International Hydrogen Fuel and Pressure Vessel Forum 2010
Beijing, China
September 27-29, 2010

Background

The China Association for Hydrogen Energy, the Engineering Research Center of High Pressure Process Equipment and Safety of the Ministry of Education in China, and the United States Department of Energy (DOE) conducted the International Hydrogen Fuel and Pressure Vessel Forum 2010, at Tsinghua University in Beijing, China, September 27-29, 2010. The Forum was co-organized by Professor Z.Q. Mao of Tsinghua University, Professor J.Y. Zheng of Zhejiang University (Hangzhou), and Antonio Ruiz of DOE. The main objective of the Forum was to gain better understanding of the technical and institutional challenges in harmonizing test protocols and requirements for compressed natural gas (CNG), hydrogen, and CNG-hydrogen (HCNG) blend pressure vessels and to define next steps for China and the U.S. to collaborate and address harmonization of regulations, codes and standards (RCS) for pressure vessels.

The Forum was conducted to follow-up on technical topics and issues identified during a previous international workshop on hydrogen and CNG fuels, including blends, held in Washington, DC, in December 2009. At the Forum, technical experts presented information and data on testing and certification of storage tanks for compressed hydrogen, CNG, and HCNG fuels.[1] Specific objectives of the Forum were to:

- address and share data and information on hydrogen, CNG, and HCNG vehicle research, development, and demonstration (RD&D)
- address and share data and information on testing and certification of Type 3 and Type 4 tanks, PRD testing and validation, tank inspection and monitoring
- review test protocols and requirements under specific regulations, codes and standards for on-board hydrogen tanks, including SAE J2579 and Global Technical Regulations in preparation
- identify and discuss key issues requiring additional research and development (R&D) and testing to ensure safe use of onboard and bulk storage tanks for hydrogen, CNG, and HCNG fuels
- enhance harmonization of RCS for storage tanks for hydrogen, CNG, and HCNG fuels
- identify and initiate future collaborations to address R&D, testing, and validation needs and to harmonize requirements in RCS among participating nations

Results of Forum

The results of the workshop in addressing its objectives are summarized below.

Address and share data and information on hydrogen, CNG, and HCNG vehicle research, development, and demonstration (RD&D)

There has been notable progress in advancing fuel cell and hydrogen technologies during the past decade in terms of performance, durability, cost reduction, and deployment, according to Dr. Sunita Satyapal, Program Manager of the U.S. DOE Office of Fuel Cell Technologies (*U.S. DOE Hydrogen and Fuel Cell Activities*). Deployment of fuel cells and hydrogen technologies for near-term markets has been

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[1] The presentations are available at http://www1.eere.energy.gov/hydrogenandfuelcells/m/forum_international.html.

particularly strong in the U.S. under the American Recovery and Reinvestment Act (ARRA) enacted to jump-start economic activity and create jobs after the recent global crisis and recession. Under the ARRA, the DOE has funded R&D in twelve projects that will deploy up to 1,000 fuel cells in a number of application areas, including backup power, residential and small CHP, and specialty vehicles. Total funding for these projects, including industry cost-share, is almost $100 million and includes large, established corporations and small, start-up companies. States are also actively deploying hydrogen and fuel cell technologies, particularly California, where there are twenty hydrogen fueling stations in operation with another ten planned. In addition, over 4,000 fuel cell vehicles are planned for deployment by 2014 and over 50,000 by 2017 in California. The DOE Safety, Codes and Standards sub-program is supporting R&D and testing so that all critical codes and standards are in place to enable widespread commercial deployment of fuel cell and hydrogen technologies by 2020.

In his keynote address (*HCNG in China*) on day 1, Professor Mao noted that of 27 "frontier technologies" that China has identified in its *Long-term Scientific and Technological Development Plan (2006-2020),* fuel cell materials are ranked 11th, hydrogen energy15th, and fuel cell power stations 16th. According to Professor Mao, China attaches importance to hydrogen, and he expects that fuel cells and hydrogen energy will be developed rapidly in the coming fifteen years. China is developing HCNG fuel as a "bridge" to a hydrogen economy, the most practical way to use hydrogen while at the same time reducing emissions from CNG vehicles while fuel cell technology matures as a commercial product. Such a transition strategy is feasible for China as natural gas accounts for about 4% of current national energy consumption (about 1 billion cubic meters), which is expected to double by 2015. In addition, there are about 400,000 CNG vehicles in China, with growth to 1million CNG vehicles with 3,000 CNG and HCNG fueling stations expected by 2015. It should be noted that India and Brazil, both countries with large CNG vehicle fleets, are also pursuing such a transition strategy to a hydrogen future.

China is also conducting R&D to improve the efficiency and reduce emissions of HCNG combustion engines. The Ministry of Science and Technology (MOST) funded a 9.4 million RMB project during 2006-2008 that included HCNG engine R&D, HCNG bus RD&D, and hydrogen produced by renewable energy. Professor Fanhua Ma (*Research and Development of HCNG Internal Combustion Engines*) of the State Key Laboratory of Automotive Safety and Energy at Tsinghua University presented extensive test data showing that HCNG fuel in a CNG engine can increase the burning speed and widen the lean burning limit for higher efficiency and reduced emissions compared to CNG fuel. Professor Ma acknowledged the importance of initial R&D supported by DOE at Colorado State University, Hydrogen Components, Inc., and Westport, Inc., in demonstrating engineering parameters and performance of HCNG fuels and engines for efficiency gains and reduction of CO, NO_x, and hydrocarbon emissions. Professor Ma and his colleagues have thoroughly mapped HCNG engine operation and fully characterized the performance and emissions benefits, limits, and tradeoffs of hydrogen enrichment of CNG fuel. Their testing shows that lean-burn utilizing spark-timing optimization is an effective approach for obtaining efficiency and emission benefits from a HCNG engine and that a 15-25% volume fraction of hydrogen is a good choice for HCNG fuel. With a 20% by volume fraction of hydrogen, engine emissions can meet stringent Euro Enhanced Environmental Vehicle (EEV) requirements while maintaining the same power output and with about a 7% reduction in fuel consumption compared to a CNG engine.

