Validation Testing in Support of Hydrogen Codes and Standards Development

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ABSTRACT

New codes and standards are being developed to facilitate the safe deployment of emerging hydrogen technologies. Hydrogen markets will benefit from standards that address the specific properties of hydrogen, hydrogen effects on strength of materials and hydrogen compressed gas storage at pressures up to 70 MPa. The need for validation of new hydrogen requirements has been identified by codes and standards technical committees. The US Department of Energy (DOE) office of Energy Efficiency and Renewable Energy (EERE) has tasked the National Renewable Energy Laboratory (NREL) with the role of supporting hydrogen codes and standards research and development needs. NREL has provided validation test support to several new standards development efforts including pressure testing of 70 MPa on board vehicle storage systems, flaw testing of stationary hydrogen tanks, fill protocols for hydrogen fuel dispensing and hydrogen compatibility testing for hydrogen pressure relief devices (HPRD's). Validation test results are presented for these four specific standards development needs.

1.0 INTRODUCTION

Hydrogen specific codes and standards are an enabler for the growth of emerging hydrogen fuel cell markets by providing a sound basis for certification and permitting activities. New hydrogen codes and standards are being written to address safety requirements for technologies that include high pressure hydrogen storage systems, fueling protocols at automotive dispensing stations and temperature activated pressure relief devices (PRDs) designed to safely release high pressure hydrogen in case of fire. For each of these technologies, technical committees are tasked with writing requirements that are based on the best available knowledge. As with many new technologies, there are gaps in the knowledge base that need to be filled to make certain that these devices and systems safely meet end user needs.

NREL has worked closely with the responsible technical committees to define ways to close gaps in codes and standards requirements. In support of these efforts, NREL has helped in identifying technical subject matter experts for involvement in the codes and standards development process, has worked with industry to encourage active participation in the process and has supported validation testing of newly proposed test protocols. This paper addresses four validation testing programs that have been conducted through NREL/DOE support. Each of these four test programs is described in detail in the following sections. Detailed descriptions include a defined test scope which will ensure that objectives are met, thus providing the standards technical committee the needed technical basis for proposed code language. In addition to the utility of the test data, these programs also assist in building test facility infrastructure, in identifying best practices, in disseminating lessons learned and providing prequalification feedback to component manufacturers.

Codes and standards documents are living documents that are under a continual review process. SAE (Society of Automotive Engineers) document J2579, referenced in further detail later in this paper, has been published at the TIR (Technical Information Report) level. Under the SAE system of standards, this is the first level of published document, the next step being an upgrade to RP (Recommended Practice) and then to a full standard. NREL's objective in supporting research and development needs is designed to help move these documents toward becoming full standards. The role of NREL's validation test program includes involvement in the code development process, direct support of technical committees and helping to identify future development needs. One of the future needs is expanding national standards to a global marketplace and harmonization of requirements on an international basis.

2.0 Validation Testing of Vehicular 70 MPa Compressed Gas Storage Systems

2.1 Background

SAE TIR J2579 'Technical Information Report for Fuel Systems in Fuel Cell and Other Hydrogen Vehicles' was released in January 2009 [1]. In developing this standard the technical committee chose to use a performance based, systems level approach to on board storage requirements. Tests defined in the standard, including hydrogen pneumatic cycle testing are designed to provide assurance that onboard storage systems will meet end of life requirements for the vehicle. By using performance based requirements, system designers should not be limited to a specific design type; however special consideration of composite overwrapped pressure vessel (COPV) designs was used as a basis for specific tests to capture known failure modes for these vessels. Known COPV failure modes experienced in hydrogen compressed gas storage application as well as compressed natural gas (CNG) storage applications were considered. Some examples include chemical exposure tests designed to capture glass wrapped tank failures from battery acid exposure and localized fire testing designed to capture CNG tank failures in cases where the PRD did not release pressure prior to composite failure.

