>2</sub>. However, it is likely that hydrogen gas will be “salted out” more effectively using one or more salts that dissolve as doubly or triply charged ions. Aluminum sulfate is one such salt. This method of hydrogen containment is technically and economically appealing because diffusive loss of gaseous hydrogen is diminished using a material that is much cheaper and much more flexible than a layer of polymeric or metallic material.



The second manifestation leverages the relatively rapid rates of hydrogen diffusion through many types of liquids (e.g., high-purity water and dilute aqueous solutions) compared to polymeric materials. As noted above, the solubility of hydrogen in polymeric materials is high compared to many liquids. Significantly, however, diffusive flux of gaseous hydrogen is generally much faster in liquids than in polymers. Therefore, hydrogen-permeable membranes physically connected to one or more liquid layers in the multi-layer barrier material can be used to “tap off” substantial masses of the gas, thus reducing the total amount of gas that escapes through the outermost layer (or wall) of the barrier system.

The third manifestation affords enhanced containment of escaping hydrogen gas by trapping it in one or more flowing liquid interlayers, and transporting it advectively to one or more distal locations in the barrier system, where it is either consumed (e.g., used as a fuel), or reinjected into the structure from whence it came (e.g., Fig. 7). In the liquid interlayer(s), the hydrogen will be mainly transported either: (i) as a dissolved gas, (ii) as entrained bubbles of varying sizes, or (iii) as a continuous “headspace” gas overlying subjacent liquid material. Clearly, this capture, transport, and use/recovery operation decreases the mass of hydrogen gas that ultimately diffuses through the outermost/uppermost layer(s) of the container.

#### 4. Capture of hydrogen gas escaping from pipe-to-pipe connections

The fourth main embodiment of HDI’s inventions affords enhanced containment of escaping hydrogen gas by capturing it prior to release into the surrounding environment. An example of this technology is illustrated schematically in Figure 4, where it can be seen that diffusing hydrogen gas released into the sealed annular space surrounding a gasket placed between two interconnected sections of polymer/metal pipe is readily removed through a small port connected to a tee and two capillary tubes (see the top of Fig. 4). Using this and other similar strategies (e.g., Fig. 5), escape of hydrogen through pipe-to-pipe and “end” connections—if found to be a problem—can be readily managed, thereby eliminating the need to completely prevent such loss. This observation indicates that new connecting technologies will *not* be required for safe and cost-effective field deployment of multi-layered polymer and polymer/metal hydrogen pipes and pipelines.

### 3. Strategies for Large-Scale, Stationary Hydrogen Storage

In this section we briefly discuss the three principal options for *large-scale*, stationary hydrogen storage. The main purpose is to convince the reader that strong technological and economic synergies exist between remote, high-capacity hydrogen storage and long-distance hydrogen delivery.

#### Metal-Hydride Storage

It is certainly conceivable that, sometime in the future, metal-hydride technologies will improve sufficiently to allow low-cost, offboard metal-hydride storage of large masses of hydrogen gas. However, at the moment, prospects for that advancement are dim. The main problem is that the metal hydrides *with the highest hydrogen-storage capacities* must be heated well above 25°C to achieve substantial dehydrogenation. This increases capital and O&M costs because additional

equipment is needed to heat the material and, in addition, hydrogen storage operations overall become much more complex because *rates* of metal-hydride hydrogenation/dehydrogenation are always critically dependent on ambient temperature and hydrogen pressure. For these reasons, alternative methods for bulk hydrogen storage have much brighter prospects for near-term technical and economic viability.

### Geologic Storage

It has been suggested recently [1, 9] that huge masses of hydrogen can be stored indefinitely in subterranean geologic formations. The most amenable lithologies are salt domes, which can be solution mined to create the space needed to store the gas. In the U.K., >1,000 metric tons (MT) of hydrogen gas, produced for industrial use, are stored in several solution-mined salt caverns. The ChevronPhillips hydrogen-storage salt cavity near Freeport, TX—formed by pumping water into a salt formation over a five-year period—has been in operation since the 1980s. It’s “useable capacity” is very large, ~2500 MT, due in part to its high operating pressure, ~2,000 psi. However, hydrogen-storage facilities such as these can only be constructed in geologically favorable locations; i.e., where subsurface salt domes or salt beds are present at acceptable depths. Moreover, geologic hydrogen storage in more permeable lithologies (limestones, granites, etc.) is very problematic because satisfactory long-term hydrogen containment would be difficult to achieve. Consequently, alternative methods for bulk (>1000 MT) above/below-ground hydrogen storage should be considered.

