STP-PT-014

DATA SUPPORTING COMPOSITE TANK STANDARDS DEVELOPMENT

FOR HYDROGEN INFRASTRUCTURE APPLICATIONS

ASME STANDARDS TECHNOLOGY, LLC

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FOREWORD

Commercialization of hydrogen fuel cells, in particular fuel cell vehicles, will require development of an extensive hydrogen infrastructure comparable to that which exists today for petroleum. This infrastructure must include the means to safely and efficiently generate, transport, distribute, store and use hydrogen as a fuel. Standardization of pressure retaining components, such as tanks, piping and pipelines, will enable hydrogen infrastructure development by establishing confidence in the technical integrity of products.

Since 1884, the American Society of Mechanical Engineers (ASME) has been developing codes and standards (C&S) that protect public health and safety. The traditional approach to standards development involved writing prescriptive standards only after technology has been established and commercialized. With the push toward a hydrogen economy, ASME has adopted a more anticipatory approach to standardization for hydrogen infrastructure which involves writing standards with more performance based requirements in parallel with technology development and before commercialization has begun.

The ASME B&PVC Standards Committee appointed a project team to develop new Code rules in the for hydrogen storage and transport tanks to be used in the storage and transport of liquid and gaseous hydrogen and metal hydrides. Rules for gaseous storage tanks with maximum allowable working pressures (MAWPs) up to 15,000 psig (100 MPa) will be needed. Research activities are being coordinated to develop data and technical reports concurrent with standards development and have been prioritized per Project Team needs. This Technical Report has been developed in response to Project Team needs and is intended to establish data and other information supporting separate initiatives to develop ASME standards for the hydrogen infrastructure.

Established in 1880, the American Society of Mechanical Engineers (ASME) is a professional not-for-profit organization with more than 127,000 members promoting the art, science and practice of mechanical and multidisciplinary engineering and allied sciences. ASME develops codes and standards that enhance public safety, and provides lifelong learning and technical exchange opportunities benefiting the engineering and technology community. Visit www.asme.org for more information.

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ABSTRACT

Composite cylinders have been used for over 50 years in commercial, vehicle, defense and aerospace applications. New materials, processes, design approaches and applications have been incorporated during that time. The industry has maintained a high level of safety. The industry has adapted to these changes and has developed new and revised standards to address these changes and to reflect a better understanding of service conditions.

Recommendations are made that the industry:

- Continue to monitor field use and incorporate changes to requirements, standards and codes that reflect knowledge gained for composite pressure vessels,
- Use a failure modes and effects analysis (FMEA) approach to standards, using the knowledge gained from field experience,
- Develop standards for composite pressure vessels that are more performance based to improve both safety and performance,
- Address requirements using performance testing, not by using excessive safety factors,
- Use stress ratios for the various reinforcing fibers that accurately reflect their stress rupture and fatigue characteristics to achieve high reliability,
- Harmonize testing requirements where practical,
- Use qualification tests that are appropriate for the application and for the materials and design features of the pressure vessels being used, and
- Consider using fleet leader programs for new materials, designs or applications if there is likely to be a significant safety issue

To support these recommendations, history of use of composite cylinder in aerospace/defense, commercial and vehicle applications is reviewed. This includes review of applications, materials of construction; standards used and field service issues.

The use of performance-based requirements is discussed, as is the background of safety factors used for various reinforcing fibers. Recommendations are made for validation testing of materials and pressure vessels, with consideration for failure modes and effects analysis (FMEA) involving the field use of the vessels.

Cyclic fatigue and stress rupture are discussed, with examples of laboratory testing and correlation from field experience.

1 HISTORY OF SAFETY EXPERIENCE OF COMPOSITE PRESSURE VESSELS

Note: Different industries use different nomenclature for pressure vessels and their components or features. This report attempts to reflect the terminology of the industry being discussed, although terms may be used interchangeably. The ASME boiler code and the industry addressing stationary units generally use the term “pressure vessel.”. The transportation industry generally uses the term “cylinder”. The alternative fueled vehicle industry generally use the terms “container” or “cylinder”. The term “tank” may also be used. The boiler and stationary applications generally use the term “nozzle” for the end openings where the gas moves in and out, while other industries often use the term “boss”.

1.1 Aerospace/Defense Use of Composite Pressure Vessels

1.1.1 Applications

The origin of fiber reinforced pressure vessels was with the development of composite rocket motor cases in the 1950s. These motor cases were made with glass fiber reinforcement with a rubber liner/insulator. They were designed for single use, and had safety factors lower than might be used on a compressed gas pressure vessel due to the short duration of pressure and loading. Early composite motor cases included Polaris and Minuteman.

The 1970s brought the use of aramid and carbon fibers for rocket motor cases for military and space applications, including Peacekeeper, Trident D-5 and Orbus.

The technology from these early rocket motor cases was the basis for compressed gas pressure vessels that were used for applications such as aircraft engine restart or emergency floatation bag inflation. Internal volume of these cylinders was typically in the range of 100–3000 cubic inches.

The 1960s and 1970s brought the use of metallic liners. Applications included U.S. Navy life raft inflation, escape slide and flotation bag inflation for aircraft, and pressurant sources for missile systems such as Titan and Pershing and aircraft such as the F-16 and X-29. Composite pressure vessels with stainless steel liners and glass fiber reinforcement were used as oxygen containers on Skylab. Spherical pressure vessels with titanium liners and aramid fiber reinforcement are used on the Space Shuttle to contain helium and nitrogen.

Pressure vessel sizes have ranged from an internal volume of 66 cubic centimeters (4 cubic inches) to 17 cubic meters (600 cubic feet). Operating pressures typically range from (150 bar to 415 bar (2200 psi to 6000 psi), but some applications have used pressures of 35 bar (500 psi) or lower and as high as 1725 bar (25,000 psi).

1.1.2 Materials

Glass fiber reinforcements have been used since the 1950s. Aramid and carbon fiber were used for pressure vessels beginning in the 1970s, although the carbon fiber had a high specific cost (dollars per unit strength) at the time. Strength and cost improvements to carbon fiber made carbon fiber a more competitive fiber beginning in the late 1980s.

Resin matrix materials were generally epoxy or modified epoxy. Polyester and vinyl ester resins were also used.

Liners were initially made of rubber, with use in both rocket motor cases and pressure vessels. Metal liners were then developed for pressure vessels, often using aluminum and steel. Titanium and Inconel liners were used for high performance applications such as the Space Shuttle.

1.1.3 Standards

Custom and specialized military specifications were often used for pressure vessels, such as SEASYSCOMSPEC Ser 3428 and Mil-C-24604 for life raft inflation pressure vessels, and Mil-T-25363 for aircraft engine restart pressure vessels. Mil-Std-1522 was often used for space applications. Rocket motor cases have generally not been designed and built to standards. They generally are built to a custom specification, with burst and applied loads testing to verify performance.

Safety factors for carbon and aramid are often as low as 1.5 for well controlled and shorter term applications such as for missile pressurant systems and space shuttle. Safety factors for glass fiber reinforced pressure vessels are typically between 3.3 and 4.0 for military applications.

American National Standards Institute (ANSI)/American Institute of Aeronautics and Astronautics (AIAA) S-081 [39] is a more recent standard developed for composite pressure vessels used in aerospace applications, and focuses more on performance requirements and reliability than on safety factors.

1.1.4 Field service

There are likely a few hundred thousand composite pressure vessels in defense and aerospace applications, typically from 150 to 415 bar (2200–6000 psi) service pressure. Service life typically ranges from 5 to 15 years. Some applications have shorter or longer lifetimes. The life for a pressure vessel in a missile application might have a lifetime of only 1 year. The pressure vessels on the Space Shuttle have been in service for over 25 years, although not at full pressure for much of that time.

The field service has shown a high level of safety, with few, if any, field failures. One exception was the life raft application. In this case, pressure vessel ruptures did occur. This was due to a combination of a specification that did not fully meet the needs of the application, quality problems during manufacturing of the pressure vessels, and stress rupture characteristics of glass fiber. When the problem was identified, the quality problems were corrected and a new specification was prepared. There were also three pressure vessel ruptures in a military aircraft application. This occurred when glass fiber cylinders were left in service beyond the life specified and the contained pressure was higher than specified. This resulted in stress rupture failures.

1.2 Commercial use of Composite Cylinders

1.2.1 Applications

Commercial use of composite cylinders developed significantly starting in the 1970s based on the defense/aerospace technology, using metallic liners with full composite or hoop overwrap reinforcement. The initial commercial applications were for emergency breathing cylinders, such as for firemen and for mine safety, and escape slide inflation, such as for the Boeing 767 aircraft. These cylinders were of a size as to be easily portable, up to 230 mm (9 in.) in diameter and 760 mm (30 in.) long.

The size of cylinders gradually increased with time, and the number of applications increased. Cylinders might also be used for ground storage or as accumulators, such as for tensioning systems on off-shore oil platforms [16]. These cylinders might be up to 610 mm (24 in.) in diameter and 3 m (10 feet) long.

More recent applications include pressurant tanks, such as for paint ball guns, and liquefied propane gas (LPG) tanks.

Although not strictly pressure vessels, composite risers for oil platforms were developed based on technology from composite pressure vessels. These composite risers must be capable of containing internal pressure, as with pressure vessels, but must also withstand external pressure, tension loads and bending loads.

1.2.2 Materials

The first commercial composite cylinders were made of glass fiber and epoxy resin with helical and hoop windings over an aluminum liner, typically a 6061 alloy. Shortly after, cylinders with thicker liners and only a hoop wrapped were manufactured. Carbon steel liners were then introduced for hoop wrapped cylinders. Polyester and vinyl ester resins were introduced as alternatives to the epoxy resin matrix.

Aramid fibers were introduced in the early to mid-1970s. Carbon fiber was available in the 1970s, but the cost per unit strength was not competitive. Carbon fiber development resulted in a cost effective solution by the late 1980s and early 1990s. Plastic liners were introduced to the market in the early 1990s.

1.2.3 Standards

Composite cylinders were introduced into the market without the benefit of enabling regulations. Therefore, regulatory approvals in the form of special permits or exemptions from the regulations were required in order to transport pressurized composite cylinders. Industry standards were developed that served as the basis for the regulatory approvals.

ASME developed Section X for composite pressure vessels in the 1960s. This code is intended for stationary applications. This code is largely design based, with testing limited to cycle and burst testing. Section X was originally developed for glass fiber reinforcement. It required a safety factor of 5.0 for a fully wrapped cylinder with continuous fiber reinforcement. Subsequent revisions allowed the use of aramid and carbon fibers, but the safety factor has not changed.

FRP-1 (full-wrapped cylinders) and FRP-2 (hoop-wrapped cylinders) were developed by the Compressed Gas Association (CGA) in cooperation with the Department of Transportation (DOT) in the early 1970s, and have been largely unchanged since that time. These standards were developed to address the smaller transportable cylinders. Safety factors were set at 3.0 for burst and 3.33 for stress based on the use of glass fiber reinforcement for fully wrapped cylinders. Hoop-wrapped cylinders use a safety factor of 2.5 on burst. FRP-1 and FRP-2 also use performance tests for cylinder qualification rather than being fully design specific.

CFFC was developed by the Department of Transportation in 1997 to address carbon fiber reinforced, aluminum lined breathing cylinders, and was based on the exemption requests of three or four manufacturers. The safety factor was set at 3.4 based on the commonality of designs in the applications, which was necessary to meet non-shatterability (gunfire), drop and liner cyclic fatigue requirements based on the common size, materials and conditions of use.

FRP-3 was developed in the 1990s, and was based on ANSI NGV2, discussed later. It was developed by the Compressed Gas Association as document C-19 to address composite cylinders with non-loadsharing liners. FRP-3 was more performance based than previous industrial cylinder standards, and was intended to include a larger variety of materials, different design approaches, and larger sizes. It has the same safety factors as NGV2, 2.25 for carbon fiber reinforced cylinders, 3.0 for aramid fiber reinforced cylinders and 3.5 for glass fiber reinforced cylinders.

ISO 11119 was developed based on FRP-1, FRP-2, NGV2 and European standards. It is more performance based than FRP-1 and FRP-2, but it is based more on smaller cylinders as addressed in FRP-1 and FRP-2 rather than larger transportation cylinders.

Standards for cylinders used in marine applications, such as cylinders for oil platforms, risers for oil platforms and cylinders for transportation of compressed gases on ocean vessels have been developed by agencies such as Det Norske Veritas (DNV). These standards are more performance oriented than other standards discussed, and focus more on reliability than on specific safety factors.

1.2.4 Field Service

There are over 3 million high-pressure composite cylinders in use worldwide. Service pressures are typically from 140 bar (2000 psi) to 415 bar (6000 psi) for high-pressure compressed gas cylinders. These high-pressure cylinders were typically reinforced with glass fiber in the 1960s and 1970s, and with aramid fiber reinforcement in higher performance applications in the 1980s. Carbon fiber reinforcement became the reinforcing fiber of choice for many applications in the 1990s.

There are also over 2 million composite propane cylinders in use. Propane cylinders typical operate below 17 bar (250 psi), and are typically reinforced with glass fiber.

Overall, the safety record of these cylinders has been very high. The service life is typically 15 years. Inspections have been set at 3 years, but this has recently been extended to 5 years for some cylinders. These cylinders may be dropped or damaged in accidents. They are protected by pressure relief devices in the event they are exposed to fire.

Expected service temperature ranges are from –51˚C to 60˚C (–60˚F to 140˚F). These cylinders are designed for 10,000 fill cycles during their lifetime. Contained gases typically include air, oxygen, inert gases and flammable gases.

There have been a limited number of cylinder failures in service. In one instance, a glass cylinder failed due to exposure to hydrofluoric acid, which attacks the glass. In another instance, cylinder rupture occurred during filling after the cylinder had been run over by a heavy vehicle. During a controlled study, a cylinder ruptured during pressure cycling while being exposed to the elements, including temperature fluctuations and ultraviolet exposure from sunlight [33]. There have also been sustained load cracking failures of liners that used a susceptible aluminum alloy.

No aramid reinforced pressure vessels have ruptured to date, although there are fewer of them in service than fiberglass vessels. Aramid fibers are weakened by exposure to high temperatures, >204˚C (>400˚F), and by exposure to sulfuric or nitric acid. Aramid-reinforced pressure vessels are manufactured under Department of Transportation (DOT) Exemptions or Special Permits, and many are used in aerospace applications.

1.3 Composite Containers for Natural Gas and Hydrogen Vehicle Applications

1.3.1 Applications

The use of compressed gas cylinders used as fuel containers for natural gas powered vehicles has been growing since the 1970s. Italy and New Zealand were the early leaders in the use of natural gas vehicles. North America, Europe, and other areas of the world accelerated their use in the late 1980s. The vast majority of NGV cylinders used in North America are composite reinforced.

First applications were in personal and fleet automobiles and in taxis. As the market has developed, there has been a shift to heavier duty vehicles such as trucks and buses (Figure 9 and Figure 10) [12], [13], [15]. About one-fourth of all new buses in the United States are fueled by natural gas, with all using composite cylinders. Advantages of composite cylinders are their lighter weight and better corrosion resistance.

Light weight is a significant issue for buses for two reasons. First, reduction in passenger capacity may be necessary if the empty vehicle weight approaches the gross weight limit for the vehicle. Second, more low-floor buses are being put into service to accommodate passengers with limited mobility, and cylinders must be mounted on the roof rather than under the floor. Lighter cylinders lessen the impact on the vehicle structure, particularly the roof supports.

NGV cylinders currently range up to about 560 mm (22 in.) in diameter and 3 m (10 feet) in length. Service pressures are typically 207 and 248 bar (3000 and 3600 psi). Passenger vehicles normally use one or two cylinders, while buses may use between 7 and 12 cylinders, with total capacity up to about 570 scm (20,000 standard cubic feet) of natural gas.