2.2 Scope

Test scope was divided into three parts for the purposes of the SAE TIR J2579 validation test program:

> Part 1 – Evaluation of expected service life test duration – Traditional tank level standards in the CNG market require approximately eight weeks of testing to complete the certification program. New J2579 requirements include pneumatic testing in hydrogen gas which will lead to significantly increased test time for the pressurization and depressurization cycles. The importance of using hydrogen gas is to capture transient thermodynamic effects that are particular to hydrogen and can have an effect on the performance of high pressure components.
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> Part 2 – Confirmation that known failure modes are detected by SAE TIR J2579 test sequences – CNG on road experience has shown that battery acid exposure causing weakening of glass fiber strength can result in tank failure below the normal working pressure (NWP). Tests defined as part of the durability under extreme conditions and extended usage section will be conducted to show that glass fiber wrapped tanks will not pass chemical exposure tests defined in the SAE TIR J2579 standard.
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> Part 3 – Demonstration of the Expected Service-Life Performance Test – Testing will be conducted on current production tanks that are in hydrogen service to verify the test methods defined in SAE TIR J2579.

2.3 Results

2.3.1 Expected service life duration testing as defined in Figure 1, represents the SAE TIR J2579 test requirements. If the test is automated and operated in a continuous operation mode, the total time required is determined to be 13-16 weeks. This includes three weeks of setup/soak time, four weeks of pneumatic cycle testing, six weeks of "parking performance" extended pressure hold and one day to three weeks to complete the permeation leak check and post test burst. The test time is a significant issue when considering cost and time to market for new technologies. If the test is conducted manually with a limit of eight hours of test time per day, the defined test sequences could become time prohibitive.