### “Warehouse” Storage

A central theme of this paper is that polymer/metal-interlayered FRP pipes can be used not only to transmit and distribute gaseous hydrogen, but also to store it in bulk quantities. The latter result is achieved by building hydrogen “warehouses” filled with tightly packed aggregates of polymer/metal-interlayered FRP pipes (e.g., Figs. 1 and 2), arranged in one of two general, geometric configurations:

- horizontal rows of parallel pipes of equal/near-equal length, stacked vertically to a height close to the ceiling of the warehouse, and
- a group of axially concentric coiled pipes with outside pipe diameters decreasing progressively from the outermost coil to the innermost coil—e.g., 36-inch O.D. pipe for the outermost coil progressing to 4-inch O.D. pipe for the innermost coil.

For each configuration, suitable “superstructures” would be erected to provide adequate structural support for the pipes, and to hold them in place. In the linear configuration, individual pipes would be pulled into, and out of, steel troughs using procedures similar to those currently applied to pull polymer pipes through the interiors of abandoned steel pipelines (William D. Stringfellow, PE, Smart Pipe Company, personal communication). For the coiled configuration, a “basement” beneath the storage facility would be needed to allow individual pipes to be pulled into, and out of, a wound position. For each configuration, the ability to remove a pipe enables servicing or replacement as required. It is expected that polymer/metal pipes will have service lifetimes as long as 50 years, depending on susceptibility to the potentially damaging effects of

prolonged exposure to high-pressure hydrogen, and to hydrogen pressure-cycling. Total long-term costs of hydrogen storage should be low due to: (i) the overall simplicity of the storage strategy, (ii) unique methods of on-site FRP pipe production (Smart Pipe Company), and (iii) very low O&M costs after facility construction is completed.

Significantly, it is easy to show that warehouses similar in size to those currently operated by Wal-Mart have ample space to store huge masses of hydrogen gas in the manner described above. For example, a *single* Wal-Mart-style warehouse with 1.2 million square feet of floor space and a 30-foot high ceiling can store ~1250 MT of hydrogen gas in 24-inch I.D. polymer/metal FRP pipes pressurized to 2000 psi [10]. This is, first, nearly the same mass of hydrogen stored in salt caverns in the U.K., and second, approximately half of the mass of hydrogen that can be loaded into the ChevronPhillips storage cavity when it is filled to capacity. *Thus, warehouse storage of hydrogen could be a viable alternative to geologic storage, particularly if it can be shown to have comparable or lower capital costs amortized over average facility lifetime.*

#### **4. Planned Future Work**

To demonstrate the operational and economic feasibility of polymer/metal pipes and pipelines for bulk hydrogen delivery and storage, it is necessary first to show that short sections of tubing and pipe can provide satisfactory hydrogen-containment and hydrogen pressure-cycling performance under environmental conditions that closely simulate those expected for field-emplaced pipes and pipelines. Thus, it is our intention to perform bench-scale laboratory experiments, described below, to obtain most of the technical information that is needed to qualify multi-layer polymer and polymer/metal pipes for high-capacity hydrogen service.

##### Laboratory Tests at Constant Hydrogen Pressure

The hydrogen-containment capabilities of 6-18 inch lengths of 1-4 inch O.D. polymer and polymer/metal tubes and pipes will be determined by: loading a specimen into a tube/pipe test fixture (Fig. A1 in Appendix A), and submerging the entire apparatus in a water bath (Figs. A2 and A4); simultaneously evacuating, to a pressure <1 psi, first, the interior of the specimen, and second, the hydrogen-capture equipment on the downstream end of the tube/pipe test system (Fig. A4); filling the interior of the test specimen to a target hydrogen pressure, and keeping that pressure constant by connecting the upstream end of the tube/pipe test system to a large cylinder of hydrogen gas equipped with a high-precision pressure regulator; logging the time it takes for hydrogen to “break through” the wall of the test specimen; and finally, recording the rate at which hydrogen pressure rises in the hydrogen-capture section of the tube/pipe test system (Fig. A4). It is anticipated that time intervals to hydrogen break-through will be much longer for multi-layer tubes/pipes that contain a thin metal (Cu, Al, or stainless steel) interlayer, and that *overall* rates of hydrogen flux through the walls of those cylinders will be much lower than those measured for tubes/pipes that do not contain a metal interlayer.