The success of composite cylinders in the natural gas vehicle (NGV) application has led to their use in other related applications. These applications include use as fuel containers on vehicles powered by compressed hydrogen gas, tube trailers and stationary cascades.

Composite cylinders for hydrogen vehicles are similar in size to those for NGVs. Service pressures are typically 350 or 700 bar (5,000 or 10,000 psi). Early applications include passenger vehicles, buses and fork lifts.

Cascades for filling NGVs have typically been made of steel. However, due to the higher pressures used for hydrogen vehicles and due to the concern for hydrogen embrittlement, composite cylinders are generally used in cascades for filling hydrogen vehicles.

Use of composite cylinders on a tube trailer to transport compressed natural gas or hydrogen is a natural extension of their use on buses. The operating conditions are virtually equivalent, materials are compatible, and light weight and corrosion resistance are of value.

1.3.2 Cylinder Construction

NGV fuel containers are categorized according to their type of construction. This began in ANSI/ Canadian Standards Association (CSA) NGV2, and has been adopted by other composite cylinder standards.

Type 1 fuel containers are all-metal, and are typically steel, but may also be aluminum.

Type 2 fuel containers have a metal liner and a hoop overwrap that only reinforces the cylindrical portion of the liner. The liner must have sufficient strength to carry all of the pressure loads in the dome and the longitudinal loads in the cylindrical portion of the container. The liner is either steel or aluminum. The liner of a Type 2 fuel container has sufficient thickness and strength to contain the service pressure even if the composite overwrap is removed. Liner materials are generally the same as used in Type 1 fuel containers. Reinforcing fibers are typically glass and carbon.

Type 3 fuel containers have a metal liner and a full composite overwrap. The liners of Type 3 fuel containers typically carry from 15% to 25% of the load at operating pressure. The liner is typically aluminum, but austenitic stainless steel may be used in hydrogen applications. Reinforcing fiber is typically glass and/or carbon, wound in interspersed helical and hoop layers. The helical layers carry primarily the longitudinal load, and some of the hoop load. The hoop fibers carry the remainder of the hoop load in the cylindrical portion of the fuel container.

Type 4 fuel containers have a nonmetallic liner and a full overwrap. The liner is commonly a thermoplastic, with metal end bosses. Although the bosses carry load, the liner itself typically does not. Reinforcing fiber is typically glass and/or carbon, wound in interspersed helical and hoop layers. The glass or carbon fiber may be wound as a single reinforcement, or may be combined in the same winding band as a hybrid reinforcement in order to take advantage of the strength and environmental resistance of the carbon fiber and the added damage tolerance provided by the glass fiber. Glass fiber may also be wound on the outside of the fuel container as a sacrificial material, providing improved resistance to damage by abrasion, cuts, and minor impact damage.

1.3.3 Materials

Composite fuel containers for natural gas vehicles were initially reinforced with glass fiber. Carbon fiber has now emerged as the primary reinforcing materials as its structural and cost performance has improved. Aramid fiber has also been used as the reinforcing material on some fuel containers. Typical fiber properties are given in Table 1 - Typical Fiber Properties. This table reflects the tensile strength of fiber in a composite cylinder, as opposed to a strand tensile strength that is often found in the manufacturer's literature. The working strength at service pressure reflects the application of the fiber stress ratio (strength as demonstrated at burst pressure divided by stress at service pressure), which addresses reliability in a stress rupture condition.

Table 1 - Typical Fiber Properties

Property	E-Glass	S-Glass	Aramid	Carbon
Tensile strength, MPa (ksi)[1]	1500 (220)	2500 (360)	2500 (360)	3100 (450)
Working strength, MPa (ksi)[2]	430 (63)	710 (103)	830 (120)	1380 (200)
Tensile modulus, GPa (msi)	72 (10.5)	87 (12.6)	131 (19)	220 (32)
Density, g/cc (pounds per cubic inch)	2.55 (0.092)	2.50 (0.090)	1.44 (0.052)	1.80 (0.065)

1. Nominal design fiber strength in the hoop direction of a pressure vessel at minimum burst pressure.
2. Nominal design fiber strength in the hoop direction of a pressure vessel at service pressure.

Carbon fiber is often chosen as the primary reinforcing material because of its high strength, resistance to stress rupture, high cyclic fatigue life and environmental resistance. Resistance to stress rupture is a key aspect of determining factors of safety for pressure vessels (see Section 2.2.2 for further discussion) [2]. As an example, carbon fiber held at 80% of its average ultimate strength has a typical life of over 1 million years, while glass fiber would only have a typical lifetime of about 1 hour. At a 2.25 safety factor, the reliability of carbon fiber would be 0.999999 for a lifetime in excess of 100 years [3]. Glass fibers require a safety factor of 3.5 to achieve similar levels of reliability.

Carbon fiber also has excellent cyclic fatigue properties. At a 2.25 safety factor, it would have a fatigue life in excess of 1012 cycles [7]. However, this cyclic fatigue life is for a laminate with a unidirectional loading. In the cylindrical portion of the fuel container, the laminate is in tension in the longitudinal and hoop directions, and is in compression in the radial direction. In the dome region of the fuel container, bending is generally present as well, which creates shear loading between composite layers. Multi-axial loading and bending reduce the fatigue life, potentially by several orders of magnitude. Therefore, it is advisable to confirm container suitability through testing.

E-glass fiber is also used as a reinforcing material because of its impact resistance and low cost. The fatigue properties of glass fiber are not as good as those of carbon fiber, but fatigue resistance is addressed by its higher safety factor. Even at a higher safety factor, glass remains cost effective as a reinforcing fiber, although at a higher weight than carbon fiber. Glass fiber has some susceptibility to attack by acids and bases, but corrosion resistant glass fibers are available that are much more resistant to environmental attack.

Glass and carbon fibers may be commingled in the same winding band when the fuel container is manufactured. One of the key aspects in glass fiber's contribution to damage tolerance is simply the increased wall thickness it provides.

The resin matrix systems used on vehicle fuel containers are generally an epoxy, modified epoxy, polyester, or vinyl ester. There has also been some use of thermoplastic resin matrix systems. The resin system function includes holding the fiber in place over its lifetime and transmitting loads between fibers. It must withstand the operating environment over its lifetime, including exposure to fluids, ultraviolet light, and temperature extremes.

Metal natural gas fuel container materials commonly used include American Iron and Steel Institute (AISI) 4130X steel, and AA6061 and AA7032 aluminum alloys. These materials have significant history of safe use in pressure vessels. These materials are also used for liners on hoop wrapped fuel containers. Metal hydrogen fuel containers commonly use aluminum or austenitic stainless steels, particularly alloy SS316, as there is concern about hydrogen embrittlement of ferritic and martensitic steels.

Metal liners for full-wrapped containers typically use AA6061 aluminum. Hydrogen containers may also use SS316 stainless steel liners. Plastic liners for full-wrapped containers typically use high-density polyethylene (HDPE). HDPE has been used for over 30 years in natural gas distribution pipelines, and is used in gasoline tanks and containers for petroleum products. This provides assurance of its long-term suitability for use in containing natural gas fuel in an automotive environment. Experience to data also shows it to be suitable for hydrogen storage.

Bosses for plastic lined containers are typically AA6061 aluminum or SS316 stainless steel, although AA7075 aluminum and A286 stainless steel have also been used.

1.3.4 Standards

The NGV market began to grow significantly in the early 1990s in North America and other parts of the world. Standards were needed to facilitate this growth. Standards provided a means for assuring safety in a new application while providing acceptance of new materials and technologies.

ANSI/CSA NGV2 [18], CSA B-51 Part 2 [19] and ISO 11439 [20] started their development in the early 1990s. These standards considered existing industrial gas cylinders standards that were available at the time, specifically FRP-1 and FRP-2, and existing DOT regulations for metal cylinders, with consideration of the industry experience with those standards. Federal Motor Vehicle Standard 304 (FMVSS 304) [17] was developed by the National Highway Traffic Safety Administration based on an early version of NGV2, to address natural gas vehicle fuel containers.

These natural gas vehicle standards were developed with a performance based approach to allow for greater choice in materials and design technology, while maintaining or improving safety and reliability. These standards were developed cooperatively to harmonize requirements, particularly for qualification testing.

These NGV fuel container standards have been de facto standards for hydrogen vehicle fuel containers as new standards specific to compressed hydrogen storage are being developed. CSA B-51 has been updated (CSA B51-005) to include compressed hydrogen storage for both vehicles (Part 2) and ground storage (Part 3). Standards that are in the development and approval cycle for hydrogen applications include ANSI/CSA HGV2 (based on NGV2) and ISO/DIS 15869 (based on ISO 11439). The European Integrated Hydrogen Project (EIHP) was also working on draft regulations (EIHP Rev. 12b) that used ISO 11439 as a base, but has since halted their activity. As with the natural gas standards groups, the hydrogen standards groups are cooperating to harmonize requirements of their standards.

The qualification tests of these standards represent requirements for field service, addressing damage tolerance, environmental resistance and durability over a service life ranging up to 20 years or longer. The testing requirements are discussed further in Section 2.3. Meeting these qualification test requirements assures safety over the life of the fuel containers, as evidenced by field service history of cylinders build to these standards.

1.3.5 Field Service

Currently, over 6,400,000 vehicles globally have been manufactured or converted to use natural gas fuels, with about 150,000 in North America [14].

The service life of an NGV fuel container is expected to be from 10 to 25 years. During that time, visual inspections are recommended at three year intervals, or in the event of an accident, exposure to fire, or reinstallation. High use applications often have shorter primary lifetimes, but the vehicles or containers may have an extended lifetime. The lifetime of a transit bus is typically from 12 to 15 years in its first use, but may be sold for use in another jurisdiction. The lifetime of a taxi is typically from 3 to 5 years, but the fuel container may be reinstalled in another vehicle.

Fuel containers may be damaged by impacts caused by accidents, road debris or handling during shipment and installation. Proper installation and placement of the container within the vehicle, use of shields from road debris, and proper packaging and handling during shipment and installation are means for protection of fuel containers. Fires may also damage fuel containers. The use of pressure relief devices, which are generally temperature activated, provides protection from fires.

Service pressures for natural gas applications are typically 207 and 248 bar (3000 and 3600 psi). Hydrogen applications are typically 248, 350, 500 or 700 bar (3600, 5000, 7000 or 10,000 psi). Filling to 125 percent of the service pressure is permitted for temperature compensation, providing it would not exceed the nominal service pressure at the specified settled temperature. Gas temperatures of 57˚C (135˚F) are often reached during fast fill of natural gas due to heat of compression. Gas temperatures during fast fill of hydrogen may reach 85˚C (185˚F) or higher.

Ambient temperatures in vehicle applications are expected to range from –40˚C to 85˚C (–40˚F to 185˚F). Settled temperatures for the gas in the fuel container are expected to range from -40˚C to 57˚C (–40˚F to 135˚F). Use of gas for driving would typically decrease the gas temperature by about 10˚C to 20˚C (18˚F to 36˚F. Blowdown of the fuel container could drop the gas temperature as low as –130˚C (–200˚F).

The number of fill cycles is expected to be from 750 to 1000 per year in high-use applications such as taxis, fleet vehicles or buses. In the case of buses, this would mean up to 25,000 cycles over a lifetime. Personal vehicles would typically have a much smaller number of pressure cycles over its life.

Natural gas for vehicles may include impurities such as water, carbon dioxide, oxygen, hydrogen, hydrogen sulfide, mercaptan and compressor oil. Hydrogen for vehicles is generally of high purity.

Vehicle fuel containers may be exposed to various fluids that can be found in the transportation environment. These fluids can be broadly categorized as acids, bases, solvents, surfactants, fertilizers and salts. Carbon fibers are not affected by exposure to environmental fluids. Aramid and conventional glass fibers may be affected by exposure to acids and bases, although corrosion resistant glasses are available.

Containers are also subjected to shock and vibration loading during normal operation, although this operational loading is generally insignificant. Containers have also been involved in significant accidents, discussed below, that demonstrate composite cylinders designed to current standards demonstrate a high level of safety.

There have been a limited number of field failures involving composite reinforced containers in North America, where Federal Motor Vehicle Safety Standard (FMVSS) 304, NGV2, B-51 Part 2 and/or ISO 11439 standards have been required for use. Eleven field failures (ruptures) have occurred in North America since 1993. Additional failures have occurred worldwide, where ECE R110, containing a derivative of ISO 11439, is also used for qualification. Stress corrosion cracking of glass fiber caused by exposure to acid was involved in ten of the ruptures. Four tanks ruptured due to physical damage to the tank, although excess pressure may have been a factor in two of the cases. Overpressurization may have been a factor in some of the other failures. Six tanks failed due to a localized fire. No tanks have ruptured on impact.

Table 2 - Field Failures

Date and Location	Cylinder	Approvals	Failure Description
1993 Michigan	Glass fiber, aluminum liner, hoop wrap. Located in trunk of automobile.	DOT Exemption	Ruptured during filling. Mechanically induced damage of the glass fiber, combined with possible overfill.
1994 Minnesota	Glass fiber, aluminum liner, full wrap. Mounted below bed of truck.	DOT Exemption	Ruptured during filling. Stress corrosion cracking of the glass fiber.
1994 California	Glass fiber, aluminum liner, full wrap. Mounted below bed of truck.	DOT Exemption	Ruptured during filling. Stress corrosion cracking of the glass fiber.
1997 California	Glass fiber, aluminum liner, full wrap. Mounted on airport baggage loader.	DOT Exemption	Ruptured, likely due to stress corrosion cracking of the glass fiber.
1996 Alabama	Glass fiber, aluminum liner, hoop wrap. Mounted in pickup truck bed.	DOT Exemption	Ruptured during filling. Probable physical damage, UV exposure. Likely overpressurized by 20%.
1996 Texas	Glass fiber, aluminum liner, full wrap. Mounted under body of van.	DOT Exemption	Ruptured during filling. Probably severe impact damage (gouging of composite).
1996 Argentina	Glass fiber, aluminum liner, full wrap.	Argentine approval based on Transport Canada standard for transport cylinders	Ruptured during filling. Stress corrosion cracking of glass fiber.
1996 California	Carbon fiber, plastic liner, full overwrap.	NGV2-1992 (note: would not meet NGV2-1998 or later requirements)	Ruptured during filling. Probable severe impact damage to dome region from maintenance operations.
1997 Argentina	Glass fiber, aluminum liner, full wrap.	Argentine approval based on Transport Canada standard for transport cylinders	Ruptured during filling. Stress corrosion cracking of glass fiber.
1998 Argentina	Glass fiber, aluminum liner, full wrap.	Argentine approval based on Transport Canada standard for transport cylinders	Ruptured during filling. Stress corrosion cracking of glass fiber.

Date and Location	Cylinder	Approvals	Failure Description
1998 Argentina	Glass fiber, aluminum liner, full wrap.	Argentine approval based on Transport Canada standard for transport cylinders	Ruptured during filling. Stress corrosion cracking of glass fiber.
1999 Toronto, Canada	Glass fiber, aluminum liner, full wrap.	DOT Exemption.	Ruptured during filling. Stress corrosion cracking of glass fiber.
2002 Wisconsin	Glass fiber, steel liner, hoop wrap.	NGV2	Ruptured during a localized fire within a vehicle.
March 2003 Argentina	Glass fiber, aluminum liner, full wrap.	Argentine approval based on Transport Canada standard for transport cylinders	Ruptured during filling. Stress corrosion cracking of glass fiber.
May 2003 Saarbrucken, Germany	Carbon fiber, aluminum liner, full overwrap	ECE R110	Ruptured during a localized fire within a bus.
August 2005 Montbeliard, France	Carbon fiber, plastic liner, full overwrap	ECE R110	Ruptured during a localized fire within a bus, aided by a poor pressure relief design.
November 2005 Bordeaux, France	Carbon fiber, plastic liner, full overwrap	ECE R110	Ruptured during a localized fire within a bus.
2007 Washington	Carbon fiber, plastic liner, full overwrap.	NGV2	Ruptured during a localized fire within a vehicle.
2007 California	Glass fiber, aluminum liner, full wrap.	DOT Exemption.	Ruptured during filling. Stress corrosion cracking of the glass fiber from exposure to battery acid in an accident.

