

HYDROGEN STORAGE – INDUSTRIAL PROSPECTIVES

Barthélémy, H.

Air Liquide, 75 Quai d'Orsay, Paris, 75007, France, herve.barthelemy@airliquide.com

ABSTRACT

The topic of this paper is to give an historical and technical overview of hydrogen storage vessels and to detail the specific issues and constraints of hydrogen energy uses. Hydrogen, as an industrial gas, is stored either as a compressed or as a refrigerated liquefied gas. Since the beginning of the last century, hydrogen is stored in seamless steel cylinders. At the end of the 60s, tubes also made of seamless steels were used; specific attention was paid to hydrogen embrittlement in the 70s. Aluminum cylinders were also used for hydrogen storage since the end of the 60s, but their cost was higher compared to steel cylinders and smaller water capacity. To further increase the service pressure of hydrogen tanks or to slightly decrease the weight, metallic cylinders can be hoop-wrapped. Then, with specific developments for space or military applications, fully-wrapped tanks started to be developed in the 80s. Because of their low weight, they started to be used in for portable applications: for vehicles (on-board storages of natural gas), for leisure applications (paint-ball) etc... These fully-wrapped composite tanks, named types III and IV are now developed for hydrogen energy storage; the requested pressure is very high (from 700 to 850 bar) leads to specific issues which are discussed. Each technology is described in term of materials, manufacturing technologies and approval tests. The specific issues due to very high pressure are depicted.

Hydrogen can also be stored in liquid form (refrigerated liquefied gases). The first cryogenic vessels were used in the 60s. In the following, the main characteristics of this type of storage will be indicated.

1.0 COMPRESSED HYDROGEN STORAGE

1.1 Introduction

The following 4 types of high pressure vessels are classified (see Fig. 1):

- Type I: pressure vessel made of metal
- Type II: pressure vessel made of a thick metallic liner hoop wrapped with a fiber-resin composite.
- Type III: pressure vessel made of a metallic liner fully-wrapped with a fiber-resin composite.
- Type IV: pressure vessel made of polymeric liner fully-wrapped with a fiber-resin composite. The port is metallic and integrated in the structure (boss).

The pressure vessels are generally cylinders, but composite vessels can also be polymorph or toroid (see Fig. 2).

Hydrogen can be stored in the four types of pressure vessels. The choice of the storage is based on the final application which requires a compromise between technical performances and cost-competitiveness. H₂ as industrial gas is stored in type I tanks, the pressure of which is from 150 to 300 bar (usually 200 bar). These are the most spread high pressure vessels today and are the cheapest. When only higher pressures are required – mainly for stationary applications – the type II tanks are preferred. Type III and type IV vessels are intended for portable applications, for which weight savings is essential. However these vessels are much more expensive. Technical performances will be presented in the last paragraph of this paper.