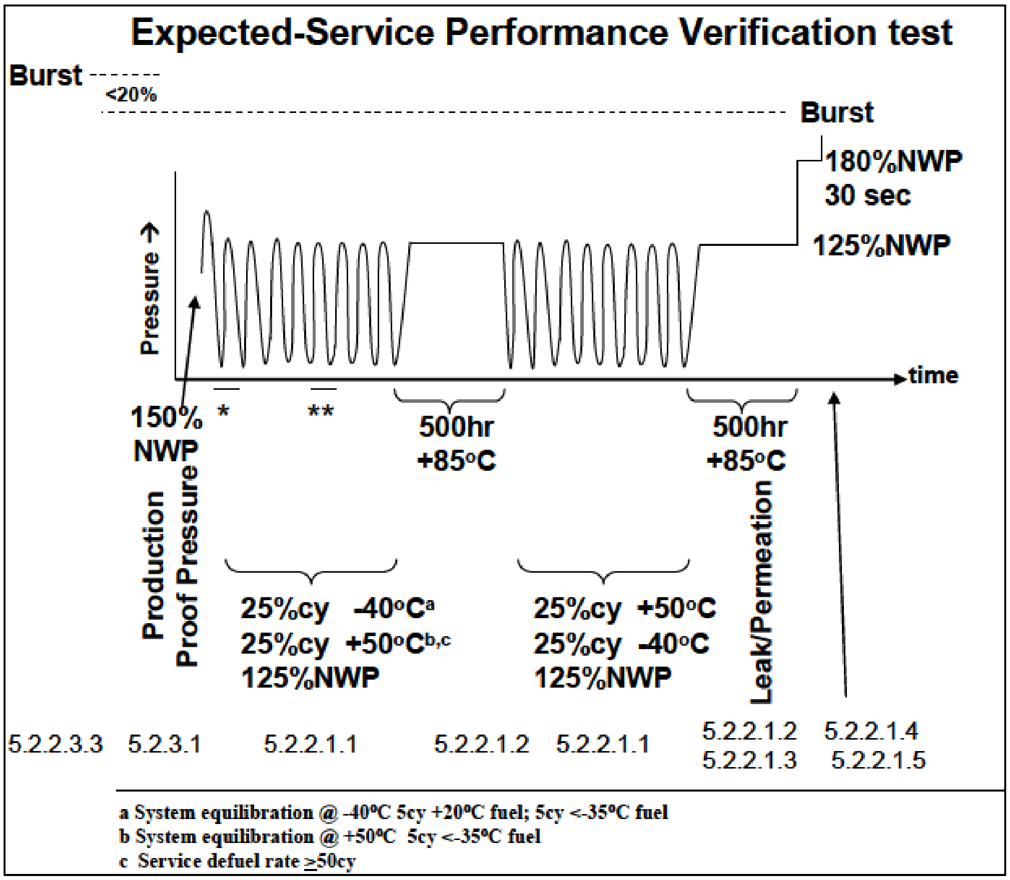

Figure 1: Expected-Service Performance Verification Test Sequence

Durability under extreme conditions and extended usage testing as defined in Figure 2 requires approximately one week. This includes one day for drop testing, four days for surface damage and chemical exposure, two days for hydraulic pressure cycling and one day for hydraulic pressure hold and residual burst.

2.3.2 The durability under extreme conditions and extended usage test was performed on a glass wrapped COPV that is known to be susceptible to chemical attack. The tank was shown to fail due to chemical exposure during the 48 hour hold at 125% NWP.

2.3.3 Four test sequences were run, including a type 3 tank, type 4 tank, type 3 system and type 4 system. Tanks representing current production models generally met the requirements of SAE TIR J2579. A notable exception was the type 4 system that did not pass the post test permeation. The leakage was attributed to liner damage in the aft end furthest from the gas inlet. This failure mode has not been experienced in service but is determined to be a realistic potential failure mode and one that the manufacturer has been made aware of. Another anomaly that occurred during cycle testing at -40°C included periodic leaks that were attributed to measured in-tank gas temperature conditions as low as -85°C. These transient conditions are a function of the pressurization/depressurization rates. Test system components should be designed to meet minimum expected temperatures, in some cases for temperatures below -40°C. Post test burst capability was consistently higher than new tank burst, in one case 10 MPa higher. This may be caused by composite wrap conditioning that is thought to occur during pressure cycling, however, based on the data quantity, it is concluded that there is no apparent degradation in burst strength as the result of the SAE TIR J2579 test sequence. A full set of J2579 validation test results can be found in NREL report number SR-5600-49867 [2].