Stress Corrosion Cracking of Glass Fibers: Investigations were conducted to evaluate the tank ruptures due to stress corrosion cracking and seek solutions to prevent additional failures. The Gas Research Institute (now Gas Technology Institute) led the investigation and funded five organizations to perform research into the possible causes. Radian Corporation investigated failure modes in composite structures [24]. Institute of Gas Technology (now Gas Technology Institute) and Southwest Research Institute investigated protective coating systems [25]. Battelle Labs investigated the automotive service environment seen by NGVs and the corrosive fluids that could come into contact with the fuel tanks [26]. Southwest Research Institute performed a field study of NGV fuel tanks [27]. Powertech Labs evaluated damage caused by moisture and road salt [28]. Acid was identified as the cause of the stress corrosion cracking of the glass fiber reinforcement.

Testing at Powertech Labs showed that if a fuel container reinforced with glass fiber (that was not resistant to acid) was exposed continuously to battery acid, it would rupture in as few as five hours. Subsequent testing at Powertech Labs showed that corrosion resistant glass fiber can withstand exposure to acid with a pH of 1 while loaded in tension for over a year without failure.

Standards were modified in response to the above failures. In particular, the environmental test was added to address exposure to corrosive fluids. The use of carbon fiber reinforcement and corrosion resistant glass fiber reinforcement has increased as a result of these failures.

Periodic inspection has also been emphasized as a result of the field failures. In all cases where damage was known to have existed, the damage could have been identified by visual examination, if conducted in a timely manner. This applies to stress corrosion cracking, impacts, cuts, and abrasions that were involved in the failures.

The Natural Gas Vehicle Coalition, Gas Technology Institute and others have taken lead roles in promoting timely inspection and providing guidelines for inspection. The National Alternative Fuels Training Consortium, headquartered at West Virginia University, and working cooperatively with CSA International, has developed a training and certification program for tank inspectors, and the Compressed Gas Association has published guidelines for tank inspection [23]. Some manufacturers provide an inspection manual for its tanks and training for inspectors of its tanks.

Impact and Fire Resistance: A number of cylinders have been involved in field incidents, including impacts and fires, without tank ruptures occurring. This reflects favorably on both the durability of the composite cylinders and on the value of the performance based standards discussed above in identifying tests that reflect real-world durability requirements. Some of the more significant field incidents involving composite cylinders are discussed below [9]:

BRIDGE HIT Type 4 composite NGV tanks were mounted transversely on a bus that impacted a bridge at about 75 kph (45 mph) with 150 mm (6 inches) of interference. The tanks were designed for 248 bar (3600 psi) service pressure, and contained about 200 bar (3000 psi) at the time of the incident. The impact on the front tank sheared off a length of the external overwrap, and parts of three to four structural layers (Figure 6 and 7). The tank was condemned and returned to the manufacturer for evaluation and burst testing. Even with this severe impact, the tank strength was only degraded about 5%, and it still met burst pressure requirements for a new tank.

BRIDGE HIT A bus with roof mounted containers being towed impacted a low bridge. The NGV fuel containers were Type 4 with carbon fiber reinforcement. They suffered impact and abrasion damage sufficient to open a hole about 50 to 75 mm (2 to 3 in.) across in one of the containers, yet none of them ruptured.

CURB HIT Type 4 composite NGV tanks were mounted beneath a shuttle van. During a routine maintenance inspection, it was noted that the tank had been impacted, likely by hitting a curb. The bracket was moved out of position, and there was a visible impact on the dome region (Figure 8). The tank was condemned and returned to the manufacturer for evaluation. A burst test was conducted, and the tank still met burst pressure requirements for a new tank.

TANK DROP A Type 4 composite NGV tank mounted below a heavy-duty vehicle dropped from the vehicle, was dragged by the vehicle, and eventually run over by the vehicle (Figure 9). The tank was condemned and returned to the manufacturer for evaluation. A burst test was conducted, and the tanks still met burst pressure requirements for a new tank.

DEBRIS IMPACT A Type 4 composite NGV tank mounted below a vehicle was inspected and found to have debris, believed to be part of a steel shelf support, lodged in the dome region (Figure 10). The tank did not rupture as a result of the impact. The tank was condemned and removed from service.

BUS CRASH A bus equipped with Type 4 composite NGV tanks mounted below the floor was hijacked, driven away from its intended route, collided with a delivery vehicle, ran through a fence and came to rest atop parked cars (Figure 11). No damage was reported to the tanks.

ENGINE FIRE A bus equipped with Type 4 composite tanks below the floor and above the engine compartment developed a fire in the engine compartment which was unrelated to the compressed natural gas (CNG) system. The fire was hot enough to melt ceramic elements in the engine's catalytic converter. However, the PRD on a tank mounted above the engine activated as intended, and vented that tank and the others in the fuel system without incident (Figure 12).

BUS FIRE A bus equipped with Type 4 composite NGV tanks on the roof was engulfed in fire. The PRDs activated and all tanks vented safely (Figure 13).

It is significant to note the following:

- Since drop test requirements were introduced into the NGV2 standard in the mid-1990s, there have been no container failures related to collision impact.
- Since the introduction of environmental test requirements into the NGV2 standard in the mid -1990s, there have been no stress corrosion cracking failures of new designs in North America or Europe, primarily because glass fiber is seldom used as a primary reinforcement, and because corrosion resistant glass fiber is available.
- The primary cause of failure of composite-reinforced designs used for vehicle fuel tanks are associated with the effects of localized fires—these could have been prevented by improved installations.
- It may be significant to note that in three instances involving the rupture of carbon fiber containers during localized fires on-board buses, the failures did not result in the failure of any adjacent containers,

2 DEVELOPMENT OF ASME AND OTHER STANDARDS

2.1 Background Data Supports Standards Development

The background information discussed above serves as a foundation for codes and standards being developed for composite pressure vessels by ASME and other organizations. There is over 50 years of experience with composite pressure vessels in aerospace, defense, commercial and vehicle applications. Several million composite pressure vessels have been manufactured.

These composite pressure vessels have been made with glass, aramid and carbon fiber reinforcements, and with epoxy, polyester and vinyl ester resins. They have liners and bosses made from rubber, plastic, aluminum, carbon steel, stainless steel, titanium and Inconel. Liners have been one piece and welded. Configurations have been cylindrical and spherical.

These composite pressure vessels have been used to store air, oxygen, methane, natural gas, propane, hydrogen, nitrogen, carbon dioxide, xenon and a number of other gases and mixtures thereof.

The operating environments have included very cold (arctic) to very hot (desert) service. Humidity levels have ranged from very high to very low. There has been exposure to a variety of fluids and chemicals, including acids, bases, detergents, solvents, surfactants, fuels and oils. There has been exposure to sunlight and its damaging ultraviolet light. There have been numerous exposures to fires.

Tanks have been scratched, cut, abraded, dropped and shot at. They have been impacted and involved in motor vehicle accidents. They have been subjected to significant shock and vibration levels.

Pressure vessels have been designed for single use or are pressure cycled several times per day for several years. They have been use for stationary, portable or transportable applications, and have been used as equipment on weapons systems, automobiles, heavy-duty vehicles, aircraft and spacecraft. Tanks as small as 165 cc (10 cubic inches) and as large as 17 cm (600 cubic feet) have been manufactured, tested, and placed in service.

This experience base contributed to a thorough understanding of the operating environment in which the composite pressure vessels operate, and how the vessels respond to these environments. This experience base includes knowledge of how different materials used perform structurally in these environments.

The knowledge gained from this experience base has been used to develop performance-based standards with qualification tests that reflect realistic environments and operating conditions and provide a valid assessment of the composite pressure vessel's capabilities. Pressure vessels designed, manufactured and tested in accordance with these performance based standards have demonstrated safe, reliable service. This knowledge can be extended to new applications by assessing the environment and operating conditions for the new application, and adjusting test requirements accordingly.

2.2 Performance vs. Design Standards

2.2.1 General Issues

Early pressure vessels were primarily made of steel. Standards for such pressure vessel were typically design based. As such, they specified materials, type of construction and methods of calculation, but they had relatively few test requirements. This was reasonable, given the limited options for designs. For example, if a given alloy was chosen, and the manufacturing process was reasonably mature, the essential changes that could be made would be to the heat treatment or wall thickness.

Design standards also make sense for steel pressure vessels when there are a limited number of pressure vessels produced, and a full range of qualification testing is not justifiable given the ease and accuracy of design and analysis for these vessels.

Composite cylinders have a wider range of options for design and materials, and design codes can be overly restrictive. In the case of composite pressure vessels, performance-based standards can offer advantages.-Performance standards have a wider range of allowed materials and types of construction. They primarily rely on performance tests to verify the adequacy of the designs. These performance tests are based on the service conditions identified for the application, and are intended to show a "fitness for purpose".

Given the number of choices available to change the materials and construction of a composite pressure vessel, performance-based standards offer more choices for optimization of safety and performance. Choices could include different fiber materials or suppliers, hybrid construction (using two or more different fiber reinforcements), different resin matrix materials, and different liner materials. Choices could also include thickness of the liner or the composite reinforcement, or different layering sequence of the composite. An external coating or barrier material could also be used.

One issue inherent with design standards is that of safety factors. With a given material, and similar performance in the field, it would be expected that a common burst factor be used. This is reasonable given a single material and limited design options. However, with a variety of composite materials and design approaches, the issue of safety factors is less straightforward. Safety factors are discussed in more detail in the following section, followed by a section on validation testing.

The use of a performance-based standard allows validation testing to assess a cylinder's fitness for purpose rather than simply specifying a safety factor to address all issues. The testing must reflect the environment that the cylinder will be subject to, including mechanical loading, damage tolerance and fluid exposure.

It is important to note that when considering the use of new materials, performance-based validation testing must be reviewed to ensure that the new material does not have a susceptibility to a condition in the environment that was not tested for because it had not affected previous materials.

2.2.2 Safety Factors

The term "safety factor" may have more than one meaning. It is often used to be the ratio of the burst pressure to the service pressure or to the maximum expected operating pressure. The term "stress ratio" is often used with composite pressure vessels in performance-based standards because it is more narrowly defined and has more validity, as it addresses a fiber characteristic known as stress rupture.

The term "stress ratio" is the ratio of the minimum strength of the fiber, determined through burst testing of a pressure vessel, divided by the stress in the fiber at operating pressure. The stress ratio is a more accurate predictor of the reliability of a fiber reinforced pressure vessel than is the ratio of burst pressure to operating pressure. The stress ratio in composite vessels is often different than the ratio of burst pressure to operating pressure.

The difference in the ratios occurs because in composite vessels with metallic liners, the load sharing between the composite and liner is not linear with pressure. In these vessels, the stress ratio, and therefore reliability prediction, can be affected by variables including the ratio of liner and fiber modulus of elasticity, ratio of liner and fiber thickness, liner yield strength, and autofrettage pressure. Table 3 - Fiber Stress Ratios lists recommended stress ratios commonly used in newer performance-based standards for the various reinforcing materials and configurations used in the pressure vessel specifications.

Table 3 - Fiber Stress Ratios

Fiber Material	Hoop-Wrapped	Fully Wrapped	All-Composite
Glass	2.65	3.50	3.50
Aramid	2.25	3.00	3.00
Carbon	2.25	2.25	2.25

Historically, metal pressure vessels have had a 2.25–2.5 factor of safety for high-pressure transportable cylinders, as seen, for example, in U.S. DOT regulations and in ISO standards. Factors of safety in this range addressed margins for overfilling, temperature compensation during fill, material variability, and strength loss due to corrosion. Over the years, this range of safety factors has proven to be safe for metallic pressure vessels. Higher safety factors have been used in stationary pressure vessels built under the ASME boiler code.

In the 1950s, as glass reinforcing fiber was being introduced for use in pressure vessels, stress rupture was being investigated. It was seen that if safety factors typically used for metal vessels were applied to those reinforced with glass fiber, then stress rupture of the cylinders would be likely to occur during service. A higher factor of safety was required for glass fiber reinforced pressure vessels in order to provide adequate reliability and avoid stress rupture.

The higher stress ratio for glass fiber solved the problem with stress rupture, and the resultant thicker wall also provides good damage tolerance and durability. Several million glass fiber reinforced cylinders with the higher stress ratio are in service worldwide and have an excellent safety record. Over the years, new fiber reinforcements have entered the market, and there have been questions as to what stress ratios to use: should the stress ratio for glass fibers be applied to the newer fibers, or should they be evaluated on their own merit?

When aramid fibers were introduced in the 1970s, they were used in pressure vessels almost immediately. Although it was known the stress rupture characteristics for aramid fibers were better than for glass fibers, long term properties were not well established. Therefore, most commercial applications of aramid pressure vessels frequently used stress ratios similar to those of glass fibers, although some commercial and aerospace applications used lower factors. Today, the characteristics of aramid fibers are well understood, and lower stress ratios are accepted and appropriate for many applications.

Carbon fibers were available to the pressure vessel industry in the 1970s, but there was little interest in using them at that time. Carbon fiber was significantly more expensive than glass or aramid fibers, its strength was no greater than glass or aramid fibers, and it was more difficult to process. Over the years, however, the strength of carbon fibers has increased, its cost has decreased, and it has become easier to process. In addition, testing over the years has shown it to be superior in fatigue and environmental resistance.

The use of carbon fiber as a reinforcing material for composite pressure vessels grew significantly in the early 1990s, and the issue of stress ratios was brought to the forefront. Although some felt it should have the same stress ratio as glass fiber, others felt it should not be unfairly penalized for the limitations of glass fiber.

The stress ratio should be related to the properties of the fiber in question in a performance based standard. Otherwise, any stress ratio applied is arbitrary, and may be either too high or too low.

However, specifying a stress ratio only addresses stress rupture and cyclic fatigue of the reinforcing materials. In any comprehensive specification or standard, it is also necessary to specify testing which reflects the environment to which the pressure vessel is exposed. The environmental conditions should address temperature extremes, fluid and chemical exposure, and mechanical damage, at a minimum.

The issue of stress rupture can only be addressed through the stress ratio. No external coatings, hybridization of reinforcement, or design features can change the fundamental characteristics of the reinforcing fibers. Only by having the proper stress ratio can reliability be achieved. Test programs which evaluated the stress rupture of glass, aramid, and carbon fibers were conducted [1] and [4]. These references discuss the background of the test programs, offer assessments of reliability, and discuss issues related to the results. Robinson [5] offers a consistent analytical basis for comparing the reliability of the various fibers as a function of stress ratio.

It can be shown that the reliability for glass, aramid, and carbon fibers, when used at their recommended stress ratios, will all be greater than 0.999999 over the lifetime specified for composite pressure vessels (15–30 years) when held as the rated service pressure (see Section 4.1, and Figure 3–5). Otherwise stated, the risk of a pressure vessel failing due to stress rupture is less than 1 in a million over its lifetime.

Comparing glass and carbon fibers directly using Robinson, it is seen that carbon fiber is far superior to glass fiber in stress rupture. If the fibers were stressed to 80% of their average ultimate strength, glass fiber would have a typical lifetime of about 1 hour, while carbon fiber would have a typical lifetime of over 1 million years. Clearly, there is no need to increase the stress ratio of carbon fiber to meet reliability requirements.

Why do these fibers behave differently? They are fundamentally different materials. Glass is a supercooled liquid, and is subject to creep flow and surface cracking. Aramid fiber is a long-chain polymer. Carbon fiber is more crystalline in nature, and is relatively insensitive to creep or surface cracking. Carbon fiber is also virtually unaffected by exposure to acids, bases, hydrocarbon fuels or other harmful fluids.