### Laboratory Tests With Variable Hydrogen Pressure

The effects of hydrogen pressure-cycling on 6-18 inch lengths of 1-4 inch O.D. polymer and polymer/metal tubes and pipes will be determined by repeatedly raising and lowering the internal hydrogen pressure of each test specimen. This will be accomplished using: the HDI tube/pipe test system (Fig. A4); a 2-3 inch O.D. metal (Cu, Al, or stainless steel) “filler rod”; a small (1-3 gallon) distilled-water reservoir; one or more 1-4 inch O.D. stainless steel bellows; and two small water pumps connected to the distilled-water reservoir and the bellows. The filler rod, placed inside the short length of pipe, reduces the mass of—and therefore, the stored energy in—the compressed hydrogen gas that is loaded into the specimen. One of the water pumps transfers water from the distilled-water reservoir to the interior(s) of the bellows, causing it(them) to expand, which raises hydrogen pressure inside the test specimen. The other water pump transfers water from the bellows to the distilled-water reservoir, causing the former to contract, which lowers hydrogen pressure inside the test specimen. The rates of expansion and contraction will be computer automated, allowing 24/7 operation of the apparatus for any chosen total length of time.

After experimentation, the test specimen will be removed from the tube/pipe test fixture and examined for possible damage—manifested visually by swelling, blistering, or delamination of the innermost tube/pipe layer, and/or evidenced analytically or microscopically by changes in the chemical composition, or the microstructure, of that layer.

### Feasibility Modeling

The results of our laboratory measurements, independent information gleaned from literature sources, and the DOE/EERE H2A spreadsheet program, will be used to conduct thorough technological and economic analyses of leading options for bulk (offboard) hydrogen storage and long-distance (pipe and pipeline) hydrogen delivery. Key factors for bulk hydrogen storage are: the number and placement of storage facilities; and the relative costs of competing approaches, such as hydrogen storage in geologic formations (particularly salt domes and bedded salt) vs. storage in long pipes housed in Wal-Mart warehouse-sized facilities. Factors to weigh in modeling of pipe and pipeline transmission/distribution of gaseous hydrogen include: outside pipe diameter, wall thickness, and internal pressure; lengths of gathering, transmitting, and distributing lines; and potential spatial/geographic synergies with hydrogen storage facilities and high-pressure (~1500 psi) electrolyzers.

## **5. Conclusions**

- A *thin, continuous* metal (Cu, Al, or stainless steel) interlayer is extremely effective in blocking hydrogen permeation through the walls of polymer/metal pipes. Therefore, it is *not* necessary to develop a new polymeric material to achieve that containment performance.
- HDI’s fully scalable, polymer/metal pipe technologies use: *proven* materials of construction (HDPE, FRP, and metal films), and *proven* manufacturing techniques (used

by Fiberspar, the Smart Pipe Company, and others) to create multi-layered polymer and polymer/metal pipes.

- Existing connecting technologies can be used to link multi-layered polymer and polymer/metal pipes of any diameter, wall thickness, and length.
- HDI's simple hydrogen-capture technologies can be used to "manage" escape of hydrogen from places where multi-layered polymer and polymer/metal pipes are connected.
- HDI's polymer/metal pipe technologies have the potential to meet *all* of the Department of Energy (DOE) technical and cost targets for bulk (off-board) hydrogen storage and high-capacity (pipeline) hydrogen delivery.
- Unique experimental equipment and laboratory techniques developed by HDI scientists can be used to rigorously test the hydrogen-containment capability (up to ~2000 psi hydrogen pressure) and the hydrogen pressure-cycling (between approximately 500 and 1500 psi) durability of 1-4 inch O.D. sections of single- and multi-layer polymer and polymer/metal tubes and pipes.