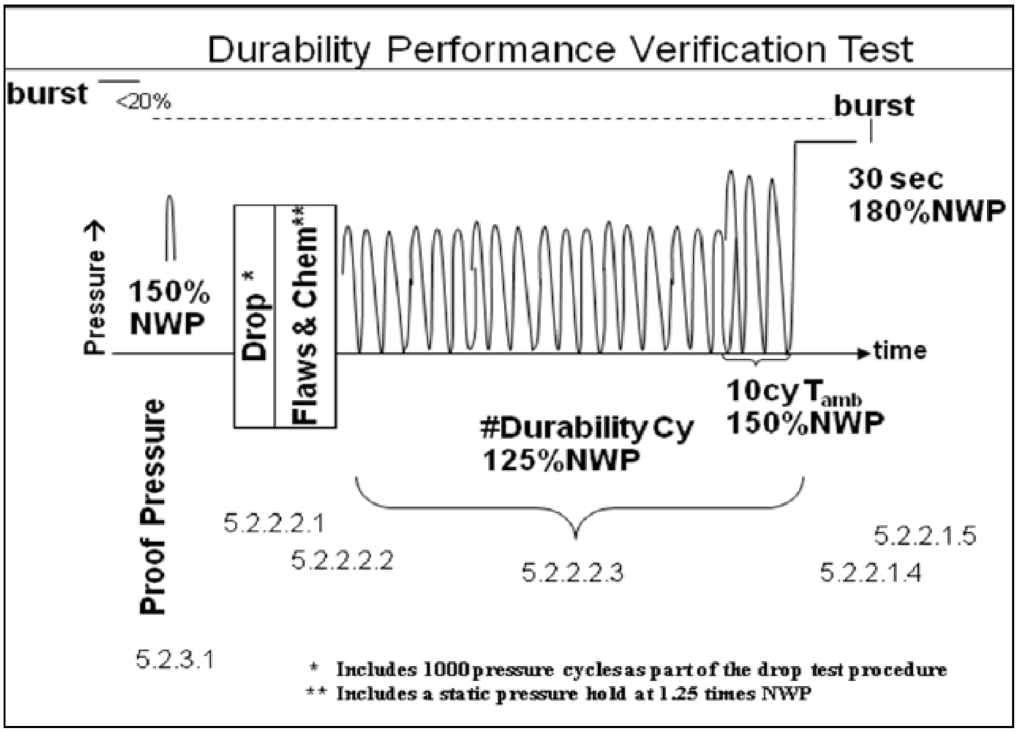

Figure 2: Durability under extreme conditions and extended usage

3.0 Flaw Testing of Stationary Composite Pressure Vessels

3.1 Background

The ASME (American Society of Mechanical Engineers) BPV (Boiler and Pressure Vessel) project team on hydrogen tanks, in developing code case 05-604 'Fully Wrapped Fiber Reinforced Composite Pressure Vessels with Non-load Sharing Liners for Hydrogen Stationary Service' identified a gap relative to flaw survivability data for COPV's. The flaw test is designed to demonstrate COPV survivability in the event of an undetected manufacturing flaw or after an in-service damaging event. These requirements have been incorporated directly into ASME BPVC Section X as Mandatory Appendix 8, Class III vessels with non-load sharing liners for gaseous hydrogen in stationary service [3].

3.2 Scope

The effect of flaws was investigated in COPV's with non-load sharing polymer liners designed for 3600 psi (25 MPa) CNG service. Flaws were machined to four depths (10%, 20%, 30% and 40% of structural layer thickness) followed by design pressure cyclic hydraulic loading of 0, 10,000 and 20, 000 cycles. Residual strength was then determined by hydrostatic burst pressure. Results are reported in terms of burst pressure ratio (BPR) which is the ratio of burst pressure to design pressure.

3.3 Results

The new tank burst pressure with no flaw was measured at 10,776 psi (74.30 MPa) or BPR of 2.99. Results for BPR vs. flaw depth and BPR vs. number of cycles are shown in Figures 3 and 4 respectively. The lowest recorded BPR was 2.13 for the 40% flaw depth and zero operating cycles. The highest recorded BPR was 3.07 which occurred for the 10% flaw with no cycles. A BPR of 3.07, which is higher that the new tank burst, can be explained by normal manufacturing variation. The effect of number of cycles on BPR shows data scatter that is determined to be a function of normal manufacturing variation, the result being little or no measurable effect. There is good correlation between increasing flaw depth and decreasing BPR. The data show that even with worst case flaw depth of 40%, there is still sufficient burst pressure margin to ensure the safety of the vessel at over two times the design burst pressure. A full report on ASME validation testing can be found in the report by Makinson [4].