The use of an appropriate stress ratio is also a means to address cyclic fatigue. Liber and Daniel [6] and Mandell [7] provide a look at cyclic fatigue characteristics of composite materials. When operated at their respective stress ratios, it can be expected that the structural life of glass fibers is in excess of 1 million cycles, and the structural life of aramid and carbon fibers would be in excess of 10 million and 100 million cycles, respectively, when in an idealized laminate and loading situation.

However, these estimates may not be conservative, as the laminates tested in these references are laboratory specimens rather than commercial products. Since the life of most composite vessels is less than 10,000 cycles, there is little risk of rupturing a composite pressure vessel due to cyclic fatigue.

One might question if the stress ratio should be increased to address how the reinforcing material responds to the environment. There are many ways in which pressure vessels can be protected against damage from the environment. For example, if a carbon fiber reinforced pressure vessel were sensitive to impact damage, this could be addressed in a number of ways. More carbon fiber could be added, effectively increasing the stress ratio. Hybridization with another reinforcing fiber could be used, which makes the container tougher at a lower cost. An external protective layer could also be used. If a higher stress ratio were specified to address damage tolerance, this would effectively rule out alternative solutions that may be both less expensive and more effective.

Another example of why stress ratios should not be increased arbitrarily is provided by earlier industry experience with glass fiber reinforced natural gas vehicle (NGV) fuel containers exposed to acids. An uncoated fiberglass vessel will rupture in about 5 hours at operating pressure when exposed to battery acid [28]. Increasing the stress ratio would increase the time to rupture, but no amount of additional fiber would really protect the vessel.

Simply increasing the stress ratio requirement would have effectively removed glass fiber pressure vessels from this market. The most effective way to ensure that a glass fiber fuel container can operate safely in an acid exposure environment is to provide a coating that is resistant to both acids and to abrasion, or to use a corrosion resistant glass fiber, and then to perform a qualification test that verifies its capability of safely operating in such an environment.

Composite pressure vessel standards should provide a broad range of environmental tests to ensure that these vessels will be safe. These tests should include flaw tolerance, drop, penetration (gunfire), bonfire, extreme temperature, and exposure to various chemicals. If unreasonable constraints are placed via artificially high stress ratios or inappropriate tests, promising technologies and designs may be kept from the market, and the consumer will not be well served. The appropriate stress ratios, in combination with the appropriate environmental tests, will result in pressure vessels that provide high reliability and safety.

In summary, there is data to support the stress ratios recommended above for the various reinforcing fibers. Testing to address environmental conditions and impact damage is also required in order to ensure that composite pressure vessel designs are safe. The service history of glass, aramid, and carbon fiber reinforced pressure vessels supports the recommended stress ratios as being appropriate. These stress ratios are incorporated into U.S. law for NGVs via FMVSS 304 issued by the National Highway Traffic Safety Administration of the U.S. Department of Transportation, they are incorporated in the ANSI/CSA NGV2 standard, they are in the ISO 11439 NGV fuel container standard, they are in the CGA C-19-FRP-3 Guidelines, and they are standardized in Canada under CSA B51-95 Part 2.

2.3 Testing to Validate Requirements

2.3.1 FMEA Approach to Validation Testing

Testing to validate requirements of a performance based standard must reflect the environment in which it is placed. The knowledge gained from prior experience, in combination with requirements for the specific application, can be used to assess suitability of cylinders for a given application.

This can be approached in the essence of a Failure Modes and Effects Analysis (FMEA). Consideration is given to materials and the finished cylinder. When looking at the potential failure modes, the potential causes and mechanisms of failure reflect the properties of the materials of construction and the operating conditions to which the cylinder is subjected to. The qualification test requirements then need to model what will be required in service.

The standards need to reflect the safety related failure modes, such as cylinder rupture or leakage of contents. Failure to function or cosmetic defects that do not affect safety should be considered by the customers, but are not significant to be addressed in safety standards for composite cylinders.

The following sections address testing of materials and cylinders with discussion of failure modes and effects.

2.3.2 Materials Testing

Materials including the fiber reinforcement, resin matrix, liner, and bosses must conform to specifications in order to ensure that basic strength, elongation, modulus, and other properties are consistent with what was used for qualification testing and what is required for safe service. In addition to the materials test discussed below, the test on the completed cylinder also test the basic strength and suitability of the materials of construction.

2.3.2.1 Fiber Reinforcement

Fiber reinforcement, including carbon, aramid and glass fiber, is the primary structural element of the composite container. If the strength were too low, there is a risk of rupture in service. If the modulus is lower than expected, it may expand more than expected, which could cause the container to damage its mounting structure or interfere with other components and cause damage.

Fiber strength and modulus is typically certified by its manufacturer. Qualification and batch burst testing will subsequently confirm fiber strength. Measurement of elastic expansion during proof testing of containers will confirm elastic modulus and that the proper amount of fiber is applied.

2.3.2.2 Resin

Resin matrix systems for composite cylinders are generally epoxy, modified epoxy, or polyester. Thermoplastic matrix materials have seen limited use. The resin material must maintain the fiber reinforcement in place, without unraveling, over the lifetime of the fuel container. The resin must be resistant to creep under load, resistant to degradation caused by ultraviolet exposure, and resistant to attack by fluids found in the automotive environment. These fluids include water, acid rain, battery acid, caustics, detergents, oils and solvents.

Resin components are certified to chemical composition and physical properties. Once mixed, properties such as viscosity and gel time are often conducted to confirm compliance. At the time a resin material is qualified, short beam shear tests are conducted to verify adequate shear strength so as to prevent fiber from unraveling. The short-beam shear tests are conducted on samples after being subjected to a 24-hour water boil, which simulates years of service in a high-humidity environment [38].

The glass transition temperature, T_g, is also a measure of the stability of the resin in a high-temperature environment. The glass transition temperature is normally at least 20˚C (36˚F) greater than the maximum temperatures seen in service, which would be up to 85˚C (185˚F).

2.3.2.3 Metals

Metallic materials, typically aluminum or steel, are used for liners or end bosses. These components must have adequate strength and elongation to contain pressure loads, and must be resistant to environmental fluids. If the metallic component strength was too low or the elongation not adequate, the cylinder contents could leak or the cylinder could rupture. Material suppliers typically certify the strength and elongation of raw materials, and the cylinder manufacturer may independently conduct strength, elongation, and hardness tests.

2.3.2.4 Nonmetallic Liner Materials

Nonmetallic liner materials contain the compressed gases without leakage and with limited permeation. Leakage could cause problems with combustion of flammable contents if they were allowed to collect outside of the cylinder. Leakage of greenhouse gases could add to environmental problems. Leakage of inert or flammable gases into an area where people are present could be a problem if breathing air were displaced. High-volume leaks could also cause overpressurization of rooms or buildings where pressurized cylinders are stored.

The unlined composite wall is generally not able to meet the permeation requirement without the use of a liner. Microcracking of resin during proof testing and cycling, fiber-to-matrix unbonds, and voids normally found in the matrix would typically allow a high rate of gas flow through the composite wall.

Liner materials require reasonable elongation over the full range of operating temperatures seen by the composite cylinder, including cold temperature seen during extreme pressure drops such as might occur during a blowdown of the cylinder.

Properties of nonmetallic liner materials are generally supplied by the material manufacturer. However, some properties may be dependent on the liner manufacturing process, so the cylinder manufacturer may conduct additional testing for tensile strength and elongation at the temperature extremes of the application. Material compatibility tests must also be conducted to verify compatibility with fluids in the operating environment and present in the compressed gases contained by the cylinder.

Permeation is of much lower magnitude than leakage, but must still be considered with flammable, greenhouse, and inert gases. Permeation testing must be conducted at service pressure conditions to get meaningful results. One might expect that as pressure increases, absolute permeation rate will increase. However, this is not always true, as permeation may be relatively constant as pressure increases. Permeation of natural gas at 248 bar (3600 psi) is generally limited in standards to 0.25 scc/hr per liter of water capacity, while permeation of hydrogen is generally limited to 2.0 scc/hr per liter of water capacity at 350 bar (5000 psi) and 2.8 scc/hr per liter of water capacity at 700 bar (10,000 psi).

2.3.2.5 Recommended Material Testing

Table 4 - Recommended Material Testing lists tests that are recommended for materials used in cylinders. These tests are intended to reflect properties that are required for the service conditions. Not all tests are required for all applications, and the details of the test may change according to the application. It is critical that the basis for the testing is understood, and that changes to test requirements be made if field experience indicates a need for changes. Some of the tests reflect standard measurements for materials. Other tests determine performance in harsh environments. Some of these tests may be optional, particularly when acceptable materials are specified, such as in the ASME Boiler Code. Typical test procedures are included as ANNEX A.

Table 4 - Recommended Material Testing

Test Name	Test Number	Basis
Fiber strength	A.1	Strength of each fiber lot
Fiber modulus of elasticity	A.2	Modulus of each fiber lot
Resin shear strength	A.3	Resin strength after aging
Resin glass transition temperature	A.4	High-temperature capability of resin
Metal tensile strength	A.5	Metal component strength
Metal elongation	A.6	Metal component elongation
Nonmetallic liner elongation	A.7	Elongation of nonmetallic liner material
Nonmetallic liner softening point	A.8	Softening point of nonmetallic liner material
Nonmetallic liner material compatibility	A.9	Compatibility of liner with possible contents and environmental fluids
Charpy impact test	A.10	Steel toughness and ductility
Sulfide stress cracking	A.11	Steel resistance to stress cracking form hydrogen sulfide
Corrosion tests for aluminum	A.12	Resistance of aluminum to cracking under a sustained load
Sustained load cracking	A.13	Susceptibility of aluminum alloys to intercrystalline corrosion and stress corrosion
Ultraviolet resistance of coatings	A.14	Durability of coatings required to pass environmental tests

2.3.3 Cylinder testing

Once materials have been tested and qualified, the cylinder itself must be tested to confirm that the design and the manufacturing process are correct, that the materials have been appropriately incorporated, and that the requirements of the application are met. The cylinder testing will be discussed in terms of strength and life cycle requirements, environmental requirements, and damage tolerance. There will also be discussion of conducting multiple tests on cylinders. Not all cylinder requirements will apply in all applications, but the requirements discussed are intended to cover most applications that the cylinder will be used in.

2.3.3.1 Strength and Life Cycle

Life cycle testing addresses the ability of the tank to perform its function over its full lifetime. Related tests include burst, ambient cycling/leak before burst, gas cycling and boss torque.

Burst: Burst testing addresses minimum strength considerations for the tank, margins above operating pressures, and stress rupture considerations for the reinforcing fibers.

Burst testing confirms the basic strength of the fiber reinforcement, and that the manufacturing process is consistent from batch to batch. To account for normal variation in fiber strength and manufacturing processes, the typical burst pressure might be 10–15% above the minimum required burst pressure.

The tanks need a minimum burst pressure to provide some margin against possible overfilling, exposure to higher than expected temperatures after being filled to capacity, and other unexpected events. The minimum burst ratio used in ANSI/CSA NGV2 is 2.25 based on settled pressure for natural gas at 21˚C (70˚F), which equals a ratio of 2.35 based on settled pressure at 15˚C (59˚F), as used in ISO 11439. In vehicle fuel container applications, it is expected that the maximum pressure would be 1.25 times the settled pressure. This maximum pressure accounts for heat of compression and elevated temperature exposure during service. This gives a burst margin of 1.8 over maximum expected operating pressure.

Stress rupture is a phenomenon by which the fiber reinforcement will fail over time when continuously loaded to a given level. The standards have different stress ratios (fiber strength at rupture divided by fiber stress at operating pressure) for different fibers, reflecting differences in the ability of the fiber to resist stress rupture. Carbon fiber has the best properties, followed by aramid fiber, with glass fiber having the poorest properties. At a settled pressure of 21˚C (70˚F), NGV2 requires that carbon has a stress ratio requirement of 2.25, aramid 3.0, and glass 3.5. At a settled pressure of 15˚C (59˚F) per ISO 11439, the respective requirements are equal to 2.35, 3.1, and 3.65. The intent of the stress ratio requirement is to achieve a reliability of 0.999999 for a stress rupture failure mode for each of the given fibers over the lifetime of the container.

Ambient Cycling: Ambient cycle testing addresses margins above the cyclic fatigue requirements of the application and a safe failure mode.

The cycling test at ambient temperature demonstrates the ability of the liner or container to meet its lifetime requirements without failure of the liner by leakage, and also demonstrates the leak-before-rupture characteristics of the container. The cycling requirement without leakage is typically from 15,000 to 20,000 cycles to maximum fill pressure without leakage.

When designed in accordance with safety factors used in NGV2 and ISO 11439, containers have typical fatigue lives based on the materials of construction and type of cylinder. Since the composite overwrap has a lower modulus of elasticity than a metal liner, it cycles to higher strain levels, and the liner is subject to fatigue failure. Type 2 fuel containers might typically go 15,000–60,000 cycles without leakage. Type 3 fuel containers might typically go 5000–20,000 cycles without leakage.

Type 4 fuel containers are less susceptible to fatigue failures. The liner typically has a much lower modulus of elasticity than the overwrap. As a result, the liner goes into a state of compressive stress at a very low pressure, and there tends to be little or no crack growth during pressure cycling. Type 4 fuel containers have cycled over 500,000 times to maximum pressure without leakage.

Leak-before-burst (LBB) addresses the characteristic of the liner or tank to leak before the fiber fails in cycling which could cause a rupture. Leak-before-burst testing extends to 45,000 cycles if there is no leakage, and is generally conducted at maximum working pressure or proof pressure. Standards consider that reaching 45,000 cycles without failure results in sufficient margin that the likelihood of rupture due to cyclic fatigue is negligible.

Accelerated stress rupture: Accelerated stress rupture testing is used to confirm stress rupture suitability, demonstrate resistance to creep at elevated temperature, and assess residual stresses from manufacturing.

Accelerated stress rupture testing addresses fiber and/or resin strength loss with time and temperature while under load. The test is conducted at maximum fill pressure and an elevated temperature, 65°C (149˚F) over a period of 1000 hours. Using an Arrhenius rate equation, the elevated temperature accelerates time at test by an equivalent factor of about 32, in addition to strength changes associated with elevated temperature. Combined with the elevated pressure, this test provides a second check on stress rupture failure mode, for which the stress ratios were chosen to address.

This test also demonstrates that materials will not be subject to creep over this time that could result in loss of strength. This test has screened out tanks having known problems with residual manufacturing stresses that resulted in composite deterioration in field service.

A similar test in some standards requires the container to be subjected to a high temperature creep test at 100˚C (212˚F) for 200 hours. No leakage or reduction in burst strength can result from this test.

Gas Cycling: Pressure cycling of the tank using the contained gas addresses temperature variations, permeation issues and static electricity buildup that can occur.

Pressure cycling can cause temperature buildup during filling due to heat of compression. Similarly, gas expansion will cause cooling of the interior of the cylinder. Cycling with the contained gas gives a realistic worst case of temperature extremes, particularly if testing is conducted from a starting point of the extreme high and low ambient conditions.

Permeation through the liner, along with pressure changes caused by gas cycling, can identify any problems associated with the decompression of permeated gas trapped in the liner or in the liner/end boss interface.

Flowing gas also has causes the generation of static electricity. In a metal lined tank, or a nonmetallic lined tank with metal bosses exposed to the flow stream, the static electricity will bleed off. If the plastic is exposed to a gas flow stream, and static electricity builds up without an opportunity to bleed it off, the static charge can build up to the point that it will exceed the dielectric strength of the nonmetallic liner, and will discharge through the liner, creating a leak point for the contained gas.

A cylinder is typically subjected to 1000 fill-and-vent cycles, and must not show signs of degradation such as leakage, liner cracking, or static discharge due to temperature extremes or static buildup. Filling typically occurs over 3–5 minutes, with discharge over about 1 hour, to simulate use as a natural gas or hydrogen vehicle fuel container. An appropriate duty cycle would be identified for other applications.