Researchers, such as Professor Ma, have tested and verified the benefits of efficiency gain and emissions reduction for properly calibrated HCNG engines. Questions concerning potential embrittlement of storage vessels due to hydrogen enrichment of CNG fuel were addressed by Mr. Frank Lynch (*International Codes, Standards and Experience Applicable to Storage of H2, Natural Gas and Blends of H2 with Natural Gas in High Pressure Cylinders*) of the Hythane® Company, LLC, and Dr. Jay Keller (*Hydrogen Effects on Materials for CNG / H2 Blends*) of Sandia National Laboratories. Mr. Lynch, as did Professor Mao and Professor Ma, noted the efficiency and environmental benefits of HCNG as well as the benefit of introducing hydrogen as a fuel through HCNG in a cost-effective way that also opens a market for renewable hydrogen. Hythane® is mostly methane (97 mass%, 93 energy%, 80 mole%) with similar ignition and flame characteristics. Like methane, Hythane® can be odorized, and, unlike hydrogen, its flame is not invisible in daylight. Mr. Lynch noted that worldwide most CNG cylinders are Type 1 (metal only), and, although there were a number of cylinder failures in the 1970s, research during the 1980s and manufacturing standards established during the 1990s have helped to eliminate problems with CNG cylinders. Mr. Lynch, quoting from an European Industrial Gas Association (EIGA) document, noted that "the performance [of steel] in hydrogen can be improved by limiting the maximum ultimate tensile strength and by closely controlling the alloy composition and heat treatment."

There are several ISO standards that address hydrogen embrittlement in steel cylinders, including ISO 9809-1 (Gas cylinders—refillable seamless steel gas cylinders—design, construction and testing—Part1: Quenched and tempered steel cylinders with tensile strength less than 1100 MPa) and ISO 1114 (Transportable gas cylinders—compatibility of cylinder and valve materials with gas contents—Part 1: Metallic materials and Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement). Under regulation GB-5099 in China, "Seamless Steel Gas Cylinders," the tensile strength limit for most gases is 1100 N/mm^2 (MPa), for "stress-corrosive gases" 880 N/mm^2 (MPa), and the sulfur limit is 0.035%. Furthermore, methane and the presence of other constituents in natural gas, such as oxygen, butane, and carbon dioxide act as "poisons" that inhibit hydrogen embrittlement, and coal gas, which typically contains 30-50% hydrogen, is allowed by cylinder Rule 23 (3) in India as long as sulfur compounds and water are limited. In summary, Mr. Lynch concluded that Hythane® [and presumably a HCNG blend with 20 mole % or less hydrogen] can be allowed in Type 1 steel cylinders certified under ISO 9809-1 because the partial pressure of hydrogen at 200 bar (3,000 psi) is below the limits set by ISO 9809-1.

In his presentation, Dr. Keller further addressed hydrogen embrittlement in Type 1 and 2 tanks as hydrogen-assisted fatigue cracking under pressurization and depressurization cycles during service life. Such tanks are commonly designed according to ANSI/CSA NGV2-2000, "Basic Requirements for Natural Gas Vehicle Containers," and ISO 9809-1, referred to by Mr. Lynch. Dr. Keller noted that all HCNG blends are compatible with steel CNG cylinders as along as the tensile strength of the steel is less than 950 MPa. However, cylinders designed to ISO 9809-1, as noted by Mr. Lynch, allow tensile strength up to 1100 MPa. Under ISO 1114-4, cylinders may be designed to ISO 9809-1 (with tensile strength up to 1100 MPa) if the working pressure of the embrittling gas is less than 20% of the test pressure of the cylinder <u>or</u> (emphasis added) if partial pressure of hydrogen in the gas mixture is less than 5 MPa (50 bar). Dr. Keller noted, however, that these allowances are not based on fatigue cracking data or on service experience and recommended evaluating hydrogen-assisted fatigue cracking in higher strength steels at low hydrogen partial pressure. Under ISO 1114-4, steels with tensile strength greater than 950 MPa can be qualified for blends containing more than 20% hydrogen by materials testing in hydrogen gas, but the specified qualification tests do not directly evaluate hydrogen-assisted fatigue cracking. Although limited fatigue testing on higher strength steels shows promising results, Dr. Keller recommended evaluating hydrogen-assisted fatigue cracking in higher strength steels at hydrogen partial pressures in the blend.

Dr. Keller also addressed whether the presence of CNG modifies hydrogen-assisted fatigue cracking in HCNG blends. Here the data are not conclusive as some tests show that hydrogen-assisted fatigue cracking rates are the same in HCNG blends as in pure hydrogen while other test data show lower rates in blends. The difference may be attributable to the presence of non-methane constituents in CNG, such as CO and oxygen that are known to inhibit embrittlement, but more test data are needed to determine whether such "contaminants" or CNG itself may provide an extrinsic mechanism to mitigate hydrogen-assisted fatigue cracking in steel cylinders.

Professor Jiyang Zheng of Zhejiang University (Hangzhou), while noting the positive aspects of HCNG fuels, also addressed hydrogen embrittlement as it affected the design and manufacture of steel cylinders for stationary storage of CNG, HCNG, and hydrogen *(R&D of Large Stationary Hydrogen/CNG/HCNG Storage Vessels*). Professor Zheng's team addressed embrittlement by using a multilayer steel ribbon wound design in which a thin inner layer made by rolling and welding steels compatible with hydrogen and HCNG, such as austenitic stainless steels, is wrapped by a second layer made by hot-rolling flat steel ribbon and a outer protective layer made of high-strength steels. The two outer layers form an enclosed space that allows leak monitoring and detection and ensures, according to Professor Zheng, a unique leak-before-burst failure mode. Built-in leak detection also enables safety monitoring and shutdown of compressors if a leak occurs. To reduce cost of materials, Professor Zheng's team designed weight optimization software that computes optimum structure parameters for given pressure and volume requirements. The design pressure of the vessels for inner diameters of 300-1500 mm is 10-100 MPa for a temperature range of -40 to 80ºC. Professor Zheng showed an example of three such tanks in service at a hydrogen refueling station in China, one of which is the largest 70MPa hydrogen storage vessel in the world. Vessels of this design are also used in the world's largest HCNG refueling station in Shanxi, China. For design and construction of the vessels, Professor Zheng's team relied on design codes in China and on the ASME Pressure Vessel Code, case 2229 (Division 1) and 2269 (Division 2). A national standard for "Stationary Flat Steel Ribbon Wound Vessels for Storage of High Pressure Hydrogen" is in the approval stage. Professor Zheng concluded by stating that large stationary flat steel wound vessels provide an economic and reliable method for bulk gas storage of CNG, HCNG, and hydrogen in China.