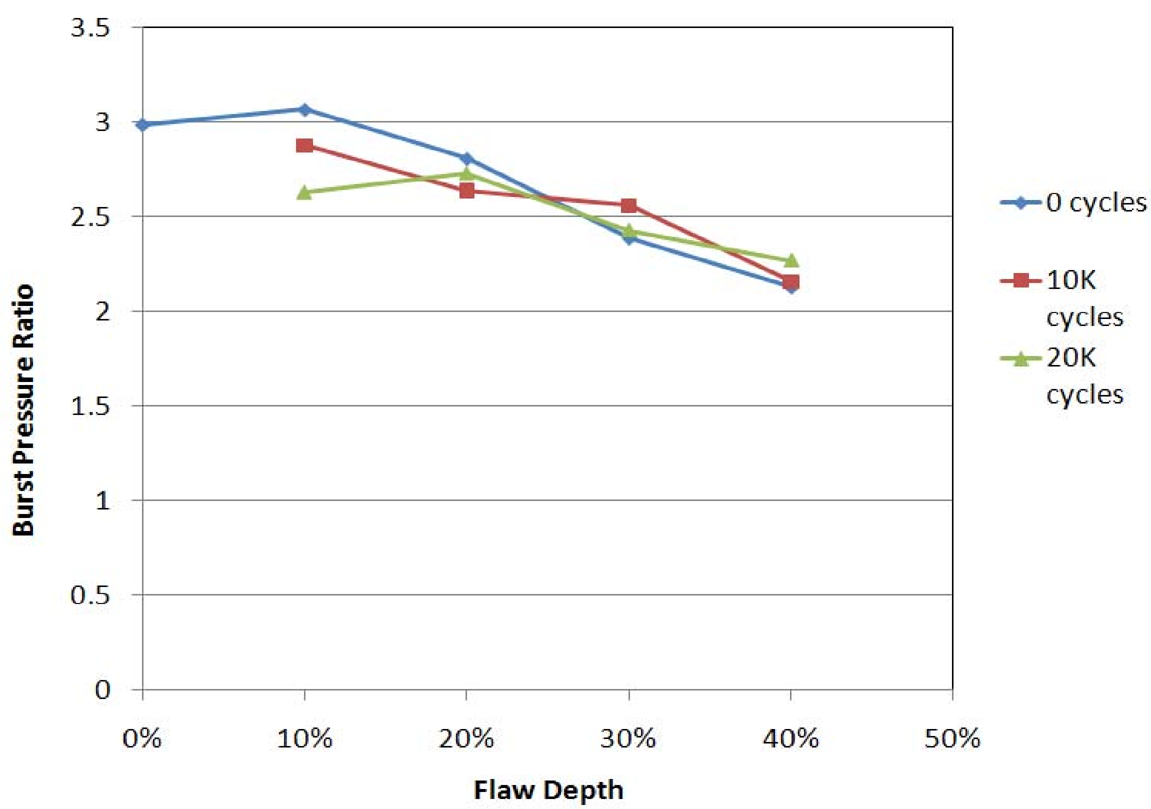

Figure 3: Burst Pressure Ratio vs. Flaw Depth

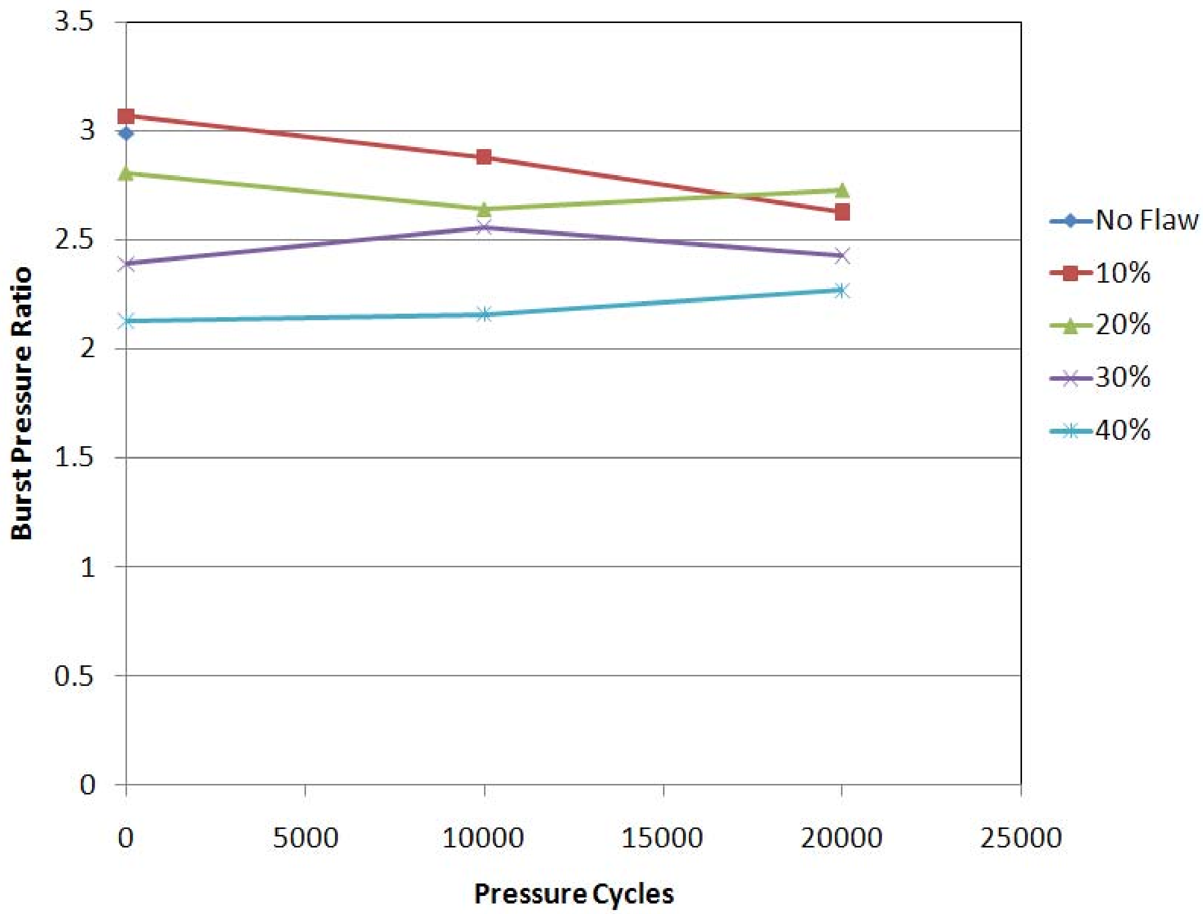

Figure 4: Burst Pressure Ratio vs. Number of Pressure Cycles

4.0 Validation Testing of High Pressure Dispensing Protocol

4.1 Background

The SAE fuel cell interface working group is developing draft document SAE TIR J2601 'Fueling Protocols for Light Duty Gaseous Hydrogen Surface Vehicles' [5]. This document will offer dispensing guidance in the form of tabulated results for fueling rates, pressures and pre-cooling levels. Thermodynamic modeling was used to determine maximum fill rates based on maintaining a peak temperature below 85°C and achieving a SoC (State of Charge) in the 90-100% range. The tables generated by analysis required validation testing prior to publishing the SAE J2601 document as a TIR.

4.2 Scope

A test matrix includes testing of type 3 and type 4 tanks, designed for 35MPa and 70 MPa fueling at representative initial conditions and dispenser pre-cooling temperatures of -20°C and -40°C. Tests are designed to 1) evaluate over-temperature conditions, 2)evaluate over-density conditions and 3) validate thermodynamic model predictions for SoC.

4.3 Results