Boss Torque: Valves, fittings and other components are attached to the cylinder bosses or nozzles.

The boss neck, threads, boss to composite interface and, in the case of nonmetallic lined cylinders, the boss-to-liner interface, must not be damaged by installation of valves, fittings, and other components.

Typically, a torque level equal to twice the recommended torque is applied in clockwise and counter-clockwise directions to demonstrate adequate strength of the boss, its threads, and connections to the composite and liner. The boss can be designed with adequate strength and features that lock it to the composite and/or liner, preventing rotational or axial motion in the composite.

Permeation: The liner material must prevent excessive loss of contents.

Liners, particularly nonmetallic liners, may permeate the contained gases. Allowable permeation should be at a level that does not cause safety or environmental concerns. Safety concerns include possible ignition of flammable contents.

2.3.3.2 Environmental

Environmental testing addresses the ability of the tank to perform its function over its full lifetime when subjected to extreme temperatures and environmental fluids. Related tests include exposure to environmental fluids, extreme temperature cycling, blowdown and bonfire.

Environmental Fluid Exposure: Cylinders will be exposed to a variety of environmental fluids during their lifetimes.

Cylinders must be resistant to degradation when exposed to environmental fluids that may be present during transportation and use. The container must not see significant deterioration when exposed to these fluids, as leakage or rupture could occur. As broad classes, environmental fluids include acids, bases, aliphatic hydrocarbons, aromatic hydrocarbons and salts, including ammonia compounds. Battelle [26] evaluated fluids found in automotive service, likelihood of exposure, and severity of exposure, and recommended five environmental fluids as a representative "worst case" exposure.

Based on the Battelle report, the automotive fuel container industry developed tests where the container is periodically exposed to sulfuric acid, sodium hydroxide, methanol, gasoline, ammonium nitrate, a surfactant (window washer fluid) and a salt solution with a pH of 4, which is an extreme seen in acid rain. The locations on the container where fluid exposure occurs are preconditioned by impacting with a pendulum having a mass of 15 kilograms (33 pounds) and energy of 30 Newton-meters (22.1 foot-pounds). This preconditioning demonstrates that the composite surface and any protective coatings are either durable or not affected by the environmental fluids. This impact simulates the energy of a 38-mm-diameter (1.5-in.-diameter) stone impacting the container at 100 kilometers per hour (60 miles per hour).

The container is cycled 3000 times from nearly zero pressure to maximum service pressure, then held at maximum service pressure until a total of 48 hours have elapsed. Pressure cycling during fluid exposure tends to open up resin matrix cracks and helps the fluids to penetrate the composite. The container cannot leak or rupture, and must have remaining strength to withstand a pressure 1.8 times service pressure without rupturing. This test protocol was developed such that a cylinder design that had failed in service would fail the test, while designs that have been successful in service would pass the test.

Carbon fiber reinforcement is virtually unaffected by corrosive fluids [10]. Conventional glass fibers are affected by exposure to acids and bases [8], although new corrosion-resistant glass fibers have significantly better resistance to corrosive fluids. Testing of a fuel container reinforced with conventional glass fiber from which the protective coating was removed failed after only 3485 cycles in the area exposed to the salt solution with a pH of 4. Even if the glass container does not rupture, breakage of some fibers may cause localized strain increases that could lead to premature fatigue failure of a metal liner and subsequent leakage.

Fluid Exposure on Liner: The liner may be exposed to impurities in the contained gas.

The liner may fail and release gas if the contents contain impurities for which the cylinder is not intended. Natural gas, for example, may have impurities from the oilfield or from compressors. The liner material for a CNG tank should be tested for exposure to such chemicals, including water and carbon dioxide (forming carbonic acid), and hydrogen sulfide. Methanol, which can attack aluminum, is sometimes added to natural gas. The liner might also be exposed internally to propylene glycol, compressor oil and hydraulic fluid, and externally to the fluids to which the composite is exposed. Pressure cycling could be conducted while exposed to these fluids. Stress cracking resistance tests in accordance with American Society for Testing and Materials (ASTM) D1693 may also be conducted, to confirm generally compatibility with these fluids. Other applications should determine what impurities could come into contact with the liner and test accordingly.

Extreme Temperature Cycling: The container may be cycled at hot and cold temperature extremes.

The container may be pressure cycled at hot and cold temperature extremes depending on its application. Cold temperature limits are commonly 40˚C (–40˚F) for commercial applications and 50˚C (–65˚F) for military applications. If the liner is not suitable for the required temperature ranges, a leak could occur, resulting in loss of contents, and possibly fire or explosion if the contents were flammable. If the composite reinforcement is not suitable for the require temperature ranges, rupture of the cylinder could occur.

Cycle testing at extreme temperatures is a common method to assess suitability at extreme temperatures. Vehicle fuel containers are typically cycled for a number of cycles, reflecting one-fourth of the cylinder life, to maximum service pressure at 85˚C (185˚F). They would then be cycles an equal number of cycles to service pressure at –40˚C (–40˚F).

An alternative for hot cycle testing is to combine high temperature with high humidity. A representative test is to cycle at 65˚C (149˚F) while at 95% relative humidity. Spraying with a water mist is often substituted for controlled humidity.

High Temperature Creep: Cylinders materials must be stable over their temperature range.

The materials of construction may be exposed to extreme temperatures, and should not creep under these conditions. If the liner were to creep, a leak might occur. If the resin softens, the strength of the laminate could be compromised.

Fast Fill: Cylinders may be filled rapidly during their lifetime.

Cylinders may be filled at high mass flow rates during their service life. If the liner, boss, or seals have problems with the resulting stresses at extreme temperatures, leaks could occur. This test is generally not required in standards, as it has not been an issue in current field service. However, it could be a useful test in some applications.

At cold temperatures, some of the components may have changed dimensionally compared with adjacent materials, so loading may change on pressurization or seals may not seat out properly. At hot conditions, the heat of compression may expose materials to gas temperatures near the upper use temperature. From ambient conditions, natural gas typically might reach temperatures to 57˚C (135˚F), and hydrogen might reach 85˚C (185˚F).

Fast fill times should reflect actual conditions for the application. For vehicle fuel tanks, this could be a full fill in 3–5 minutes.

Blowdown: A container may be vented rapidly during its lifetime.

Rapid venting, or blowdown, will cause extreme cold conditions in the cylinder, particularly if the venting occurs when the cylinder is being held at the cold limit of ambient conditions. Valves may be designed to limit venting rates. If the liner cracks due to brittleness or temperature-induced stresses, the contents could leak, with possible fire or explosion if the contents are flammable.

Gas temperatures of –135˚C (–210˚F) or colder can occur during blowdown. Cylinders should be able to withstand such conditions without developing cracks or leaks. Blowdown tests are normally not required by composite cylinder standards, but it can be a useful test to run if plastic or metallic components are subject to embrittlement in cold conditions.

Bonfire: Cylinders may be exposed to the action of fire.

Cylinders may be exposed to fire, which could be localized, partially engulfing, or fully engulfing. There is significant risk of a cylinder rupture in a fire. Pressure relief devices are used to protect the cylinder, but the cylinder must have some degree of fire resistance itself.

In a fire situation, aramid fibers will char or burn, and glass fibers will melt. Even though carbon fiber is not significantly affected by fire, the resin will burn out of the composite and weaken the structure significantly. Aluminum can lose significant strength in a fire. Steel is less affected in a fire, but excessive internal pressure caused by elevated temperature could rupture a steel pressure vessels.

Thermally activated, quick response pressure relief devices are the most effective way to protect a composite cylinder in a fire situation. These devices will activate somewhat independently of internal pressure, thereby protecting partially filled cylinders. Pressure activated relief devices, such rupture disks, are sometimes used, particularly for steel pressure vessels. However, they may not effectively protect partially filled cylinders, as cylinder strength drops below the level of contained pressure prior to the disk rupture pressure is reached.

Relief devices for steel pressure vessels are generally specified independently of the cylinder. However, since composite pressure vessels are more significantly affected by fire, and because thermally activated relief devices are used that have different activation times and flow rates, the pressure vessel and relief device are typically tested as a system for industrial and vehicle use.

Most fires are partially or fully engulfing, so testing has focused on this scenario. The fuel container is placed in a bonfire in conditions of partial and full exposure and at 25% and 100% of service pressure. Flame temperatures measured by thermocouple typically reach from 425˚C (800˚F) to over 815˚C (1500˚F). These bonfire tests demonstrate that a properly designed and installed pressure relief device will vent the container before it ruptures due to overpressure or material degradation.

Venting typically initiates within 1–3 minutes using a thermally activated device. Once venting initiates, pressure typically drops by about half in about 1 minute, and within 10 minutes the container is almost empty. The time to empty will increase as the cylinder volume increases relative to the flow capacity of the relief device and vent lines.

Multiple devices may be required for each container to get higher flow rates or to get more release points that could be exposed to a fire. Without a relief device, a composite fuel container might rupture within 5–20 in a fire. Pressure relief devices for vehicle applications are compliant with ANSI/ International Accreditation Service (IAS) PRD 1 [21].

Highly localized fires, such as the flame from a propane torch, are less likely to cause a rupture, as they degrade the strength of the composite in a very small area. However, a somewhat localized fire, a larger area might be degraded, yet the area is small enough that there is not a nearly pressure relief device, so a rupture could occur.

When two or more cylinders are connected together, there are issues of how to manifold the cylinders and the relief device system. This affects how many cylinders vent from the time a single cylinder is affected by fire, and the pressure in the tanks vs. time while venting.

2.3.3.3 Damage Tolerance

A well-designed and manufactured cylinder provides damage tolerance in the field. Damage in the field can occur from flaws such as localized cuts and gouges, blunt impact such as occurs if the cylinder is dropped during shipment, blunt impact of higher energy such as occurs in a collision and high-energy localized impact such as can be modeled by gunfire.

Flaw Tolerance: Flaws represent typical field damage.

Flaws such as cuts, gouges and abrasion occur during normal field service. These flaws are generally not so severe as to cause an immediate problem, but the composite or liner may be affected over time. Cuts are generally more severe than abrasions, since there will be a sharper stress concentration that is more likely to grow with time.

One possible failure mode is rupture of the cylinder if the flaw grows extensively, which is unlikely with the current cylinders in service. Another possible failure mode is leakage, as the flaw decreases the wall stiffness locally, therefore a liner would see higher localized strain, which could result in flaw growth and allow contents to leak.

To test for flaw tolerance of fuel containers, flaws up to 0.75 mm (0.030 in.) deep by 200 mm (8 in.) long and 1.25 mm (0.050 in.) deep by 25 mm (1 in.) long are machined into the outside of the container, which are representative of flaws seen during visual inspection of these containers. The container is then cycled 15,000 times to 125% of service pressure. The tested container may not leak or rupture during the first 3000 cycles of this test, and may not rupture during the remaining cycles. The size of flaws would need to be evaluated for other applications.

Impact: Impacts, including drops and collisions, may occur during transportation and use.

Low-energy impacts, such as dropping a cylinder, may occur during field usage. Immediate rupture is not likely, but leakage or rupture could occur with time, as was possible for flaws.

Cylinders that are human-portable, generally 60 liters (3660 cubic inches) water capacity or smaller, might be dropped while being carried or transported, and are more likely to be subject to careless handling than larger cylinders. If designed only to contain pressure, they generally have thinner walls than larger cylinders.

Intermediate size cylinders may be subject to drops during transportation, but are more likely to be handled with equipment that decreases the likelihood of drop or to be permanently mounted so as to limit the possibility of dropping.

Large cylinders will be transported with powered equipment or will be permanently mounted, such as on a tube trailer. Likelihood of dropping is small, but it could still be impacted, say by a fork lift.

Testing for resistance to damage from low-energy impacts for a human-portable cylinder typically involves drop in various orientations, including horizontal, vertical and at a 45-degree angle. The height is typically 3 meters (10 feet). The horizontal drop may be onto an angle iron placed on the ground with the corner up. All drops are onto a flat, hard surface, such as concrete. Cycles to service pressure are conducted with no leakage or rupture allowed. Burst testing may be conducted, requiring the cylinder to maintain a given percent of the initial burst pressure requirement.

Testing of intermediate size cylinders typically involves drop in horizontal, vertical, and 45-degree orientations onto a flat, hard surface such as concrete. The test height is 1.8 meters (6 feet). Cylinders must be cycled to maximum service pressure without leakage or rupture, and up to 45,000 cycles without rupturing.

The dome region near the cylindrical section of a composite cylinder is generally more sensitive to drop impact damage, as it is generally thinner. This region can be hit of 45-degree and horizontal drop tests, particularly on a rebound. Composite cylinders may have additional composite, or energy absorbing materials in this area to protect against impact damage. Testing has shown that as wall thickness of a composite cylinder increases, such as with increasing service pressure or larger diameter, the cylinder is more robust, and less likely to be damaged in a drop test. Testing and field events also show that if a cylinder is pressurized, the wall is stabilized and less likely to deflect. Therefore, it sustains less damage than if the cylinder is unpressurized.

Large cylinders have not been subjected to drop testing. A test where the impact is of a defined energy level and placement is more practical than a drop test for large cylinder, and more representative of the type of impact to which it could be subjected.

Cylinders that are mounted on vehicles, either as fuel containers or as compressed gas containers in a tube trailer, are more likely to be subjected to high-energy impacts than small portable cylinders. Cylinders subjected to high-energy impacts could leak or rupture.

Vehicles with compressed natural gas fuel containers must certify compliance with FMVSS 303, which requires collision tests, including a frontal barrier crash, a rear moving barrier crash, a lateral moving barrier crash and a moving contoured barrier crash. Front and rear barrier crashes are conducted at 48 kph (30 mph), while the lateral barrier crash is conducted at 32 kph (20 mph). The contoured barrier crash is conducted at speeds up to 48 kph (30 mph), at any point and angle. Compressed gas is not allowed to escape following these impacts.

In a typical test in accordance with FMVSS 303 [22], two Type 4 fuel containers were subjected to a rear impact crash test. This testing was conducted by the Transportation Research Center, under contract to DOT National Highway Traffic Safety Administration (NHTSA) [11]. The containers were mounted in the trunk of a 1989 Chrysler New Yorker. The impact velocity was 47 kph (29.3 mph). The containers were not damaged by the test.

Such an impact might represent a vehicle impact for cylinders used in tube trailers. However, it would be recommended that impact testing on cylinders in tube trailers be conducted on them in their mounting features and with protection that would be afforded by the trailer assembly.

Penetration: The wall of cylinders may be penetrated during an impact.

Cylinders may be subjected to an impact that has sufficient energy to penetrate the wall of the cylinder. If the penetration is through the wall, venting of the contents will occur. The cylinder could rupture when subjected to this level of impact. It is generally accepted that for a penetrating impact, leakage will occur, but that rupture and fragmentation should be avoided.

The resistance to rupture and fragmentation during a penetrating impact is generally evaluated by means of a gunfire test. A container is pressurized with air, nitrogen or the gas to be contained and impacted by a 7.62 mm (30-caliber) armor piercing projectile having a velocity of 850 meters per second (2800 feet per second). The container must show no signs of fragmentation.

Hybridizing glass or organic fibers into a laminate where carbon is the primary structural fiber increases damage tolerance. As composite walls get thicker, which occurs with increasing diameters and pressures, the cylinders also become more resistant to impact damage.

2.3.3.4 Test Interaction

Standards have traditionally set up qualification test requirements so that a cylinder is subjected to a single test. For example, in a drop test, one cylinder could be used for the horizontal drop, one for the vertical drop, and one for the 45-degree drop. After drop, a cycle test is performed per the cycle test requirements of the drop test. Similarly, cylinder passing the ambient cycling test requirements does not need to pass the burst test requirements.

Using a single cylinder for each test is based to some extent on the philosophy that a cylinder would not be subject to multiple "significant" events in field service. If a damaging event occurred, the cylinder would be removed immediately or after its next inspection cycle. Conducting a single test on a cylinder also helps the manufacturer to understand the response of a cylinder to certain damaging events, and therefore helps to identify what design or material changes could be made to improve overall performance.