China is a major manufacturer of Type 1 tanks, and Mr. Xia Kang Ma, President of the Zhejiang Jindun Holding Group (*Large Diameter High Pressure Gas Cylinder Manufactured by Piercing and Drawing*), described the advantages and disadvantages of three different processes for producing seamless steel cylinders for high-pressure (>20 MPa) CNG storage: steel plate (deep drawing), seamless tubes (hot spinning), and steel billet (piercing and drawing). A key requirement under ISO standards is that for proper stress distribution, the thickness of the cylinder must increase progressively a minimum of 2x in the transition region between the cylinder wall and base. Mr. Ma showed finite element stress analyses demonstrating the high stress at this transition. This critical requirement can be most easily and consistently attained by using the steel billet process in which the bottom of the cylinder is formed by forging a hot billet into a mould to allow precise design and production of the bottom profile. The forging process requires a heavy-duty vertical press, and the larger the diameter of the cylinder, the heavier the vertical press required along with more difficulty in controlling wall thickness. The Zhejiang Jindun Pressure Vessel Company, Ltd., successfully developed the billet process and is the first and only CNG cylinder manufacturer in the world capable of producing cylinders larger than 356mm in diameter. Their cylinders are manufactured under the applicable standards issued by ISO, China (B1, B3), U.S. (DOT-3AA), Canada (TC-3AAM), India (IS 7285-2), and other countries.

Address and share data and information on testing and certification of Type 3 and Type 4 tanks, PRD testing and validation, tank inspection and monitoring; review test protocols and requirements under specific regulations, codes and standards for on-board hydrogen tanks, including SAE J2579 and Global Technical Regulations in preparation

Mr. George Hansen of GM (*Pressure Vessels for Hydrogen Vehicles: An OEM Perspective*) noted that the ideal situation from an automotive company's perspective would be that tank systems certified in one country would be allowed in other countries, which, in turn, would enable supplier-based development of tank systems on a global basis. To enable deployment on a global scale, there must first be global harmonization of

- codes and standards for on-vehicle hydrogen storage
- fueling protocols and interfaces
- hydrogen vehicle certification requirements and processes

Certification requirements and test protocols for on-board tanks along with fueling protocols and interface, among other things, would have to be harmonized with global standards, including those of ISO and SAE, as well as the Global Technical Regulations (GTR) under the U.N. (described below). Facilities to conduct tests and validate tank systems would have to be networked and round robin tests conducted to enable interchangeability of data among countries. Mr. Hansen recommended a round robin test among international testing facilities as an important step in harmonizing test protocols for certifying COPVs. Such harmonization would allow tanks certified in one country to be accepted in other countries. Special procedures and processes to permit small-volume operation, such as demonstration projects and pre-commercial rollouts are also needed. Global harmonization of requirements and certification procedures and processes for vehicle components and systems must also trigger discussion of infrastructure development to meet projected vehicle demand.[2]

Ms Min Lei of the Beijing Special Equipment and Inspection and Testing Center (BSEIC) described tests of Type 4 tanks that apparently led to the prohibition of these tanks in China (*Safety analysis of in-use vehicle wrapping cylinder*). The BSEIC was established in 1995, and under national law is responsible for the safety inspection of special equipment, including boilers and pressure vessels (www.bseic.com.cn). Tanks are inspected and tested after ten years in service and every three years thereafter. There are three manufacturers of carbon overwrap pressure vessels (COPV) in China, and some of the test data have been published in a Chinese journal.[3] Ms Min's presentation focused on the test approach and methods applied under the first periodic inspection of all composite wrapped (i.e., Type 4) CNG cylinders used by a public transport company in Beijing. The BSEIC assessed the safety condition of the cylinders through a performance test and the environmental effects of service through an acid resistance test. The performance testing was also conducted to assess failure modes and mechanisms. After about 5-9 years of service, the burst strength of the cylinders decreased to their design value because of cumulative fatigue damage and material aging, but the burst strength was more than 2.5 times the working pressure and indicated that the safety margin of the batch of cylinders remained adequate.

In fatigue testing, the cyclic loading pressure was 2 to 26MPa, and the cycling rate was 10 per minute. After 7500 cycles, "most of the cylinders" did not show signs of leakage or fracture. A cylinder was also subjected to burst tests after a surface defect deeper than the outer layer of wrap was inscribed on the middle part of the cylinder. The burst pressure of the cylinder was 74.5 MPa, and the burst occurred at the cylinder head, not at the location of the defect. An "air tightness" test showed that the cylinders were "safe and reliable." Test results also indicated that the acid resistance of the cylinders is good and that the composite material has good corrosion resistance. A cylinder immersed in an oil incubator at a constant temperature of 65℃ for 48 hours and subjected to a loading pressure of 2 to 26MPa three times per minute up to 7520 times did not leak and had a burst pressure of 56.4MPa (at the cylinder head).

The tests conducted by BSEIC included dissection of cylinders that revealed "macro" cracking in the liner, deformation of the liner and separation from the outer wrap, and cracks in the liner at the boss of the cylinder. The liner material is polyethylene, and outer layer is made of epoxy resin and glass or quartz fiber. At 70℃ the tensile strength and modulus of the liner material are about 50% and 34%, respectively, of that at "normal temperature." At -20 ℃ the tensile strength and modulus are about 1.6 times and 2 times, respectively of that at normal temperature. Between -20℃ and 70℃, the average linear expansion coefficient of the liner material is over 10 times that of the outer layer. Temperature fluctuation during the

[2] The "H2 Mobility" public-private initiative in Germany by government and industry (auto OEM, oil, utility, industrial gas) to jointly build a hydrogen fueling infrastructure represents a notable effort to do just this. Refer to Dr. Rasche's presentation at www1.eere.energy.gov/hydrogenandfuelcells/m/forum_international.html.