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## **Appendix A. Experimental Equipment**

### Hydrogen Test System for Polymer and Polymer/Metal Tubes and Pipes

The hydrogen-containment and hydrogen pressure-cycling performance of 6-18 inch lengths of 1-4 inch O.D. polymer and polymer/metal tubes and pipes can be tested in one or more specially designed experimental facilities (tube/pipe test systems), each consisting of: (i) a tube/pipe test fixture (Figs. A1 and A4); (ii) a constant-temperature water bath (Figs. A2 and A4); (iii) two Haake DL-30 immersion heater/circulators (Fig. A3); (iv) a high-pressure cylinder of pure hydrogen gas; (v) a high-pressure gas regulator to control hydrogen test pressure; (vi) high-precision, hydrogen-service pressure transducers to measure (a) internal pipe pressure and (b) the pressure of hydrogen in a calibrated “leak volume” (Fig. A4); (vii) high-precision thermistor probes to measure the temperature of the water bath, and the gas inside the pipe test fixture; (viii) a high-pressure gas sampling cylinder (“leak volume”) to measure the volume of hydrogen gas diffusing out of the pipe being tested; (ix) high-pressure capillary tubing, fittings, and valves; (x) a vacuum pump and thermocouple vacuum gauge to evacuate the pipe being tested; and (xi) a custom data-acquisition system consisting of a desktop computer, National Instruments signal conditioning modules, and computer code developed using LabView™ test and measurement software (Fig. A5).

### Tube/Pipe Test Fixture

The tube/pipe test fixture (Fig. A1) will seal the open ends of a short length of polymer or polymer/metal tubing or pipe. Sealing will be accomplished using two high-pressure metal gaskets that force hydrogen gas to diffuse through the wall of the cylinder. The closures supporting the gaskets will use a proven high-pressure, split-ring design. The external support structure for the tube/pipe will be formed by welding stainless-steel flanges to the ends of a length of porous stainless-steel tubing, the external surface of which will be sealed to trap migrating hydrogen gas—forcing it to flow through a porous stainless steel tube connected to a calibrated “leak volume” by high-pressure capillary tubing (Fig. A4).