Some qualification tests are destructive, so no additional tests can be conducted. Examples include the burst test, bonfire test and penetration test.

Manufacturers frequently do conduct multiple tests on a given cylinder. For example, flaws may be cut in a cylinder, and the tank is cycled as required for this test. If the tank is then able to meet the full ambient cycle test requirements, and the minimum burst test requirements, the one cylinder has successfully passed three qualification test requirements. Similarly, a single cylinder may be used to do the horizontal, vertical and 45-degree drop tests, followed by cycle and burst testing. Cylinders are generally able to pass multiple test requirements of this sort.

There is a basis for requiring a cylinder to meet multiple test requirements, such as to address the case of low-level damage that may not be immediately obvious in an inspection, or the need to maintain some minimum burst strength after pressure cycling. These are, to an extent, incorporated into current standards, e.g., the cycle requirements incorporated into the drop test and the flaw tolerance test.

Some standards are proposing to incorporate the concept of multiple tests on a single cylinder [36] to show end-of-life capability. Tests such as boss torque, gas cycling, extended high pressure hold at elevated temperature and permeation were combined to address full service life performance. Tests such as drop, flaw tolerance, environmental fluid exposure, and pressure cycling were combined to address durability under extreme conditions and extended usage. Following these combinations of tests, the residual strength requirement of 1.8 times service pressure is intended to reflect strength at end of life sufficient to take a maximum fill pressure. An accelerated stress rupture test addresses reliability in regards to stress rupture over a lifetime. Even when a standard requires multiple tests on a single cylinder, it is advantageous to perform some single tests to understand structural response to the test.

2.3.3.5 Recommended Cylinder Performance Testing

Table 4 lists tests that are recommended for cylinders used in typical applications. These tests are intended to reflect performance requirements for field service. Not all tests are required for all applications, and the details of the test may change according to the application. It is critical that the basis for the testing is understood, and that changes to test requirements be made if field experience indicates a need for changes. Typical test procedures are included as ANNEX B.

Table 5 - Recommended Cylinder Qualification Testing

Test Name	Test Number	Basis
Burst	B.1	Confirm strength of cylinder and stress rupture reliability
Ambient cycling	B.2	Confirm cycle life without leakage
Leak before break	B.3	Confirm failure mode is by leakage or with margin of safety
Accelerated stress rupture	B.4	Confirm stress rupture characteristics, confirm limited residual stresses
Gas cycling	B.5	Confirm that there will be no buildup of static electricity or leakage in plastic liners that will be detrimental to the cylinder
Boss torque	B.6	Confirm the strength of the boss neck and threads, and the interface between the boss, plastic liner and composite
Permeation	B.7	Confirm that there will be no detrimental loss of cylinder contents due to permeation.
Environmental fluid exposure	B.8	Confirm environmental stability of composite
Liner fluid exposure	B.9	Confirm compatibility of liner with contents
Extreme temperature cycling	B.10	Confirm high- and low-temperature capability of liner and composite
High-temperature creep	B.11	Confirm no creep at elevated temperature
Fast fill	B.12	Confirm suitability to handle pressure rise and high temperature of fast fill
Blowdown	B.13	Confirm suitability to handle pressure drop and low-temperature of blowdown
Bonfire	B.14	Confirm ability of relief device to vent cylinder without rupturing in a fire
Flaw tolerance	B.15	Confirm ability to cycle without leakage or rupture in the presence of flaws
Impact	B.16	Confirm ability to cycle without leakage or rupture after a low-energy impact
Penetration	B.17	Confirm no rupture when penetrated by gunfire

2.3.3.6 Qualification by similarity

Qualification testing for a composite cylinder has been discussed above. It is common to make new products that represent changes from the original cylinder design. This change could be in materials, dimensions or service pressure. Standards allow qualification by similarity for design changes. A table is generally used to identify required tests as a function of the type of design change. Table 6 identifies tests required for various design changes.

The fundamental question to ask when making a design change is, “what testing is required to evaluate the change in pressure vessel performance that results from a given design change?” The recommended qualification tests for a given design change in Table 6 are intended to answer that fundamental question.

Table 6 - Qualification for Design Changes

Test No.	Test	Length <= 50%	Length > 50%	Diameter <= 20%	Diameter >20% <= 50%	Liner Thickness >20% or Manufacture	Liner Material	Equivalent Fiber	Service Pressure <=20%	Service Pressure >20% <=60%	Boss-to-Liner Interface	Resin Matrix
B.1	Burst	X[1]	X[1]	X[1]	X	X[1]	X[1]	X	X[1]	X	X[1]	X[1]
B.2	Ambient cycle			X[1]	X	X	X	X[1]	X[1]	X	X[1]	
B.3	Leak before burst			X	X	X	X	X	X	X		
B.4	Accelerated Stress Rupture[1]				X[5]		X			X[5]	X	X
B.5	Gas Cycling						X				X	
B.6	Boss Torque						X				X	X
B.7	Permeability					X	X			X[4]		
B.8	Environmental fluid						X	X				X
B.9	Liner fluid						X					
B.10	Extreme temperature cycling						X					
B.11	High-temperature creep						X					X
B.12	Fast fill						X				X	
B.13	Blowdown						X				X	
B.14	Bonfire		X[2]		X[2]					X		
B.15	Flaw tolerance[1]				X							X
B.16	Low-energy impact (drop)		X		X		X	X		X		X
B.17	Penetration (gunfire)				X[3]				X[4]	X[4]		X

Notes:
1. Only one cylinder required for design change.
2. Not required if cylinder volume decreases and same PRD is used.
3. Not required if diameter increases.
4. Not required if pressure increases.
5. Where burst pressure to test pressure ratio of design variant is over 20% greater than the same ratio for the approved design.

These changes are often identified as being based on an “original design,” and not from another “design change.” This is to avoid the possibility that by basing a design change on another design change, a test would that should be run would be missed.

A more appropriate application of a design change test matrix is to base the required tests for a change on what tests have been conducted on other cylinders. This may seem to be a subtle difference, but can have a significant impact on the number of tests required for a design change.

As an example, consider that the change from design “A” is to increase cylinder length by 40%. From the design change chart, one is only required to conduct a burst test on design “B”. If one makes a second design change from the original design that increases cylinder length by 80% for design “C”, then one is now required to conduct a burst test, bonfire test, and drop test.

Now consider a third design change, design “D”, that is 60% longer than the original design “A”. If one considers only changes from an “original design”, one is again required to do a burst test, bonfire test, and drop test. If one considers the broader question of how the design change varies from cylinders on which tests that have been conducted, one can consider that only burst, bonfire, and drop would be required based on a change from design “A”, but that by virtue of having conducted bonfire and drop tests on design “C”, and that one is changed less than 50% from the test conducted for design “C”, then one is only required to conduct a burst test on design “D”.

This logic is particularly appropriate given that design “D” length is between that of design “A” and design “C”, therefore bounded by these successful results, and it is logical that design “D” would pass these tests also, and therefore no additional bonfire or drop test is necessary.

Consider now the case where design “C” is a change from design “B”. Although design “C” is less than a 50% change in length from design “B”, and while no bonfire test or drop test would be required for a length change for less than 50%, design “B” did not conduct these tests, so it cannot be used as a basis for a design change where these tests are involved.

To the extent that a test could be considered appropriate for qualification by similarity, it is also appropriate to be considered as a subscale for qualification of an original design. For example, since the liner fluid exposure test is only rerun if the liner material changes, it is appropriate to use a cylinder of different size or pressure for qualification.

If there is a question as to what qualification testing is required for a design change, or for a design change that is not listed in the design change table, then there should be a consensus of the manufacturer and the inspection agency as to the appropriate testing requirements. The fundamental question of what testing is required to evaluate the change in performance that results a design change must be considered.

2.4 Batch and Acceptance Testing

Production of pressure vessels commences following characterization of materials and qualification of the pressure vessel design. Typically, one or more pressure vessels are destructively tested from each production batch of up to 200 cylinders to confirm strength. All pressure vessels are inspected and subjected to acceptance tests that confirm compliance with design intent.

One composite pressure vessel from each lot is typically subjected to a burst test. This confirms the strength of the reinforcing fiber and the consistency of the manufacturing process.

One pressure vessel is typically subjected to a cyclic fatigue test to confirm that the liner properties and manufacturing process are in accordance with specification. However, the industry is moving towards testing only one out of every 5 or 10 batches, or removing the batch cycle test entirely if the margin of safety is high.

The concept of batch burst and cycle testing is based on processing the cylinders in the batch in the same manner, with the same equipment, and within a reasonably close time period. Samples are typically picked at random out of the finished lot of cylinders, or a cylinder with a known defect is picked for burst or cycle testing. One factor that sized lots at 200 cylinders was the capacity of heat treating ovens used for steel cylinders.

The concept of a batch loses some continuity when very large cylinders are manufactured. It may take several days or weeks to manufacture a set of cylinders. Reinforcing fibers may be used from more than one fiber lot. In this case, it makes more sense to consider periodic, versus batch, testing of cylinders. This will show that the process is under control, even though the units manufactured are not strictly in a single batch.

A pressure vessel might be burst near the start of a production run, then after a given number of units have produced. The use of periodic burst testing requires continued emphasis on material control and nondestructive means to evaluate the pressure vessel integrity, such as proved by the elastic expansion testing.

Each composite cylinder is visually inspected and subjected to a hydrostatic proof test pressure that is typically 150% of service pressure during acceptance testing. The hydrostatic test, or proof test, confirms a minimum level or strength of each cylinder. More importantly, expansion of the cylinder is measured during the test.

Permanent expansion measures growth in the internal volume of the cylinder at zero pressure after the hydrostatic pressure test. Permanent expansion has typically been measured for metal cylinders to confirm that yielding did not occur during proof testing, which in turn confirmed that the wall thickness and material properties were in accordance with specifications.

Composite cylinders with metal liners are generally subjected to an autofrettage pressure cycle that yields the liner, causing it to be in compression when pressure is released. This autofrettage process reduces the mean stress in the liner, increasing its cyclic fatigue life. Measuring permanent expansion of a metal lined composite cylinder may confirm that it has been subjected to an autofrettage cycle.

Elastic expansion is a measure of elastic growth between zero pressure and the hydrostatic test pressure. Measuring elastic expansion gives a measure of the amount of composite reinforcement on each cylinder, confirming that the cylinder was manufactured with the proper number of layers and winding circuits per layer, and that the fiber yield is correct. This test is arguably the single most important measure of proper construction of a composite cylinder.

Volume of a cylinder is approximately $V = \pi r^2 L$. The elastic expansion of the cylinder is then represented by the formula $V + \Delta V = \pi (r+\Delta r)^2 (L+\Delta L) = \pi r^2(1+\varepsilon_\theta)2\ L(1+\varepsilon_L)$. This simplifies to $\Delta V = V (2\varepsilon_\theta+\varepsilon_L)$ if higher-order terms are ignored. If fiber is missing from the laminate for any reason, the elastic expansion changes accordingly. For example, if ten percent of the fiber is missing, the elastic expansion will show a 33% increase over nominal. Elastic expansion is often measured in a water jacket, but may also be measured using a direct method [37].

Each cylinder may also be subjected to a leakage test. Liners may have defects such as cracks, pinholes or other defects that could cause leakage. Damaged o-rings or sealing surfaces might also cause leakage.

Table 7 lists recommended batch testing. Burst and cycle tests are performed on representative units, while proof and leak tests are performed on all units.

Table 7 - Recommended Batch Testing

Test Name	Test Number	Basis
Burst	C.1	Confirm strength of materials and manufacturing process (sample unit)
Cycle	C.2	Confirm fatigue life (sample unit)
Hydrostatic Proof	C.3	Confirm manufacturing process and amount of material (all units)
Leak	C.4	Confirm no liner, boss or seal defects (all units)

3 RECOMMENDATIONS FOR FATIGUE TESTING

3.1 ASME Section VIII Division 3, Para KD-1260 Approach

Paragraph KD-1260, Experimental Determination of Allowable Number of Operating Cycles, of the ASME Boiler and Pressure Vessel Code was considered as to applicability of characterizing composite fatigue. KD-1260 uses experimental methods to determine the allowable number of operating cycles of components and vessels as an alternative to Article KD-3.

There are some limitations to using this approach for composites as compared with metals, according to requirements of KD-1260. This approach is only to be used for vessels or components that have been shown to demonstrate a leak before break mode of failure. While the fatigue failure of a metal liner would meet this criterion, the composite itself would generally fail in a rupture mode in cycling.

KD-1260 uses a test component or portion thereof is required to be constructed of the same material with the same processing and resulting equivalent mechanical properties. A representative composite pressure vessel would need to be tested, as opposed to a portion of one, in order to maintain proper similarity of structure and loading conditions.

KD-1260 works from a fatigue design curve from KD-3, and develops a line that is essentially offset from the design curve, requiring testing to a higher load level for the same number of cycles, a greater number of cycles at the same load level, or an interpolated combination of higher load and greater cycles. The factors used to develop the offset line are based on a number of factors related to loaded areas, temperature effects, surface roughness and statistical variation.

In order to use the KD-1260 approach on composite pressure vessels, corresponding factors would need to be developed for composites, based on parameters that are significant for them, some of which may be different than those factors considered for metal components and vessels.

One factor that needs to be considered is whether composite materials can be adequately characterized by this method. The fact that metals are isotropic and reasonably homogeneous offers advantages in terms of consistency of test specimens. Composites can vary in construction, with different orientation and placement of fibers in composite layers.

This might lead to significant differences in cyclic fatigue response, as reflected in Figure 2, comparing fatigue performance of composite specimens of different configurations and of composite pressure vessels. Testing of composite pressure vessels has also shown that subtle differences in the composite layup that do not affect burst pressure can have significant differences in cyclic fatigue performance because of differences in local stress states.

In this case, it may be safer to specify that testing must be conducted on specimens that are full scale, or perhaps allowing them to be shorter in length, so that the test results have a greater validity.

3.2 Composite Cyclic Fatigue

Carbon, aramid, and glass fiber reinforcements are subject to failure by cyclic fatigue. Carbon fiber is normally has the best cyclic fatigue life, followed by aramid fiber, and with glass fiber having a lesser cyclic fatigue life. Cyclic fatigue lives for the three fibers are shown in Figure 1 [7].

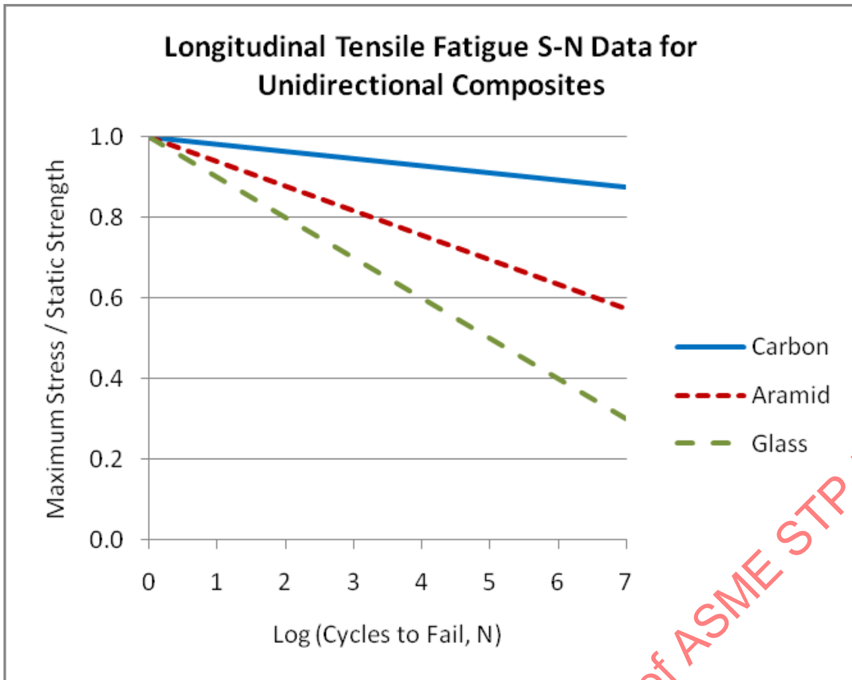

Figure 1 - Composite Cyclic Fatigue Lives

There are factors that may influence the fatigue life of a give fiber, such as resin modulus and elongation, transverse and shear stresses, and localized bending stresses. One may obtain different fatigue lives with cylinders having different levels of the above, even though the burst pressure of the cylinders is the same.