[3] An English translation of a preliminary report is summarized in Attachment A.

duty cycle of the cylinders, the pressurization and depressurization process, and the difference of material expansion coefficient between inner liner and outer wrap of the cylinders contributed to the deformation process and eventual failure of the cylinders.

Ms Min concluded that after the early stage of use, the residual strength of the cylinders decreased in varying degrees but the cylinders still met service requirements. The main failure factor of the cylinders was failure of the plastic inner liner due to quality defects that led to cracking and leakage. Other failure factors included damage to the outer surface of cylinder from road hazards and inadequate joining of the cylinder body and the boss. Therefore, the main factors affecting the safety of the cylinders were the reliability of the inner liner, the reliability of cylinder-boss joints, and residual strength of the wrapped layer around the cylinder. Unexpected factors in use include external surface damage, vibration, and impact.

Dr. Chris Sloane, consultant to DOE, explained the rationale behind the performance-based requirements of SAE J2579, a possible framework and template for such harmonization (*Test Protocol for Hydrogen Storage Systems in SAE J2579 and GTR Requirements for Hydraulic Cycling Testing and Its Effects on Type 3 and 4 Containers*). Dr. Sloane first contrasted performance-based versus prescriptive standards. Performance-based standards require demonstration of capability by a component or system to perform safely under known and anticipated on-road conditions as well as under extreme conditions. Prescriptive standards test for known failure modes of existing or previous technologies and project performance under extreme conditions. Performance-based standards facilitate rapid technology advancement whereas prescriptive standards could inhibit or delay technology advancement. Dr. Sloane also contrasted design guidelines versus safety design qualification (verification) requirements. Design guidelines incorporate techniques such as root cause analysis, failure modes and effects analysis (FMEA), material properties and tests, environmental factors, and safety strategy to capture known and anticipated safety issues and to ensure design robust enough to withstand hazards and exposures during service. Design qualification, or verification, employs test methods to capture on-road performance profiles under extreme conditions.

Dr. Sloane described two test sequences required for design qualification/verification in SAE J2579, *Technical Information Report for Fuel Systems in Fuel Cell and other Hydrogen Vehicles*. The first test sequence in SAE J2579 captures extreme demand profiles for compressed hydrogen storage vessels in on-road service by passenger vehicles: the number of fueling/defueling pressure cycles; duration of sustained pressure; and exposures to ambient temperature extremes, chemicals (acids, bases, solvents), and over-pressurization (failure of dispenser control systems at fueling stations). Under this profile, the worst-case on-road conditions for storage vessels include 5,500 pressure cycles (or 11,000 cycles for commercial heavy-duty service) up to 125% and 150% of normal working pressure (NWP) at temperature extremes of -40ºC and +85ºC (fueling/de-fueling), sustained exposure to high pressure (equivalent to 25 years at NWP (parking)), in-use impacts (scratches, abrasions) and chemicals exposures consistent with on-road service.

The second test sequence in SAE J2579 involves hydrogen-gas pneumatic pressure cycles and static pressure exposures of the full system, which includes the pressure vessel, the shut-off valve (automatic fail-safe closure valve), check valve (to prevent reverse flow in the fuel line), and the temperature activated pressure relief device (to release the content safely and rapidly and prevent burst from pressure build up during a fire). The full system must maintain full function, no leak, low permeation, and no rupture through expected service. In addition to the two sequential test series, a test to require the demonstration of a safe release of hydrogen during localized and engulfing fire conditions is being finalized for inclusion in SAE J2579. Requirements for leakage and absence of rupture during vehicle crash conditions are specified in SAE J2578.

According to Dr. Sloane, there are three open issues for completion of the SAE J2579 requirements. First, the localized/engulfing fire test, which was developed from fire test data presented by vehicle manufacturers, should be verified by performance of the test in an independent testing facility to demonstrate safe procedures for conduct of the test and to verify that appropriate system performance is demonstrated. Second, procedures to qualify metals for resistance to hydrogen embrittlement under the extreme temperature and pressure conditions used in compressed hydrogen storage need to be verified. Third, additional testing to establish expected stress rupture resistance of vessels under long-term static pressure should be undertaken and documented.

The purpose of the keynote address on day 2 was to discuss the link between R&D and regulations at the international level. Nha Nguyen (*R&D Needs for Global Technical Regulations for Hydrogen Vehicle Systems*) of the National Highway Traffic Safety Administration (NHTSA), U.S. Department of Transportation, described the development of global technical regulations (GTR) under the United Nations World Forum for Harmonization of Vehicle Regulations (WP.29) and the 1998 Global Agreement, which includes, among 30 contracting parties, Canada, China, the EC, India, Japan, and the U.S. The GTR, which is nearing completion, will be data and science-driven, performance-based (not design-based or prescriptive), and transparent (developed in an open, consensus process). When compliant with the objectively measurable requirements of the GTR, hydrogen vehicles will attain a level of safety equivalent to that of conventional gasoline powered vehicles. The GTR will address the high-pressure fuel container system (described above in Dr. Sloane's presentation), in-use and post-crash leakage limits of the fuel system, and in-use and post-crash electrical integrity of the high-voltage system. Results of R&D and testing underway in Japan, Canada, the U.S., and elsewhere have been discussed in the process of formulating the GTR. NHTSA is conducting R&D on cumulative life cycle testing, leak/permeation hold time, and residual strength testing of cylinders at end-of-life, as well as education and outreach on removal of defective and expired containers. Additional R&D needed include fire testing, cycling tests of the high-pressure fuel container system, and whole vehicle safety tests. If, as under discussion, the verification tests for performance durability and on-road performance, as set out in SAE J2579 and described in Dr. Sloane's presentation, are integrated in the GTR, it will provide a notable example of harmonizing vehicle regulations through incorporation of performance-based requirements. The GTR provides an example of how consensus on performance-based verification test procedures for components and subsystems can facilitate harmonization of vehicle regulations.