4.3.1 Tests for over-temperature conditions were conducted twice, with averaged results reported. In only one test case, the bulk temperature exceeded the 85°C limit as specified in the draft standard. Results were used to validate thermal modeling analysis.

Table 1 – Over-Temperature Fueling Case Test Results

Pressure Class	PreCooling Temp *	Tank Capacity	Ambient Temp	Tank Initial Temp	Tank Initial Press	Tank Type	Results	
70MPa	-40 C	> 6kg	20 C	40 C	2MPa	9.8 kg IV	Max Temp = 85C	Bulk = 84C
70MPa	-40 C	> 6kg	-20 C	15 C	2MPa	9.8 kg IV	Max Temp = 83C	Bulk = 83C
70MPa	-40 C	< 6kg	20 C	40 C	2MPa	4.7 kg IV	Max Temp = 87C	Bulk = 85C
70MPa	-40 C	< 6kg	-20 C	15 C	2MPa	4.7 kg IV	Max Temp = 89C	Bulk = 87C*
35MPa	-20 C	<10kg	20 C	40 C	2MPa	9.8 kg IV (70MPa NWP))	Max Temp = 80C	Bulk = 76C
35MPa	-20 C	<10kg	-20 C	15 C	2MPa	9.8 kg IV (70MPa NWP))	Max Temp =86C	Bulk = 85C

4.3.2 Tests for over-density conditions were conducted by a two step process. The first step entailed venting a full tank to achieve an over density (cold temperature) condition in a partially filled tank followed by a fill cycle per J2601 tables. For these tests the minimum vent temperature reached was -14.9°C and the maximum SoC reached subsequent to the fill cycle was 96.9%.