Therefore, it is necessary to test a given cylinder design to a level sufficient to demonstrate safety in service. Safety can be assured either by demonstrating a safe failure mode, such as leak-before-break (LBB), or by demonstrating a sufficient margin of safety over the required fatigue life of the cylinder.

Figure 2 provides a comparison of different sample construction and how that can affect fatigue life. The upper line, with data from Mandell [7] is a unidirectional carbon fiber reinforced specimen loaded in tension. The middle line, with data from Liber and Daniel [6], is a carbon fiber reinforced, flattened tube with a symmetric laminate having longitudinal fiber layers and +/- 45-degree layers loaded in tension. This is a more complex laminate, with more complex loading within the laminate, and a reduction if fatigue life compared with the unidirectional specimen.

The lower line, with data from carbon fiber reinforced pressure vessels that are pressure cycled. This is a more complex laminate, with more complex loading within the laminate, than the specimens used by Mandell and by Liber & Daniel. Correspondingly, the fatigue life is lower than the less complex specimens. The lower line is based on limited data, and is conservative, in part because much of the data is from tests where failure did not occur, and the number of cycles conducted was plotted.

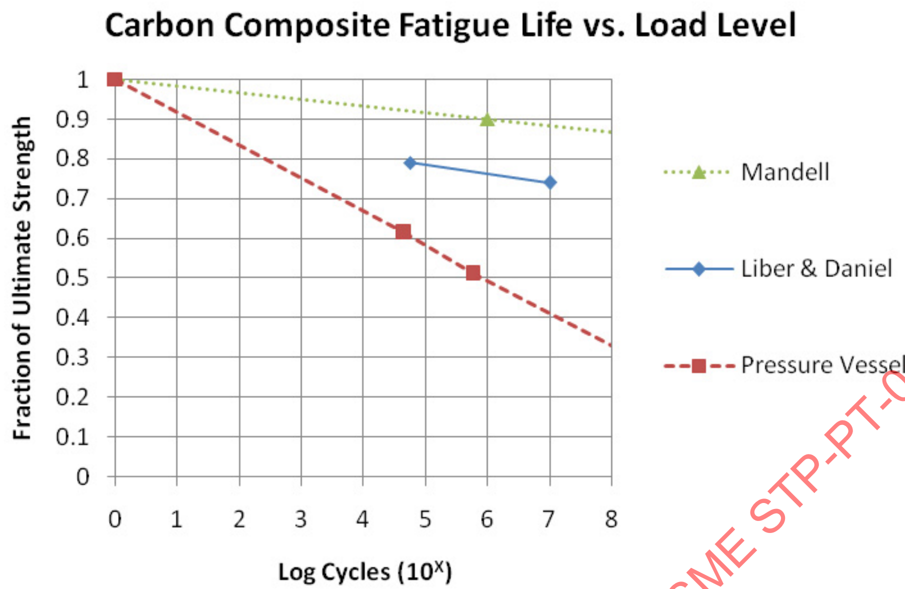

Figure 2 - Carbon Composite Fatigue Life vs. Load Level

The data presented in Figure 2 emphasize what was stated earlier in this section, that the cyclic fatigue performance can vary significantly depending on the laminate construction, loading, and other factors. Therefore, it is necessary to demonstrate through cyclic fatigue testing, that the cylinder has leak-before-break behavior or that there is a demonstrated margin between the cycles required in service and the number of cycles in the test.

The data presented in Figure 2 also indicate that cyclic fatigue testing can be accelerated by increasing the upper pressure limit. However, to make full use of this, there must be sufficient cyclic fatigue data to accurately represent a full range of life versus load level.

3.3 Liner Cyclic Fatigue

Metal liners are typically steel for type 2 tanks and aluminum for type 3 tanks. These metal liners are typically yielded in an autofrettage pressure cycle during the manufacturing process in order to reduce the mean stress during pressure cycling in service. Cycling at a lower mean stress will extend the fatigue life of the liner. Even so, a metal liner is typically cycled relatively close to its yield point, and the liner will have a limited fatigue life.

A plastic liner has a significantly lower modulus of elasticity than the composite reinforcement. The liner will, therefore, carry almost no load, and its strains in the hoop and longitudinal direction will be equal to that of the inside surface of the composite. However, as the internal pressure increases, this results in compressive stresses over most of the pressure range. As a result, crack growth in plastic liners will be significantly lower than in metal liners as long as the plastic material has sufficient elongation.

A hoop-wrapped, type 2 tank will typically fail by leakage between 15,000 and 60,000 cycles. A full-wrapped, type 3 tank will typically fail by leakage between 5000 and 20,000 cycles. The fatigue life will, of course, be affected by the materials of construction, surface finish, material thicknesses, the liner tensile strength, its stress-strain behavior, the pressure range and other factors.

One means of predicting the fatigue life of metal liners is to use fracture mechanics. One such program is NASA/FLAGRO [29]. Information on material properties, crack model, initial flaw size, and loading is entered. Crack growth rate, safe life, critical crack size, and numerical values of stress-intensity factor are calculated.

Liner fatigue life may also be calculated using empirical methods. Manson [30] developed the empirical equation:

$$\Delta\varepsilon = 3.5\ (\sigma u/E)\ Nf\text{-}0.12 + D0.6Nf\text{-}0.6$$

where: $\Delta\varepsilon$ = total strain range

σu = ultimate tensile strength, psi

D = ductility, ln (1/(1 – RA))

RA = reduction in area

Nf = number of cycles to failure

Liner strains as a function of pressure may be calculated using closed form methods, finite element methods, or other analytical methods. Knowing the ultimate tensile strength and reduction in area of the liner material, the fatigue life may be calculated. Several pressure loading cycles may be combined using Miner's rule, typically reduced by 20% to avoid having an unconservative life prediction.

3.4 Composite vs. Liner Fatigue Limits

One can compare the predicted fatigue life of a liner with that of the composite overwrap and assess if the liner is likely to leak before the composite fails, resulting in leak-before-break (LBB) behavior. Calculations are also necessary to show that at the time of liner failure, either the liner must not fail catastrophically, or the load carried by the liner, when transferred dynamically into the composite, will not cause rupture of the composite. Testing should be conducted to confirm LBB characteristics.

Since a plastic liner has a high fatigue life, it is necessary to conduct fatigue tests to confirm that either a leak will develop in the liner or that there is there is sufficient margin between service requirements and the tested cycles. Most composite cylinder standards set the level for testing to be three times the maximum expected service cycles. Some standards require two or three tests to be conducted to increase confidence levels in the test results.

4 STRESS RUPTURE TESTING

4.1 Stress Rupture Studies

Stress rupture is a phenomenon in which tensile failure will occur in the fiber under stained load with no other phenomenon being present. Related discussion of stress rupture is given in Section 2.2.2. Several studies have been conducted over time on glass, aramid and carbon fibers.

Investigators of stress rupture characteristics of glass fiber include Outwater [32] and Glaser, Moore and Chiao [1]. The data presented by Outwater were of relatively short duration. The data presented by Glaser, Moore and Chiao of Lawrence Livermore National Laboratory (LLNL) were gathered over a longer period of time on impregnated strands under constant load. This study was interrupted after about 10 years by an earthquake, and there was some evidence of ultraviolet (UV) light influence on the specimens later in the study. Robinson [5] evaluated the data from LLNL with results as shown in Figure 3.

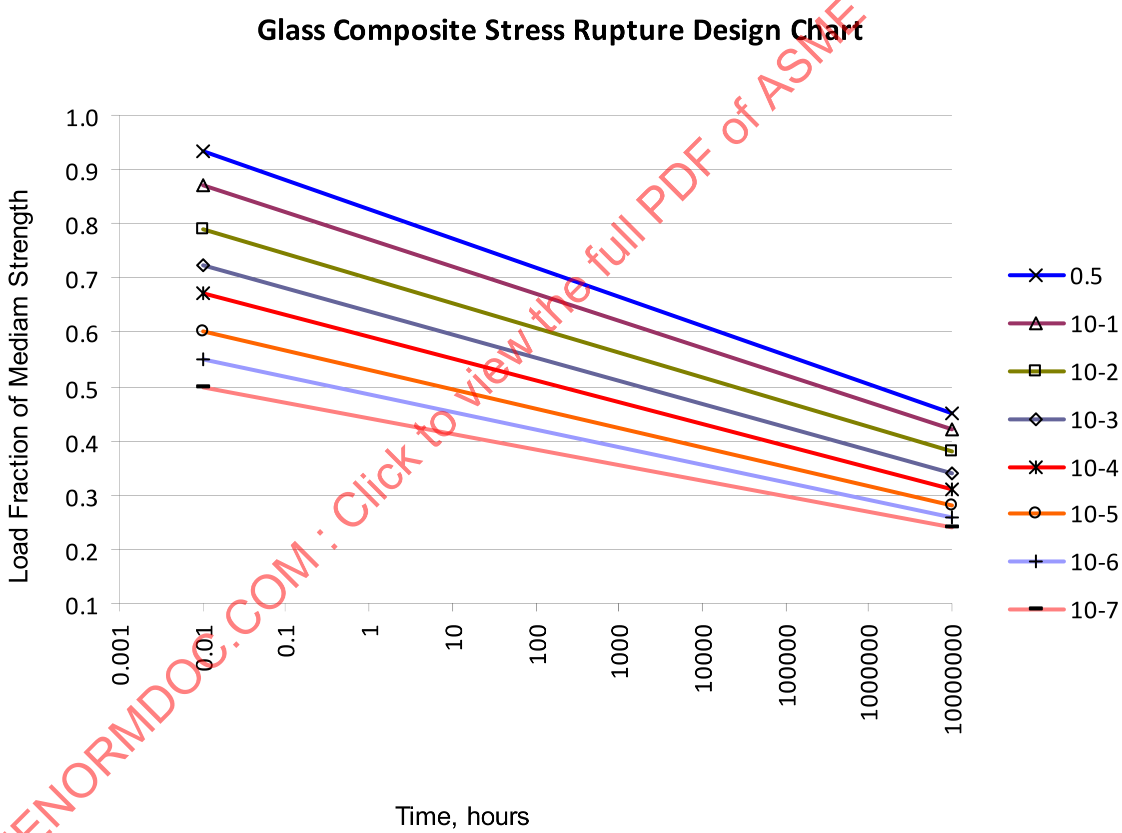

Figure 3 - Glass Composite Strand Stress Rupture Design Chart

Investigators of stress rupture characteristics of aramid fiber include Glaser, Moore and Chiao [2]. The data of Glaser, Moore and Chiao included some specimens that were influenced by UV light, and some that were kept in darkness. Both strands and pressure vessels were included in the testing program. Figure 4 shows that the stress rupture characteristics of aramid fiber are better than those of glass fiber.

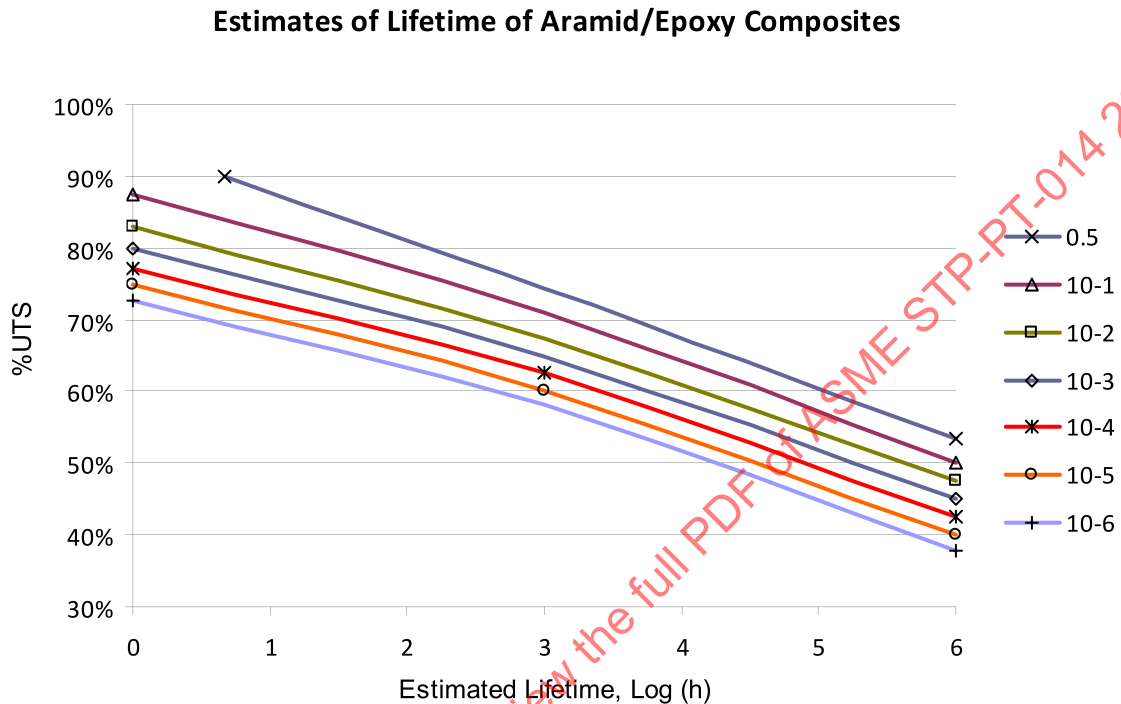

Figure 4 - Maximum Likelihood Estimates of Lifetimes of Aramid/Epoxy for Vessels, with Quantile Probabilities

Investigators of stress rupture characteristics of carbon fiber include Shaffer [31], Babel, Vickers and Thomas [3], and Chiao, Chiao and Sherry [4]. Robinson [5] evaluated the data from Shaffer with results as shown in Figure 5. It should be noted that the data from Shaffer is conservative to the extent that the tests were conducted at elevated temperature, which would accelerate the stress rupture phenomenon. Figure 5 shows that the stress rupture characteristics of carbon fiber are superior to both those of glass and aramid fiber.