Mr. Joe Wong of Powertech Labs, Inc., (*Composite Tank Testing, Certification, and Field Performance*) pointed out that all fuel cell vehicle OEMs are using composite tanks for on-board fuel storage and that composite tanks are also used as industrial gas cylinders, transportable bulk tanks, and for stationary ground storage. Type 4 composite tanks are in wide use because they are lightweight, less susceptible to fatigue cracks (than metal tanks), have high toughness and elongation of liner material, have lower cost (than metal tanks) for higher pressures (up to 1000bar) and large volumetric capacity, and have a low capital cost for manufacturing. Vehicle OEMs are also advancing the use of 70MPa on-board storage pressures that will require up to 100MPa stationary storage tanks at fueling stations to enable fast filling of vehicles. Mr. Wong pointed out that "traditional Type 1 steel designs for large tanks" cannot be used for such high pressures, but it should be noted that Professor Zheng, in his presentation on the ribbon-wound, multi-layer steel design described above, stated that such tanks are capable of storing hydrogen at up to 100MPa and are now in service at hydrogen fueling station in China. Mr. Wong noted that ASME has recently approved the use of composite tanks for stationary hydrogen storage[4] and that data are needed to obtain regulatory approval of such tanks for stationary storage. Mr. Wong also showed that composite tank designs can be scaled up from 35MPa to 100MPa by adding extra carbon fiber wrap. While composite tanks for stationary applications must store large volumes of hydrogen at very high pressures, in contrast to extreme conditions in vehicle applications described by Dr. Sloane, they will be subjected to much less severe service conditions, particularly fewer and less severe pressure cycling and little or no exposure to road environments and collision. That said, Powertech found that tanks in a

[4] ASME Boiler and Pressure Vessel Code, Section X, 2010 Edition.

cascade storage bank for a CNG fueling station serving up to 100 vehicles/day typically underwent 10,000 pressure cycles per year at 70-125% of service pressure.

Powertech has maintained an international database of catastrophic CNG cylinder failures (ruptures) as well as major leaks attributed solely to the cylinder. The database includes 26 catastrophic failures involving rupture of CNG cylinders in the period 2000-2008. There were also other failures attributable to leakage due to pinhole leaks in steel cylinders (50) and to leakage in plastic liners in fully wrapped composite cylinders ("hundreds"). In addition to such leakages, causes of failure tabulated in the database are mechanical and environmental damage and their combination, overpressure due to faulty fueling equipment or cylinder valves, and vehicle fire due to the absence or malfunction of pressure relief devices (PRD). Type 1 (steel) cylinders were involved in nearly 50% of the failure incidents. Over half of the reported incidents also involved aftermarket vehicles, which typically are fitted with readily available Type 1 cylinders without strict adherence to installation codes and other best practice procedures.[5] Metal liner (Type 3) problems involved manufacturing defects, including pinhole leaks, laminations, and poor heat practice. Plastic liner (Type 4) issues in the database involved manufacturing defects, including cracking at the boss-liner interface, flawed welds, and liner seal failures. More generally, plastic liner problems involve the long-term integrity of the connection between the liner and the metal end boss (mechanical connection, O-ring seal, adhesive seal), effects of aging due to extreme temperatures, welding of plastic liners (to the end boss), permeation, buckling, and static discharge.

Powertech has also conducted tests to help validate SAE J2579 with a Type 3 (Dynetek 36L) tank and a Type 4 (Lincoln 80L) tank, both designed for 70MPa service. Mr. Wong noted that validation of performance based standards require testing conditions that closely simulate service conditions and that control and measurement accuracy of tank pressure, tank temperature, the test medium (hydraulic and pneumatic), and the rate of testing must be ensured. The validation effort involved verifying that the tests specified in J2579 can be performed by a test facility, that vehicle storage systems that have failed in past vehicle service will not pass the J2579 tests, and that such systems that have not failed in past vehicle service will pass the tests or fail the tests only when the reasons for failure are understood and would be expected to occur in vehicle service. Possible additions include test procedures and performance criteria for localized fire and provisions for re-qualification for additional service.

Provisions to address localized fire will be an important addition to J2579 as, according to Mr. Wong, the leading cause of CNG cylinder failure is vehicle fire, and the single leading cause of vehicle fire failures is localized fire effects. Powertech conducted tests for NHTSA to verify the effectiveness of a localized flame test developed by Transport Canada and developed a more versatile flame impingement test. As part of the tests, Powertech showed that protective coating and wrap systems that add minimal weight and wall thickness are cost-effective and that there are remote fire detection systems that work.

Better understanding of the parameters of fire tests is important to developing effective qualification tests for tanks. Professor Zheng reported (*Bonfire Tests of High Pressure Hydrogen Storage Tanks*) results of a Type 3 tank bonfire test that demonstrated proper activation of a PRD after 377 seconds and venting of the hydrogen and no rupture of the tank. Instrumentation showed that average temperatures on the surface of the tank varied considerably by location. There was no significant change in the initial pressure of 28.4 MPa for about 100 seconds after initiation of the bonfire due to the low thermal conductivities of the composite laminates of the tank walls, after which there was a gradual rise in pressure until activation of the PRD and a dramatic reduction in pressure. Professor Zheng and his team also developed and validated (with the experiment described above) a 3-D numerical model to simulate

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[5] Discussed by Mr. Wong in his presentation at the workshop on Compressed Natural Gas and Hydrogen Fuels: Lessons Learned for the Safe Deployment of Vehicles, Washington, DC, December 10-11, 2009. Available at www1.eere.energy.gov/hydrogenandfuelcells/wkshp_cng_and_h2.html.

the bonfire test and evaluate the effects of fuel type (for the bonfire), fuel flow, and filling medium (in the tank) on temperature rise. The team compared methane and propane as fuel type and determined that the heat of combustion transferred to the contents of the tank is much greater for propane than for methane and that the flow rate should be larger than 400NL/minute with methane and 150NL/minute with propane to generate a sufficient rate of temperature rise. A key finding was that the filling gas, be it air or hydrogen, has little effect on the rate of temperature rise.