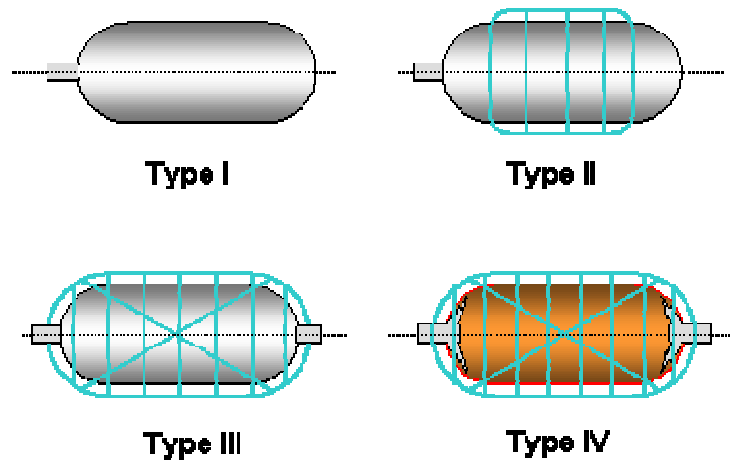


Figure 1. Schematic representation of the 4 pressure vessel types

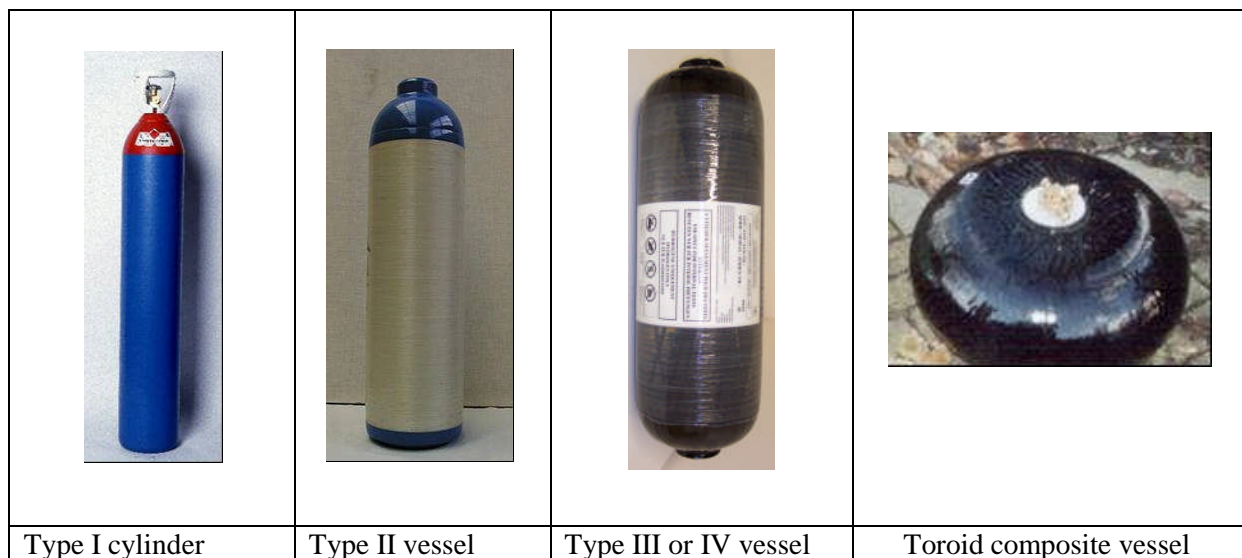


Figure 2. Pressure vessels pictures

1.2 Some history

The oldest pressure vessel type is the type I, when made of steel. It was introduced in the 19th century between 1870 and 1880. It was lead to the development of a new industry at that time: the industrial gas business. More particularly, it seems to be first linked to the high consumption of carbon dioxide for beverage, with the necessity to store it safely, in the liquid state and in high quantities. The probable date of such storage for carbon dioxide seems to be 1874. Note that low pressure vessels (< 20 bar) were already used in the mid 19th century: they were made of copper, generally low capacity and used for the very first breathable apparatus; but they cannot be qualified as high pressure vessels. Regarding the hydrogen, it was mainly used during the last 19s century for military observation balloons in Asia and in Africa (around 1880). It was stored in 120 bar pressure vessels in wrought iron vessels in 1880; of course, these cylinders were very heavy (500 kg of steel were needed to store 25 Nm³ of hydrogen), but they seem to be the first to be able to transport safely hydrogen at high pressure. Vessels made with seamless steel were introduced in 1885, manufactured by drawing and forming of plates (Lane & Taunton British patent) with a special development for making the neck. Meanwhile, the manufacturing of high pressure cylinder from seamless tubes was developed (Mannesmann German patent). High pressure cylinders manufactured from billets were also perfected

in the late 1880s. Until the 60s, the service pressure of these storages was 150 bar. From 1960, the service pressure was increased up to 200 bar. Today both 200 and 300 bar cylinders are co-existing for industrial gases.

High pressure composite vessels were introduced more than 70 years after and were especially developed for space and military applications, for which technical performances – especially weight – was a very important criteria. The experimentation of composite vessels started in the 50s but the first high pressure vessels used for space and military applications started actually in the 60s in the US (rocket motors and other pressure vessels for space shuttles, sonar equipment etc...). The market of these new high pressure vessels was very low in the 60s and the production was not regular at that time: manufactured batches could count from about ten to a few hundreds vessels (no series production). They were made of a metallic or polymeric liner wrapped with glass fiber composite. The civil market started to be penetrated in the first 70s and was linked to the will to increase the market of these high tech products. When compared to the type I conventional vessels for industrial gases, the cost of these composite vessels and the lack of regulation for composite vessels slowed down this penetration. For example, in the late 70s, 100 000 cycles were requested by the ASME code for pressure vessels – what composites tanks could not fulfill. As a consequence, for each model and each application, the composite vessels had to obtain a special authorization and their lifetime was limited. However, the first important civil market in the 70s was the breathable apparatus for firemen. From 80s on, these composite vessels started to be used for skin diving, fuel storage (mainly natural gas) and leisure applications (like paint-ball). The storage pressures were conventional: from 100 to 300 bar.