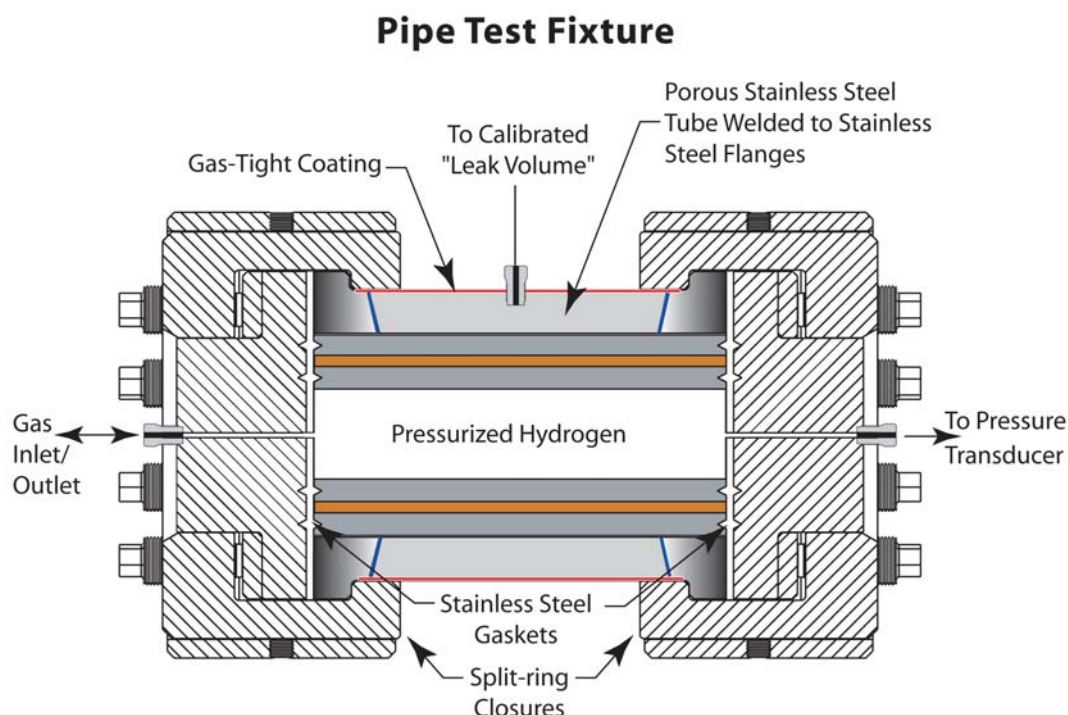


Figure A1. Multi-layer polymer and polymer/metal tube/pipe test fixture.

#### Constant Temperature Water Bath

The assembled tube/pipe test fixture will be immersed in a constant-temperature water bath (Fig. A2) with a total fluid capacity of approximately 180 gallons. The air gap between the tank and aluminum shell, and the tank top, are sealed and insulated when in service. This type of equipment has already been used by the authors to construct constant-temperature water baths for thermophysical measurements that require stable, high-precision temperature control from 20 to 50°C, with thermal control-cycle oscillations of <0.05°C over weeks of continuous operation—achieved by using: (i) a large thermal mass of water in the water bath; and (ii) two heater/circulators (Fig. A3), and two small submersible pumps, to reach experimental temperature, and to ensure rapid circulation of the water.



Figure A2. Stainless-steel water tank with an aluminum shell.



Figure A3. Haake DL-30 immersion heater/circulators.

### Polymer and Polymer/Metal, Tube/Pipe Test System

The assembled system for testing the hydrogen containment and hydrogen pressure-cycling performance of polymer and polymer/metal tubes and pipes is shown schematically in Figure A4. Hydrogen gas at pressures to 2000 psi is sealed inside a short length of tubing/pipe. Gas that diffuses through the wall of the cylinder is captured and quantitatively measured using a calibrated “leak volume” connected to a high-precision, hydrogen-service, 0-20 psi pressure transducer. Temperature in the bath water is measured at multiple positions, as well as adjacent to the sealed pipe being tested, using high-precision thermistors. Collectively, the pieces of equipment allow the performance of a polymer or polymer/metal pipe to be evaluated as functions of internal hydrogen pressure, temperature, time, and hydrogen-pressure cycling.