Dr. Norm Newhouse noted (*Lincoln Composites Tank Manufacturing, Testing, Deployment, and Field Experience*) that Lincoln Composites (LC) initiated development of composite tanks in 1990 and manufactured the first Type 4 tank certified to ANSI/AGA NGV2. There are approximately 100,000 Type 4 tanks from Lincoln Composites in CNG service. LC manufactures Type 4 tanks (the "Tuffshell"™) for hydrogen service and recent deliveries include, among others, more than 300 tanks for fuel cell lift trucks at 700bar, 350 and 500 bar tanks to Honda for fueling station service, 700 bar tanks to GTI and Shell for cascade fueling systems, and eight roof-mounted tanks certified by TUV to 250 bar for two Neoplan buses (four tanks each) in Germany. LC Type 4 tanks are also in service as high-pressure accumulators in hydro-mechanical drive systems of buses and delivery and refuse collection vehicles to store energy (compressed air) during braking for use to accelerate the vehicle, increase efficiency, and save fuel. LC tanks are qualified for service under CSA HGV2 that includes tests for strength and life cycle, environmental hazards and exposures, and damage tolerance. To date, only two LC Type 4 tanks have ruptured in service. One involved fire in a parked vehicle in which the PRD was isolated and did not receive sufficient heat from the fire to activate. The second occurred during fueling of a delivery vehicle whose tank was not properly mounted and had experienced severe abrasion in over 14 years of service; there was also no indication of inspection during this extended service time.

As noted above, the Type 4 tanks tested by the BSEIS experienced failure due to separation of the liner from the outer wrap. Polymer materials absorb and permeate gases, and gases are released as pressure in the tank is decreased. According to Dr. Newhouse, proper design, choice of materials, and manufacturing can prevent problems with release of gas from the liner. LC Type 4 tanks use HDPE liners that meet permeation requirements of CNG and hydrogen and have superior fatigue life. Maintaining minimum normal operating pressure avoids most problems with liner separation or collapse. In fact, LC tanks have demonstrated no loss of function upon re-pressurization after full collapse of the liner. There have been no known instances of fatigue problems with LC Type 4 tank liners in service. A LC Type 4 tank has been subjected to 1,000,000 cycles without leakage. Some LC Type 4 tanks have reached the end of their 15-year life with no indication of problems with permeation or loss of strength. A number of LC tanks were tested after 9 years of service and subjected to varying severity of leak and proof of strength tests. Five tanks passed visual and proof and leak tests, and one tank was subjected to 45,000 cycles and passed all proof, leak, and burst tests with no evidence of loss of strength. Still another tank after dissection showed no evidence of deterioration, including the liner. Another tank passed permeation tests, met NGV2 requirements, and again showed no signs of deterioration. In summary, LC has been manufacturing composite pressure vessels, including Type 4 tanks, for over 45 years. LC chose to manufacture Type 4 tanks for CNG and hydrogen service because of benefits and advantages outlined above in Mr. Wong's presentation, and LC Type 4 tanks have been proven to be safe and reliable in service for both stationary and on-board applications. Such record of service can be attributed to proper design, materials, and manufacturing processes and to rigorous qualification of the tanks to appropriate standards.

Dr. Chris Rasche described the Type 3 tanks manufactured by Dynetek (*Tank Manufacturing, Deployment and Field Experience*). The "DyneCell" (DC) has a seamless aluminum liner that is non-permeable and resistant to hydrogen embrittlement and enables fast-fill. The high strength-to-weight carbon fiber overwrap increases cycle life and "extreme resistance" to chemicals. The DC is the lightest CNG cylinder with a metal liner in the market and has the highest storage capacity of all lightweight designs. Dynetek tanks are in service in both stationary and mobile applications and are certified in many countries, including Canada, the EC, Japan, Korea, India, and the U.S. Dr. Rasche described the manufacturing process for the DC and its qualification process, including visual inspection, dimensional check, and TUV inspection. A DC with a intentional flaw cut to a depth of 50% of the laminate thickness (outer wrap and liner) underwent 45,869 cycles without failing, and another DC with the same flaw cut was tested to a burst pressure of 512 bar. The DCs are qualified by tests similar to those used by LC. Dynetek also uses a patented thermally activated pressure relief device that meets the requirements of ANSI/IAS PRD1-1998.

During discussion, Dr. Rasche noted that while both Type 2 and Type 4 tanks are not allowed in India, Dynetek is the only company that has received approval in India for its composite tanks and plans to start manufacturing Type 3 tanks in India. Dr. Rasche also stated that the German government does not allow pre-cooling of hydrogen fuel because it requires too much energy and that Type 3 tanks do not require pre-cooling. Dr. Rasche also recommended that ISO (TC197 and TC58) should develop an inspection manual for COPV.

In his presentation, Mr. Hansen described the use of 700 bar Type 4 tanks by GM in Europe and Asia. These tanks are used in the world's largest fuel cell electric vehicle (FCEV) fleet and market test with 117 FCEVs (as of September 8, 2010) deployed in the U.S., Europe, Korea, and China. In China, GM and SAIC have jointly developed the "Shanghai FCEV" based on SAIC's Roewe 750 model and GM's Equinox fuel cell propulsion system, including a 700 bar Type 4 tank system. The Equinox FCEV fleet has logged over 1.6 million miles and refueled with hydrogen more than 20,400 times. Mr. Hansen noted that with a 700 bar system, 10% more compression energy returns 55% more energy content (6.2 kg H2 or 207 KWh vs 4kg or 133 kWh) over a 350 bar system. The Equinox FCEV storage tank contains a HPDE liner overwrapped with carbon fiber-epoxy for structural reinforcement and an outer glass fiber-epoxy layer for protection from road hazards and environmental damage. The polar end bosses are steel. According to Mr. Hansen, Type 4 vessels compared to Type 3 vessels have 20% lower weight for the same volumetric storage density, less long-term fatigue issues with little or no liner cracking, and with current and projected lower cost carbon fibers.

Dr. Pietro Moretto of the European Commission's Joint Research Centre (JRC) described the European Union's concept of "type-approval" of vehicles (*EU Regulation on Type-approval of Hydrogen Vehicles and Its Implementation*) regulated under Directive 2007/46/EC (Whole Vehicle Type Approval System). The objective of the Directive is to ensure proper functioning of the internal market in the EU by establishing uniform requirements for marketing vehicles and fully harmonized technical provisions in all 27 member states in the EU. The Directive was amended to include type-approval of hydrogen-powered vehicles in 2009 and further augmented in 2010 by provisions to implement the requirements for such vehicles. Requirements for compressed gaseous hydrogen storage containers under the amendment are similar to those required under ANSI/CSA NGV2. The EC is working closely with the contracting parties developing Global Technical Regulations (GTR) for hydrogen vehicle systems under the U.N.