Many improvements were also performed since the first developments: for example, weight has decreased, cycling performance has increased by using thin liners with adequate mechanical properties, and other fibers than glass (kevlar, carbon). Moreover the regulation was set up for both industrial gases and fuel gas storages.

1.3 Design and manufacturing

For all pressure vessels, the design shall take into account the service and test pressures, the external stresses which are specific to the use (like impacts, aggressive media, vibrations, temperature of service, weight of connectors etc...), the real lifetime (cycling) and the safety coefficients defined for both static and dynamic conditions. The failure modes like plastic deformation, buckling, creeping, fatigue etc... for metals, delaminations, fiber ruptures, cracks, ageing etc... for composites are also taken into account for the design. All these parameters define the mechanical design and the choice of the materials. The materials shall also be compatible with the gas when in contact. It is important to note that metallic vessels and composite vessels are very different:

- The metal is isotropic, the composite is anisotropic: the mechanical properties are concentrated in the fiber direction for the composite.
- The failure modes are different.
- The ageing is different.

For example, Fig. 3 gives the main strains which are generally considered for metallic pressure vessels. In general, the domes are over designed. That is why the type II vessels, with their hoop reinforcement only on the cylindrical part of thick liners, can easily withstand higher pressures.

For the composite wrapping, an analytical and simplified calculation is generally performed (see Fig. 4) to have a first estimation of the lay-up design. Then a complete study with a finite elements software is necessary for a correct and optimized design (which should be coherent with the filament winding machine code).

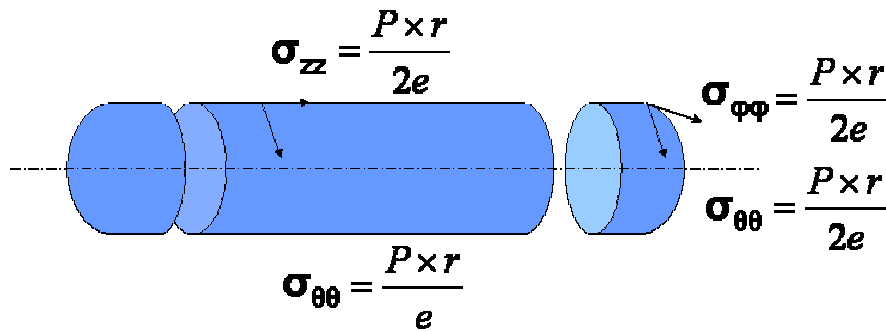


Figure 3. Main strains considered for the metallic pressure vessels design (type I and metallic liner)

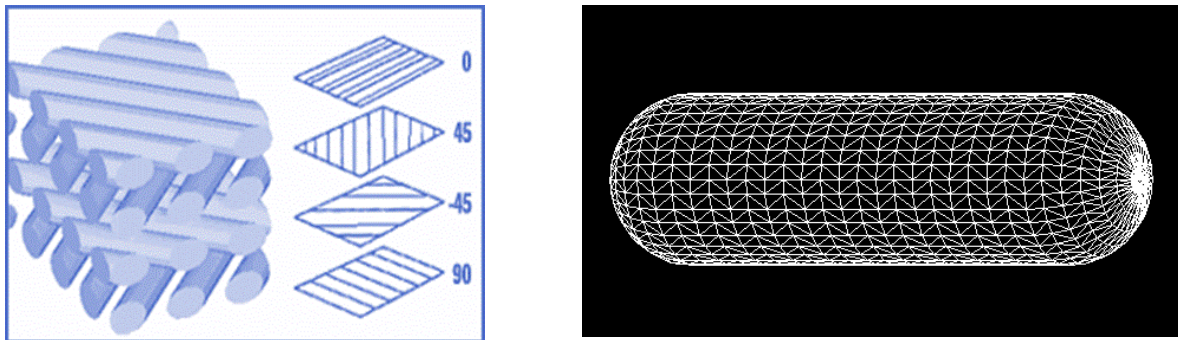


Figure 4. Multi-layered element & vessel meshes example

Type I vessels can be manufactured from 3 different processes (see Fig. 5 for the principles):