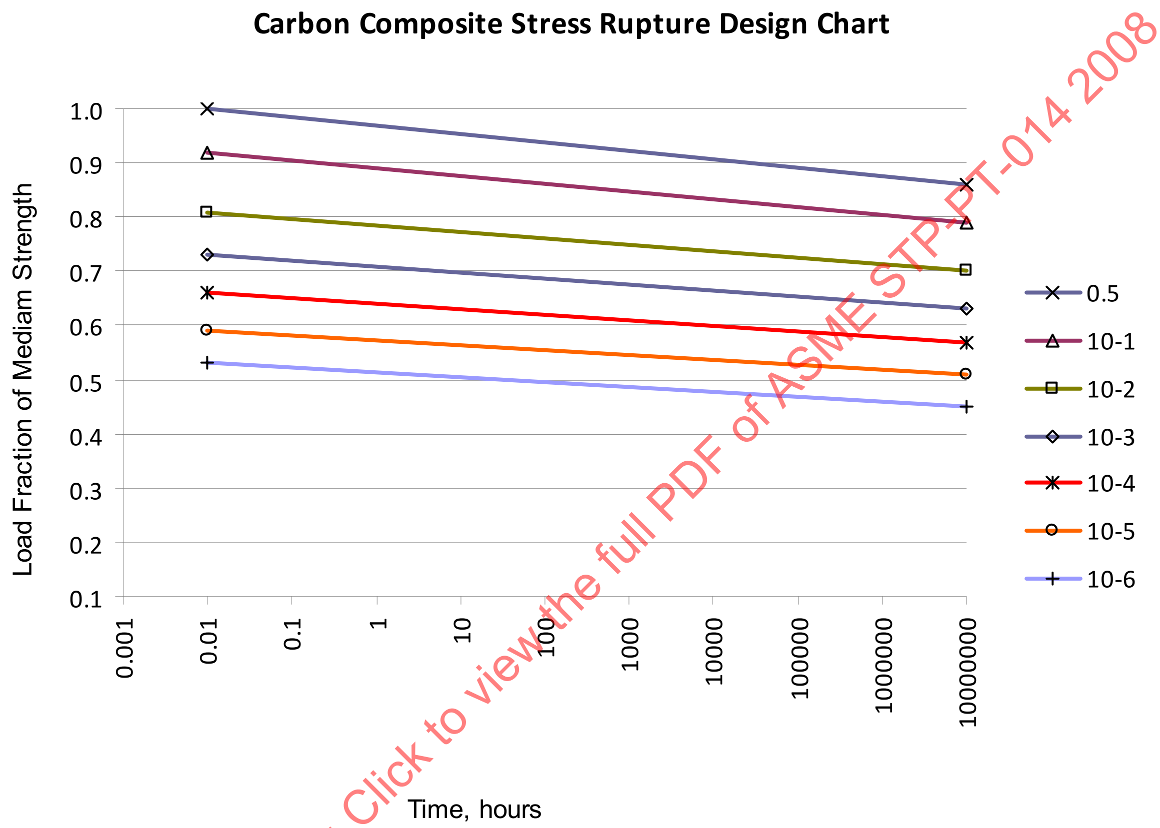

Figure 5 - Carbon Composite Strand Stress Rupture Design Chart

4.2 Field Testing and Experience

Field testing and experience have provided validation for the stress rupture studies and predictions provided in Section 4.1. There have been a very limited number of stress rupture failures in the field. The failures that have occurred have generally been as a result of damage to the pressure vessel or overfilling, i.e., excess pressure.

As mentioned in Section 1.1.4, there were failures of glass fiber reinforced vessels used to inflate life rafts, but quality issues reflected in lot sample burst performance also played a part. The glass vessels that ruptured in a military aircraft application were left in service beyond the design lifetime, and contained pressure in excess of the design specification. In this case, the number of failures compared with the population served to support the glass stress rupture curves.

There was also a study conducted at Lewis Research Center involving glass fiber reinforced cylinders with load-sharing aluminum liners that were in an outdoor environment, with full exposure to temperature extremes, –26˚C to 43˚C (–15˚F to 110˚F), and ultraviolet light, and periodic pressure cycles, which degraded the vessel strength [33]. One tank failed after 8.5 years.

No aramid fiber reinforced pressure vessels are known to have failed in service due to stress rupture.

A set of 10 aramid reinforced spherical pressure vessels were provided to NASA/Johnson Space Center for extended fatigue testing [34]. These vessels were being tested as "fleet leaders" in regards to similar vessels being used aboard the Space Shuttles. These vessels had been held at 50% of their average ultimate strength under elevated temperature conditions in Houston. One of the vessels held at elevated temperature failed in 1995, after about 11 years in test. Using an Arrhenius rate equation, with a doubling of activity for each 10˚C (18˚F) temperature rise above ambient, this correlates to an 700-year life at ambient temperature with a stress ratio of 2.0. This result is consistent with predictions from stress rupture models.

No carbon fiber reinforced pressure vessels are known to have failed in service due to stress rupture.

4.3 Methods for Accelerating Tests and Extrapolating Data

The stress rupture studies discussed in Section 4.1 used accelerated testing and extrapolation of data to address the expected service life of composite pressure vessels, which is typically from 15 to 20 years, but can be in the 30 to 50 year time frame. These studies use testing at higher service pressures to accelerate testing.

These studies evaluate the data using a Weibull distribution. Tests have been conducted at pressure levels from about 50% up to about 97% of the average ultimate strength. While it is possible to get failure of glass or aramid fibers at the lower end of this test range, the lowest load level for which carbon fiber stress rupture data has been generated is 80%. This is due to the superior stress rupture properties of carbon fiber, as tests at lower levels would require testing for times greater than the service life of the cylinders.

The use of higher pressure to accelerate stress rupture testing may be overly conservative in terms of the shape factor alpha (α) of the Weibull distribution [35]. Testing has indicated that the alpha factor for glass and aramid fiber increases as the load level decreases. With no data at a lower load level, the alpha used in the Weibull analysis of carbon fiber stress rupture has been maintained so as to yield conservative results.

Elevated temperature has also been used to accelerate testing. The Arrhenius rate equation relates molecular activity increase to temperature increase. Stress rupture was confirmed to be subject to subject to the Arrhenius rate equation on aramid fiber by C.C. Chiao of Lawrence Livermore National Laboratory (LLNL), as is expected to apply similarly to glass and carbon fiber. Elevated temperature was used to accelerate testing in the National Aeronautics and Space Administration (NASA) program [34] mentioned above.

The use of elevated temperature to accelerate testing must be done with some caution. If the strength of the fiber being tested is significantly affected by temperature, there must be additional efforts to correlate the accelerated testing to ambient results. This is also true if the elevated temperature causes thermal stresses in the composite.

Another method for adding assurance of safe operation is "fleet leader" testing such as conducted by NASA in support of the Space Shuttle program. Testing cylinders at a pressure and temperature sufficiently above what is used in service will, depending on the number of cylinders being tested compared with the cylinders in service, give some assurance that a stress rupture problem will show up in the test program prior to showing up in service.

It should also be noted that pressure normally fluctuates in service due to temperature changes, and due to use and replenishment of the contents. Cylinders are designed to withstand some pressures above the nominal service pressure. If the proper average ambient temperature is used for design, the effects of pressure fluctuations above and below that temperature should reasonably cancel out.

If the fleet leaders are kept at the maximum expected pressure, the testing would yield conservative results. It may also be suitable to test near the maximum expected ambient temperature conditions, and not be concerned about very short duration pressure increases due to heat of compression during filling.

It is also reasonable to have sufficient cylinders in a fleet leader test program to allow a cylinder to be removed periodically and burst tested to monitor any strength changes. However, this will require sufficient samples and appropriate statistical evaluation to be able to address possible loss in strength compared with expected variability in burst strength at time of manufacture.

It may also be possible to use nondestructive evaluation (NDE) methods on a fleet leader program to assess remaining strength, particularly if this could be correlated with burst strength of cylinders periodically removed from the test. Possible NDE methods could include acoustic emission, acousto-ultrasonics, and strain measurement.

5 SUMMARY AND RECOMMENDATIONS

Composite cylinders have been used for over 50 years in commercial, vehicle, defense and aerospace applications. New materials, processes, design approaches and applications have been incorporated during that time. The industry has maintained a high level of safety. The industry has adapted to these changes and has developed new and revised standards to address these changes and to reflect a better understanding of service conditions.

It is recommended that the industry:

- Continue to monitor field use and incorporate changes to requirements, standards and codes that reflect knowledge gained for composite pressure vessels,
- Use a failure modes and effects analysis (FMEA) approach to standards, using the knowledge gained from field experience,
- Develop standards for composite pressure vessels that are more performance based to improve both safety and performance,
- Address requirements using performance testing, not by using excessive safety factors,
- Use stress ratios for the various reinforcing fibers that accurately reflect their stress rupture and fatigue characteristics to achieve high reliability
- Harmonize testing requirements where practical,
- Use qualification tests that are appropriate for the application and for the materials and design features of the pressure vessels being used, and
- Consider using fleet leader programs for new materials, designs, or applications if there is likely to be a significant safety issue

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ANNEX A MATERIAL TEST PROCEDURES

A.1 Fiber Strength

Each batch of filament materials is subjected to an impregnated strand test in accordance with ASTM D 2343-95 for glass and aramid fibers and SACMA SRM 16R-94 or ASTM D 4018-99 for carbon fiber. Equivalent standards may be used. The strength of fibers shall meet specified requirements.

A.2 Fiber Modulus of Elasticity

Each batch of filament materials is subjected to an impregnated strand test in accordance with ASTM D 2343-95 for glass and aramid fibers and SACMA SRM 16R-94 or ASTM D 4018-99 for carbon fiber. Equivalent standards may be used. The modulus of fibers shall meet specified requirements.

A.3 Resin Shear Strength

Resin system materials shall be tested on a sample test panel or cylinder section, representative of the composite overwrap, in accordance with ASTM D2344, Standard Test Method for Apparent Interlaminar Shear Strength of Parallel Fiber Composites by Short-Beam Method. Following a 24-hour water boil, the composite shall have a minimum shear strength of 13.8 MPa (2000 psi).

A.4 Resin Glass Transition Temperature

The resin glass transition temperature shall be determined in accordance with ASTM D6604-00 Standard Practice for Glass Transition Temperatures of Hydrocarbon Resins by Differential Scanning Calorimetry, ASTM D3418-03 Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry, or equivalent. It is recommended that the glass transition temperature be at least 20˚C (36˚F) above the maximum service temperature.

A.5 Meta Tensile Strength

Tensile strength shall be determined using test methods of ASTM E8, Standard Test Methods for Tension Testing of Metallic Materials (Metric), and shall meet the specified requirements. Alternatively, tensile tests may be carried out in accordance with equivalent standards.

A.6 Metal Elongation

Elongation shall be determined using test methods of ASTM E8, Standard Test Methods for Tension Testing of Metallic Materials (Metric), and shall meet the specified requirements. Alternatively, elongation tests may be carried out in accordance with equivalent standards.

A.7 Nonmetallic Liner Elongation

The tensile yield strength and ultimate elongation shall be determined in accordance with ASTM D638, Standard Test Method for Tensile Properties of Plastics. Tensile or impact testing shall be conducted on samples of the nonmetallic liner material to demonstrate that the material fails in a ductile, rather than brittle, mode at temperatures down to and including minimum service temperature.

A.8 Nonmetallic Liner Softening Point

The softening temperature is recommended to be at least 10˚C (18˚F) above the maximum service temperature, and the melting temperature is recommended to be at least 20˚C (36˚F) above the maximum service temperature, when tested in accordance with the method described in ISO 306, Determination of Vicat Softening Temperature, or using an equivalent alternative method.

A.9 Nonmetallic Liner Material Compatibility

Compatibility of the liner material with environmental fluids should be determined in general accordance with ASTM D1693-07a, Standard Test Method for Environmental Stress-Cracking of Ethylene Plastics, or equivalent. Compatibility with contained gases should be determined by gas cycling tests, see Section B.5.

A.10 Charpy Impact Test

The impact properties of the steel in the finished container or liner shall be determined in general accordance with ISO 148, Charpy Impact Test (V-Notch) or ASTM E23, Standard Test Methods for Notched Bar Impact Testing of Metallic Materials. The impact test pieces are taken from the wall of the container in the transverse direction. The notch plane orientation is in the direction perpendicular to the circumference and along the length. Test pieces with a width of less than 5 mm (0.2 in.) are taken from the longitudinal direction. If the wall thickness does not permit a final test piece width of 10 mm (0.4 in.), the width shall be as near as practicable to the nominal thickness of the container wall. All impact tests shall be conducted at -40˚C (-40˚F). Impact values shall not be less than that indicated as follows:

Width of Test Piece, mm (in.)	5.0–7.5 (0–0.3)	7.5–10.0 (0.3–0.4)
Impact Strength, J/cm² (ft-lb/in.²)	44 (210)	50 (240)

Impact values for test pieces of width less than 5 mm (0.2 in.) shall be based on special studies of particular materials and particular specimens.

- Required average results of three specimens.
- Not more than one specimen shall break at less than the average value required and no single specimen shall break at less than 80% of the average value.

A.11 Sulfide Stress Cracking Resistance for Steels

If the upper limit of the ultimate tensile strength exceeds 950 MPa (138,000 psi), the steel is tested in accordance with the procedures described in Method A—National Association of Corrosion Engineers (NACE) Standard Tensile Test of NACE Standard TM0177-96, except as noted in this section. Tests are conducted on tensile specimens with a gauge diameter of 3.81 mm (0.150 in.) machined from the wall of a finished container or liner. The specimens shall be placed under a constant tensile load equal to 60% of the specified minimum yield strength of the steel, immersed in a solution of distilled water buffered with 0.5% (wt/wt) sodium acetate trihydrate and adjusted to an initial pH of 4.0 using acetic acid. The solution is continuously saturated at room temperature and pressure with 0.414 kPa (0.06 psia) hydrogen sulfide (balance nitrogen). Three specimens are tested and none may fail within the 144-hour duration of the test. Specimens that fail outside the gauge length are considered invalid tests.

A.12 Corrosion Tests for Aluminum

Tests for susceptibility of aluminum alloys to intercrystalline corrosion and stress corrosion are carried out in accordance with ANNEX A of ISO 7866, Refillable Seamless Aluminum Alloy Gas Cylinders—Design, Construction and Testing, and must meet the requirements therein.

A.13 Sustained Load Cracking (SLC) for Aluminum

The resistance to SLC is carried out in accordance with ANNEX B of ISO 7866, Refillable Seamless Aluminum Alloy Gas Cylinders—Design, Construction and Testing, and must meet the requirements therein.

A.14 Ultraviolet Resistance of External Coatings

Protective coatings required to meet environmental tests are evaluated for resistance to ultraviolet effects with a minimum 1000 hours exposure using a UVA 340 lamp in accordance with ASTM G154, Standard Practice for Operating Fluorescent Light Apparatus for UV Exposure of Nonmetallic Materials. There should be no evidence of blistering, cracking, chalking or softening.

ANNEX B CYLINDER QUALIFICATION TEST PROCEDURES

B.1 Burst

For: All applications and sizes.

Test: Pressurize hydraulically to minimum required burst pressure. Hold for sufficient time to verify stable pressure (e.g., 5 to 10 seconds). Pressurize to burst.

Criteria: Cylinder shall not burst at or below the minimum required burst pressure to meet stress ratio requirements.

B.2 Ambient Cycling

For: All applications and sizes.

Test: Pressurize hydraulically from 10% of maximum service pressure to maximum service pressure at a rate not to exceed ten cycles per minute for a number of cycles three times the required number of lifetime cycles for the application.

Criteria: Cylinder shall not leak prior to reaching the required number of cycles for the application, and shall not rupture before reaching three times the required number of cycles for the application.

Note: If the cylinder leaks before reaching three times the required number of cycles for the application, then a total of three cylinders must be tested, and all must meet the test criteria.

B.3 Leak Before Break

For: All applications and sizes. Not required if test B2 is passed without leakage.

Test: Pressurize hydraulically from ten percent of maximum service pressure to test pressure (1.5 times nominal service pressure) at a rate not to exceed 10 cycles per minute for a number of cycles three times the required number of cycles for that application.

Criteria: Cylinder shall not rupture prior to leaking or reaching three times the required number of cycles, whichever comes first.

B.4 Accelerated Stress Rupture

For: All applications and sizes.

Test: Pressurize to maximum operating pressure for 1000 hours at 65˚C (149˚F), then burst at ambient temperature.

Criteria: Cylinder shall not leak or rupture during pressure hold. Burst must exceed 85% of minimum required burst pressure.

B.5 Gas Cycling

For: All applications and sizes. Applies to cylinders with nonmetallic liners (which are subject to permeation) and cylinders in which there is a potential for static electricity to build up in the flow stream.

Test: Pressurize cylinder to nominal service pressure with gas to be contained, and hold for 72 hours. Cycle with gas between 10% of service pressure and nominal service pressure for 500 cycles, completing each cycle over a period of 60 minutes. Vent cylinder. Cycle in accordance with B.2 Ambient cycling. Examine interior of liner.

Criteria: Cylinder shall not leak or rupture. The liner shall show no evidence of blisters or damage from electrostatic discharge.