4.3.3 Validation of thermodynamic modeling was conducted using six representative initial conditions with predetermined tank types, pressure ratings and initial pressures. Test results are shown in table 2. Validation testing showed good correlation with analytic modeling. In each case tested SoC values were lower than predicted, showing that there is some level of conservatism built into the modeling that was used to generate J2601 fill tables.

Table 2 – Thermodynamic Modeling Validation Results, Expected SoC

Pressure Class	PreCooling Temp *	Tank Capacity	Ambient Temp	Tank Initial Temp	Tank Initial Press	Tank Type	Results Actual Results Theoretical
70MPa	-40 C	< 6kg	30 C	30 C	15 MPa	4.7 kg IV	SOC = 89.6%, 88% SOC = 91.2%, 91.3%
70MPa	-40 C	< 6kg	30 C	30 C	15 MPa	1.4 kg III	SOC = 89.5%, 87% SOC = 90.2%, 90%
70MPa	-40 C	> 6kg	30 C	30 C	15 MPa	9.8 kg IV	SOC = 93.0%, 90% SOC = 93.1%, 92.8%
70MPa	-40 C	> 6kg	30 C	30 C	15 MPa	1.4 kg III	SOC = 89.8%, 92.6% SOC = 91.8%, 92.6%
35MPa	-20 C	< 10kg	30 C	30 C	5 MPa	1.0 kg III (35MPa NWP)	SOC = 94.7%, 98% SOC = 94.7%, 98%
35MPa	-20 C	< 10kg	30 C	30 C	5 MPa	3.0 kg IV (35MPa NWP)	SOC = 96%, 94.9% SOC = 96%, 95.7%

5.0 Hydrogen Compatibility Testing for Hydrogen Pressure Relief Devices

5.1 Background

The CSA HPRD1 (Pressure Relief Devices for Compressed Hydrogen Vehicle Fuel Containers) [6] Technical Advisory Group (TAG) has developed a performance-based test standard for PRDs used in hydrogen service. The basis of this standard was CSA PRD1, the compressed natural gas equivalent. In creating the hydrogen version of the PRD document, special consideration to the unique properties and effects of hydrogen gas service was necessary. Hydrogen gas effects on metallic materials (hydrogen embrittlement) are a known issue that is currently being intensely studied. As no universal hydrogen embrittlement test has been defined, the HPRD1 TAG elected to include design guidance and industry references only. Hydrogen effects on non-metallic materials like seals are also a known issue that stand-alone material testing does not adequately address. The complex design of PRDs, including the use of multiple metallic and non-metallic materials, led to the development of a new test designed to evaluate overall performance of the PRD in hydrogen service. The General Hydrogen Service Suitability Test was designed to simulate service conditions and evaluate overall device performance in the as-manufactured condition.

5.2 Scope

The General Hydrogen Service Suitability Test was designed to incorporate service conditions known to be worst-case for hydrogen effects. The test includes a high-temperature high-pressure hydrogen soak, hydrogen pressure cycling at low temperature, and post cycling analysis (burst, activation, visual examination). A test program was developed to evaluate the efficacy of the test procedure, as well as the performance of current PRD designs. Also, a custom-designed PRD of known “bad” material – material known to be susceptible to hydrogen attack – was included in the testing to ensure the test protocol captured poorly designed devices. The test protocol was performed on two commercially available 70MPa PRDs for hydrogen service (one eutectic-based and one glass-bulb based), as well as two custom-designed 70MPa PRDs (one of known “bad” material and one of the same design with known “good” material).

5.3 Results

The initial test protocol was performed on three devices of each of the four designs. There were numerous failures of the devices during the initial high-temperature high-pressure soak. Many of the failures were attributed to non-metallic seal failures caused by exposure to temperatures outside of their specified operating range. Additional failures occurred during the initial low-temperature hydrogen pressure cycles and were attributed to the combined high-temperature effects followed by rapid low-temperature cycling. The testing was stopped following less than 100 pressure cycles. It was determined that these premature failures were not indicative of poor device designs or materials issues, but rather a test protocol that was unrealistically harsh.