- From plates: the process consists in deep-drawing metallic plated to form the shape (this step can be performed many times to have the desired diameter and thickness); the neck is formed by hot-spinning and the port is machined in the excess of metal coming from the spinning step. A one port cylinder is thus obtained. The heat treatments are then applied to have the desired mechanical properties.
- From billets: the billet is firstly heated to allow the drawing to be performed. The process is then similar to the previous one.
- From tubes: Tubes are purchased and in general the original thickness is kept for the hoop. The domes are formed by hot spinning and a 1 or a 2 ports cylinder can be obtained. The process is then similar to the first one.

For each technology, quality controls of the materials used and of each step of manufacturing is performed and traced. The liners of type II and type III vessels can be manufactured in the same ways.

The polymeric liners can be made by:

- From the polymer or the monomers by the rotomolding process: the polymer (or the monomers) is introduced in a mold the shape of which is the final liner shape. The liner is made by heating and then cooling the mold while rotating (the fusion temperature or the polymerisation temperature have to be reached). It can be one or two port liner. The metallic bosses are introduced during the rotomolding step or stick on the liner before wrapping.

- From tubes: polymeric tubes (made by extrusion blow molding) and domes (equipped with the metallic boss) are purchased at the desired diameter. Both shapes are welded to form the liner.

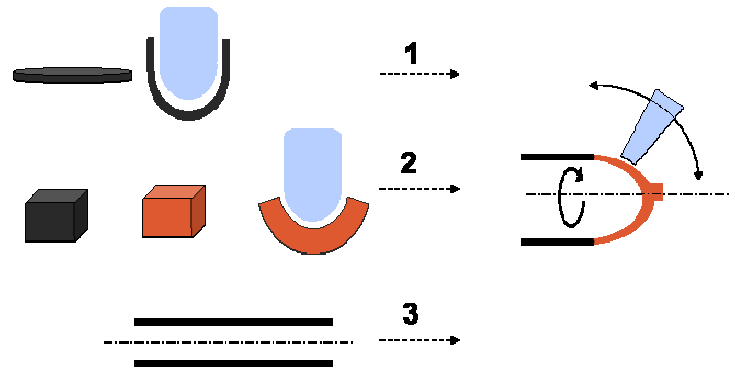


Figure 5. Principle of metallic tank manufacturing processes (1: from plates – 2: from billets – 3: from tubes)

For all composite vessels, the metallic or the polymeric liner is then hoop-wrapped or fully wrapped with the composite with a filament winding machine. For cylinder vessels, 3 wrappings are possible: hoop, polar and helical (see Fig. 6). Types II are only hoop-wrapped. Type III and IV vessels are generally a combination of hoop and polar wrapping – but a combination of the 3 wrapping can be considered. Many vessels can be wrapped in the same winding machine if it is equipped for many winding heads. Once the liner is wrapped, the resin must be cured. The curing is generally performed in ovens with the resin appropriate heat treatment.

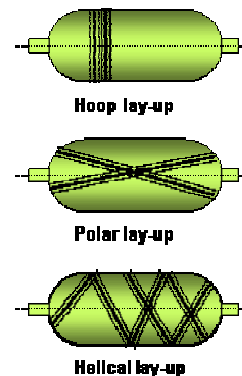
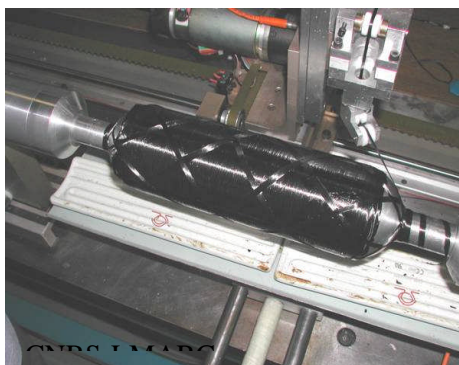


Figure 6. Winding machine and the 3 winding possibilities

1.4 Materials suitable for hydrogen high pressure vessels

From a material compatibility point of view, the main issues for the hydrogen high pressure vessels are:

- The risk of hydrogen embrittlement of the steel: this phenomena leads to a premature crack of the steel due to H atom dissolution and trap (stress corrosion cracking). The main risk is the burst of the tank. A lot of efforts on H₂ gas pressure embrittlement understanding and prevention rules were conducted in the 70s and 80 s after many accidents occurred with steel pressure vessels. The prevention rules based on 200 bar cylinders were then defined.