## HDI Multi-Layer Polymer/Metal Pipe Test System

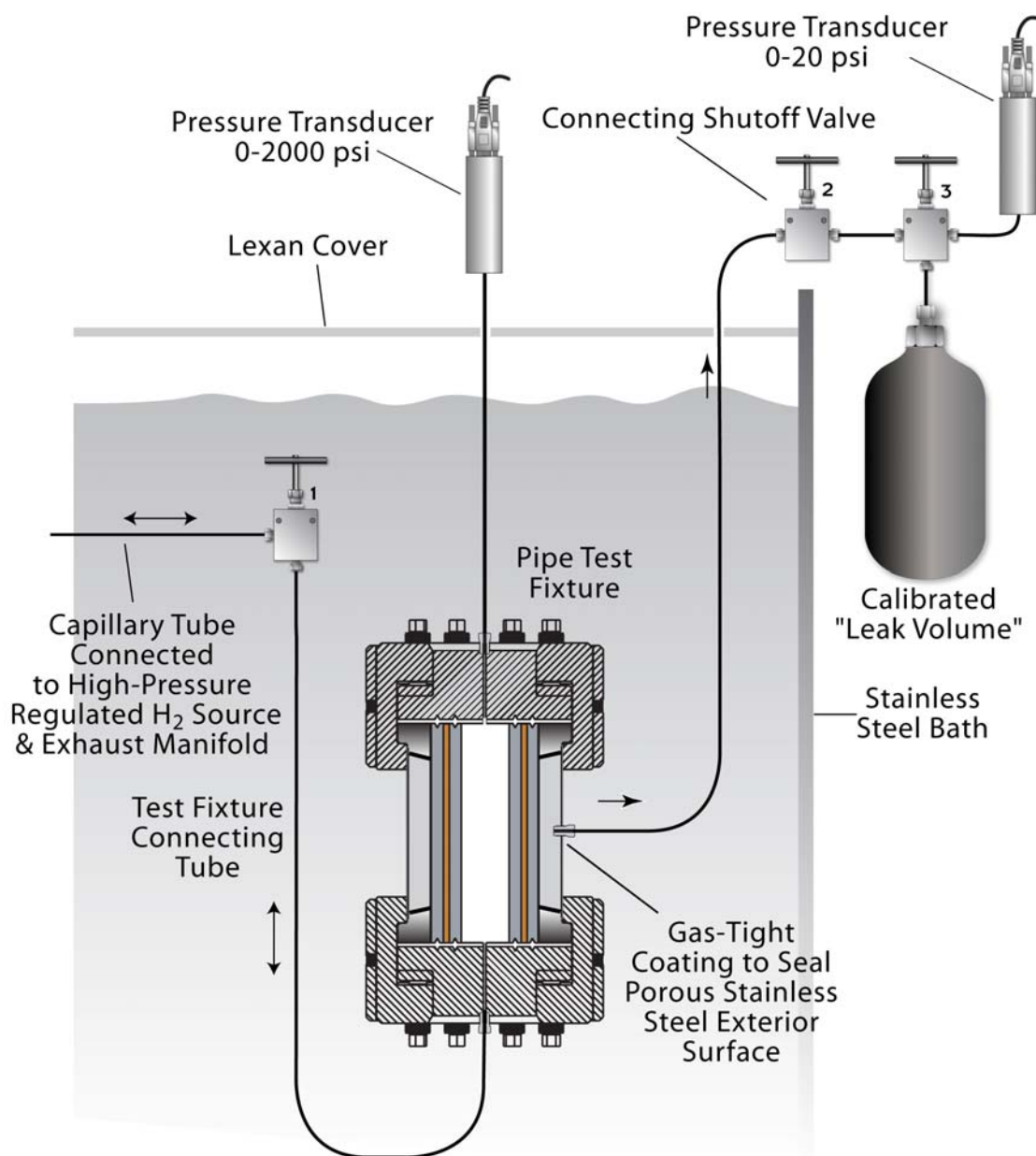


Figure A4. Multi-layer polymer and polymer/metal, tube/pipe test system.

## Data Acquisition

Signals generated by the pressure transducers and thermistors will be measured, linearized, and recorded using a data acquisition system assembled from components that include a desktop personal computer, National Instruments signal conditioning modules, and custom code developed using National Instruments LabView™ software. An example screen display for monitoring multiple pressure and temperature signals is shown in the photograph below (Fig. A5).

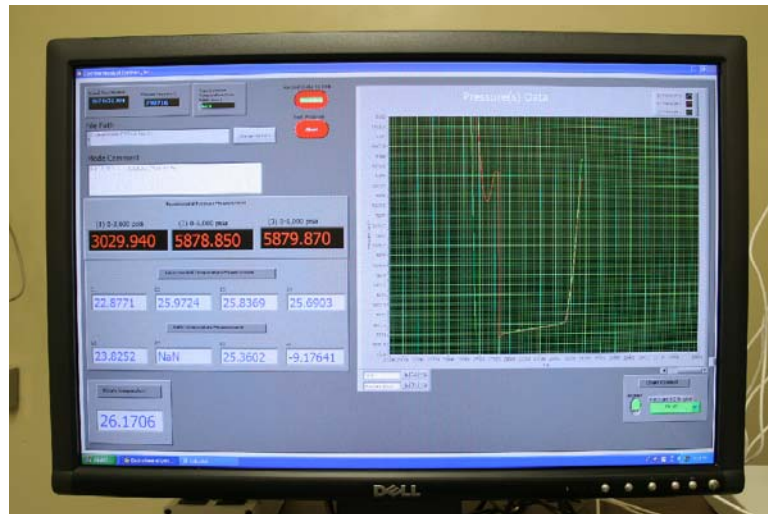


Figure A5. Example screen display for pressure-temperature-time data recorded by the data acquisition system.