Dr. Moretto identified a need for collaborative pre-normative R&D and testing to establish a sound, scientific basis for globally harmonized requirements and procedures for component certification. In addition to being science-based, this R&D and testing must be peer reviewed and results publicly available. There must be statistically reliable test results for essential commercially available products, which in the case of on-board storage tanks may be difficult as obtaining sufficient number of tanks for testing will be expensive. Furthermore, results from different test facilities must be independent and comparable, and a well-prepared and unbiased round robin test protocol and program may be required.

Dr. Moretto provided two examples where harmonization of test procedures and criteria is essential for globally consistent tank certification: gas cycle testing and allowable permeation rates (Type 4). For gas cycling tests, there are varying criteria and procedures among the EU, Japan (JARI), the U.S. (SAE), and ISO. It should be noted that the effort to develop a GTR for hydrogen vehicle systems (described in Mr. Nguyen's presentation) may enable a harmonized cycle test protocol. A collaborative fast-fill modeling and analysis effort would also improve understanding of the interactions of pressure, temperature, and strength of materials parameters and lead to cycle test parameters that more directly address potential failure modes. Allowable permeation rates in Type 4 tanks also vary among the EU, Japan, U.S., and ISO.[6] Criteria for setting allowable permeation rates need to be examined, including a better definition of and distinction between a "leak" and "permeation." Measurement location and procedures to determine permeation need to be harmonized, and additional CFD modeling and experiments are needed.

Given the expense of testing and certifying composite tanks, non-destructive evaluation (NDE) methods are widely practiced in industry and government. Mr. David McColskey of NIST, in collaboration with colleagues at NASA White Sands Test Facility (*NDE Methods for Certification and Production/Performance Monitoring of Composite Tanks*), noted that two major assessments of composite overwrapped pressure vessels (COPVs) by the NASA Engineering and Safety Center (NESC) concluded that no NDE technique is currently known to be directly applicable to prediction of stress rupture and other life-limiting damage mechanisms in COPVs. The assessments recommended that the NDE, materials, and structural technical communities collaboratively plan and undertake a feasibility study of potential NDE techniques that may be capable of detecting degradation that leads to stress rupture in carbon COPVs and identify chemical and physical changes to target NDE and any NDE response that correlates to progression toward stress rupture. Mr. McColskey described the approach, methodology, and progress of a national team of NDE experts who are attempting to develop and demonstrate NDE techniques for real-time characterization of COPVs and identify NDE capable of assessing strength degradation related to stress rupture and predicting vessel life through structural health monitoring or periodic inspections. The team would also develop more data on stress rupture progression in carbon-epoxy COPVs. Acoustic emission (AE) tests showed that increase in the rate, strength, and density of signals during stress rupture or pressurized cyclic testing can provide an indication of progression toward rupture.

For the carbon cyclic-pressurization testing project, the NASA team has assembled a state-of-the-art, 20-tank test station that can hold pressure at approximately $\pm$ 2 psi regardless of temperature. The test station has a protective enclosure that allows visual inspection and real-time NDE data acquisition during the pressurization cycles. The test team is correlating failure data with existing community databases. Initial results show some promise that failure can be predicted "`when related to damage-site monitoring.'" Results also showed that AE monitoring of the initial pressurization of "virgin vessels" can provide a "characteristic damage state" of the vessel and a measure of the repeatability of COPV manufacturing processes. Additionally, AE shows promise as an excellent choice for monitoring the health of vessels in service. In summary, NDE is highly effective in real-time characterization of COPV during testing and is reasonably effective in evaluating the health of COPVs. More work is needed to make NDE more quantitative and predictive, and much should be learned from a carbon COPV test project now underway.

Identify and discuss key issues requiring additional research and development (R&D) and testing to ensure safe use of onboard and bulk storage tanks for hydrogen, CNG, and HCNG fuels; enhance harmonization of RCS for storage tanks for hydrogen, CNG, and HCNG fuels; identify and initiate future collaborations to address R&D, testing, and validation needs and to harmonize requirements in RCS among participating nations

[6] This issue is being addressed under the on-going GTR process described by Mr. Nguyen above.

The keynote presentation by Professor Mao showed that while hydrogen and fuel cells are still important in the long-term science and technology development plans of China, the deployment of HCNG as a transition fuel is taking place and should provide evidence whether such a strategy is viable in China. Professor Ma showed that the parameters to optimize HCNG as engine fuel for efficiency and emission reduction are well understood and validated. While storage of HCNG with hydrogen content limited is approved for Type 1 steel cylinders by published ISO standards, the safety of HCNG storage in Type 1 tanks, however, is not completely resolved, and more conclusive testing is needed. Although R&D and testing of HCNG fuels, engines, and fueling infrastructure are not part of the portfolio of the DOE Fuel Cell Technologies Program, hydrogen-assisted fatigue cracking in steel cylinders is a high priority effort of the Safety, Codes and Standards (SCS) subprogram. Data from tests and materials compatibility research on hydrogen-assisted fatigue cracking in steel cylinders conducted by SNL are shared with ASME and could also be shared with the appropriate working groups and technical committees of ISO to help such entities determine whether pressure vessel requirements for HCNG storage should be further evaluated for possible changes or additions to existing design and qualification requirements. Mr. X. Ma noted that Type 1 tanks have been tested under NGV2 at a laboratory certified by a government agency in China. More information exchange between China and the U.S. on testing and certifying Type 1 tanks should be pursued. Specific follow-up actions could include:

- exchange information and data on test procedures and protocols to certify Type 1 tanks for HCNG fuels in road vehicles, buses, and other applications
- establish a collaborative project to develop procedures and protocols for an international round robin test of Type 3 and Type 4 tanks for selected duty cycles
- invite appropriate laboratories and regulatory agencies in China and the U.S. to participate in an international round robin testing effort for Type 1 tanks—invitations to laboratories and agencies in Brazil, India, and other countries where Type 1 tanks are used extensively should also be considered as should the IPHE RSCWG as a forum to catalyze and coordinate such collaboration
- establish a collaborative R&D project between China and the U.S. on hydrogen compatibility of materials for HCNG blends in selected applications and duty cycles—Professor Zheng and Dr. Keller have initiated discussion for such a project between their institutions