Through consultation with the HPRD1 TAG, a revised test protocol was designed. The new protocol reduced the temperature of the high-temperature high-pressure soak to be within the specified service conditions. Also, the rate of pressure cycling as well as the quantity of cycles was altered to more closely match expected service conditions as defined in other hydrogen standards (SAE J2579, CSA HGV3.1). Two 70MPa hydrogen PRD designs (one new eutectic-based and the same glass-bulb based) underwent the revised test protocol. The eutectic-based PRD design failed during the initial high-temperature high-pressure hold by eutectic creep. This failure helped validate the new protocol as this condition is realistic of service and this failure mode is specifically evaluated in traditional PRD tests. The glass-bulb based PRD design successfully completed the revised test protocol. Subsequent analysis revealed no degradation due to hydrogen service (burst pressure was equivalent to virgin, activation time was equivalent to virgin, no visual indications of hydrogen attack on metallic or non-metallic elements). This result reinforces the results from the limited hydrogen service the device has seen in real-world conditions.

The revised HPRD1 General Hydrogen Service Suitability Test has been validated as a feasible test for PRDs. The test has not been fully validated however, and is included in the current version of the HPRD1 standard as guidance only. Additional testing and evaluation is required prior to inclusion in the published standard as a requirement.

6.0 Conclusions and Recommendations

The safe use of hydrogen will require implementation of codes and standards that are based on specific knowledge of hydrogen properties. The basis for developing new requirements in these codes and standards is dependent in part on validation testing to close existing knowledge gaps. NREL's efforts through DOE support have been successful in providing validation test data to the appropriate codes and standards technical committees. This data has been used to provide a sound basis for requirements and in some cases to modify requirements based on test results. Validation testing has also served the purpose of building test facility infrastructure, in identifying best practices, in disseminating lessons learned and providing prequalification feedback to component manufacturers.

Analysis to identify research needs in the codes and standards development process is continuing as these documents progress from draft form to full working standard. Specific areas that have been identified for future work include the following:

- For onboard storage, localized fire is a known failure mode based on CNG service. New requirements need validation relative to the definition of surface area, time of localized fire and heat input qualification.
- COPV designs are based in part on single strand stress rupture data. The need for tank level stress rupture data has been identified as an industry gap.
- The addition of an SAE J2601 fill protocol for RFID communication is being considered. This fill protocol has been identified as a gap requiring validation testing.
- Further validation testing of HPRD1 requirements including further evaluation of eutectic creep

Additional gaps exist in several areas including PRD reliability in hydrogen service, dispensing nozzle/receptacle material brinelling, hydrogen effects on materials and conformity of production. The future development and adoption of codes and standards for hydrogen deployment will require continued support of these technical committee activities and the subsequent testing/analysis.

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ABBREVIATIONS

AMR	Annual Merit Review (DOE program review)
ASME	American Association of Mechanical Engineers
BPR	Burst Pressure Ratio
BPV	Boiler and Pressure Vessel
BPVC	Boiler and Pressure Vessel Code
CNG	Compressed Natural Gas
COPV	Composite Overwrapped Pressure Vessel
Cy	Cycles
CSA	Canadian Standards Association
DOE	Department of Energy, United States
EERE	Energy Efficiency and Renewable Energy, Office of DOE
HGV	Hydrogen Gas Vehicle
HPRD	Hydrogen Pressure Relief Device
MPa	Mega-pascal, pressure
NREL	National Renewable Energy Laboratory
NWP	Normal Working Pressure
PRD	Pressure Relief Device
PSI	Pounds per Square Inch, pressure
RFID	Radio Frequency IDentification
RP	Recommended Practice
SAE	Society of Automotive Engineers
SoC	State of Charge, expressed in percent of full charge
TAG	Technical Advisory Group
TIR	Technical Information Report

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