Information and data presented by two of the leading high-pressure composite tank manufacturers in the world show that Type 3 and Type 4 tanks are used widely in a number of applications, including buses, automobiles, and specialty vehicles, and have established a very good record of safety and durability in the field. Under ANSI/CSA NGV2 for CNG and replicated in CSA HGV2 for hydrogen, tanks are qualified by a sequence of (typically destructive) tests on separate tanks. A contrasting qualification procedure is specified under SAE J2579 where a tank is subjected to testing based on an on-road demand profile for performance durability and on-road performance under accelerated, near worst-case conditions. The approach and test sequence under SAE J2579 will likely be incorporated in the GTR for hydrogen vehicles. During discussion, Mr. Hajime Fukumoto of JARI noted that in 2012 regulations in Japan will be harmonized with the GTR (and therefore SAE J2579) and that METI is likely to approve adoption of the GTR. After the GTR is approved by the contracting parties of WP29, each of the parties will incorporate the requirements of the GTR in national regulations. For example, in the U.S., NHTSA will amend its Federal Motor Vehicle Safety Standards (FMVSS) to incorporate the GTR requirements. The test sequence in the GTR (described in the presentations by Dr. Sloane and Mr. Nguyen) can provide a template for international harmonization of qualification requirements for high-pressure storage tanks on hydrogen vehicles. Such harmonization should take place as the contracting parties incorporate the GTR requirements in their national regulations for hydrogen vehicle systems.

In his presentation, Mr. Hansen recommended a round-robin testing program among international testing facilities as an important step in harmonizing test protocols for certifying composite tanks. Such harmonization would allow composite tanks certified in one country to be accepted in other countries. In view of the international consensus forming around the SAE J2579/GTR test sequence and the validation tests on J2579 by Powertech Labs, the DOE could initiate formation of an international team to develop the procedures and protocol for a round robin test program as recommended by Mr. Hansen. The round robin test procedure and protocol should address the harmonization of cycling tests and allowable permeation that Dr. Moretto discussed in his presentation.

The presentations by Mr. Wong, Dr. Newhouse, and Dr. Rasche showed that there is an extensive database of composite tank performance and durability in the field. The data and information have been compiled by different organizations and housed in separate databases. An integrated international database of composite tank performance and durability in the field compiled and maintained by a public entity such as the DOE would be valuable. The DOE should consider organizing such an effort with partners from the EC, Canada, Asia, and South America.

Specific follow-up actions in consultation with appropriate experts who participated in the Forum could include:

- formation of a working group of key international experts to develop a round robin test program for composite tanks based on the SAE J2579/GTR test sequence and to enlist key test laboratories in the U.S., EC, Brazil, China, India, and Japan, among other countries, to participate in the program
- development of an integrated international database of field performance and durability of composite tanks
- organization of an international workshop to address composite tank manufacture, qualification, and certification, including NDE methods, and development of an inspection manual for composite pressure vessels
- formation of an international team of experts to develop a consensus fast-fill mode for Type 4 pressure vessels

Summary

The Beijing Forum, along with the international workshop held last year in Washington, DC, has yielded significant information and data about the development and deployment of CNG, HCNG, and hydrogen vehicles and infrastructure. More particularly, the Forum has opened opportunities for international collaboration that will facilitate the global deployment of hydrogen and fuel cell technologies in general and advanced composite pressure vessels in particular.

The U.S. Department of Energy would like to thank its co-organizers in China for hosting the Forum and for lending its expertise along with others in China to the workshop. The co-organizers also thank the many experts who gave presentations and participated in the Forum. These notes on the Forum tried to capture the key information presented and the potential collaborations that can follow. If there are any questions or comments about the notes, please contact the co-organizers of the Forum, Professor Z.Q. Mao (maozq@tsinghua.edu.cn), Professor J. Zheng (jyzh@zju.edu.cn), and Mr. Antonio Ruiz (Antonio.Ruiz@ee.doe.gov).

Attachment A

Thirty Type 4 tanks from CNG vehicles were tested after being in service from 5-9 years, and only 32% passed the qualification tests. Of these failures, 67% occurred in the boss and 27% in the cylinder (barrel). The principal failure mode was separation of the inner layer (liner) from the outer glass fiber wrap. Cylinders are inspected and tested after ten years in service and every three years thereafter. There are three manufacturers of carbon overwrap pressure vessels (COPV) in China, and some of the test data have been published in a Chinese journal.

A preliminary paper on the BSEIS tests, "Defect Analysis of Vehicle Compressed Natural Gas Composite Cylinder," was translated by Dr. J.P. Hsu of Smart Chemistry. According to the preliminary paper, the analysis was performed because, in addition to the high failure rates at inspection described above, there were four catastrophic failures of Type 4 tanks in service on CNG vehicles that resulted in loss of life and destruction of the vehicles. The analysis concluded that the failures were due to "poor environmental stress cracking resistance" of the HDPE cylinder liner and deformation of the liner under pressure and temperature cycling during the fueling and defueling process. The large difference of linear expansion coefficients of the liner and the composite overwrap led to increasing internal stress on the liner and inward deformation of local defect areas in the liner. After a number of unspecified cycles, the extent of deformation exceeded the deformation limit of the liner and caused separation from the outer wrap when the pressure in the cylinder dropped to 1 MPa. The paper recommended that cylinder pressure be maintained above 2.0 MPa. A presentation based on the translated report was given by Dr. Hsu during a workshop at Sandia National Laboratories, Livermore, CA, in April 2010.[7]

In order to evaluate the potential global impact of the ban on Type 4 tanks in China, more information is needed on the qualification tests performed by manufacturers before the tanks were placed in service and the pressure and temperature cycles experienced by the tanks during service (if available). A request has been submitted for a copy of a full report on the tests and analysis performed by the BSEIS for translation and further discussion. The ban on Type 4 pressure vessels in China based on testing by BSEIS shows that harmonization of qualification testing is critical for global deployment of composite tanks in vehicles.

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[7] Available at www1.eere.energy.gov/hydrogenandfuelcells/wkshp_onboard_storage.html


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