For more information on the risk of hydrogen embrittlement of steels, see [1], [2], [3], [4], [5], [7], [8], [9], [10] and [11].

- The permeation rated through the polymeric liner. The permeation is specific of type IV vessels and is an inherent phenomenon for all gases in contact with polymers. It is the results of the H₂ gas dissolution and diffusion in the polymer matrix. It had been identified at the beginning of composite vessels development and low permeable polymers are one topic of research.

Because H₂ is a small molecule, the diffusion and thus the permeation are enhanced. For safety reason, the permeation shall be lower to a certain rate. This leads to the development of special polymers which are suitable for hydrogen liners. In 2006, polyethylene and polyamide (specific semi-crystalline grades) are the most used liners for hydrogen energy type IV tanks. The other phenomenon to avoid is the blistering or liner collapse resulting from hydrogen being trapped between the liner and the composite.

No specific issue brought by hydrogen gas is met with aluminium alloys (except if presence of mercury). The aluminium alloy grades used for the high pressure vessels are 6061 and 7000. They are not specific to H₂ and are used for the types I, II and III vessels and also for the metallic boss of type IV tanks. Only generic issue for AA 6061 liners is the influence of tap water which reduces significantly the pressure cycling life [6].

Either glass, aramide or carbon fibers can be used for composite tanks wrapping. These fibers are characterized by their tensile modulus, tensile strength and elongation. Table 1 gives the usual ranges of these mechanical properties for each category of fiber. Hydrogen is stored in very high pressure vessels when used for hydrogen energy applications (service pressure ≥ 350 bar). As a consequence, from a mechanical point of view, carbon fiber is preferred. In the same way, various resins can be used (polyester, epoxy, phenolic etc...). But, for pressure vessels, due to its good mechanical properties and good stability, the most used is the epoxy resin. Pre-impregnated fibers are commercially available, but expensive. Thus, mainly for cost reasons, the fiber is impregnated just before the winding step.

Table 1. Range of fiber mechanical properties

Fiber category	tensile modulus (GPa)	tensile strength (MPa)	Elongation (%)
Glass	~ 70 - 90	~ 3300 - 4800	~ 5
Aramid	~ 40 - 200	~3500	~ 1 - 9
Carbon	~ 230 - 600	~ 3500 - 6500	~ 0,7 - 2,2

To conclude, when compared to the storage of other gases, hydrogen requires special attention for the choice of the steel for types I, II and III tanks & for the polymer choice for type IV tanks. A material test is generally requested to prove that the embrittlement is low: tensile test, disc test, fracture mechanism tests. A full permeation measurement is required on one vessel to prove that the permeation is below a specified rate (1 cm³/l/h e.g.). A measurement on a sample alone could also be sufficient to assess the permeation rate.

Compared to industrial gas, hydrogen energy has brought new constraints for pressure vessels and this, mainly for the transportation field. Before entering into details for pressure vessels dedicated to hydrogen energy, it is important to remind the main hydrogen energy applications. They are listed below and are depending on the fuel cell development (PEMFC mainly):

- Fuel for transportation: buses, car, scooters, other leisure vehicles. These vehicles can be powered by a fuel cell or by an internal combustion engine fuelled with H₂. Use in boats is also considered. For this application, the main constraint at the moment is the weight and volume savings. As a consequence for the pressure storage, types III and IV could be considered but the cost of the storage system is also important. This is why type I or II is used.
- Stationary applications: back-up power supply or power generator for residential. For this application, the cost of hydrogen supplied is the main parameter. The pressure cycle life is also an issue.
- Portable applications: portable back-up power supply, portable power generators, electronics (computers, mobile phones, etc...).

Weight and volume savings are primordial. The performances which are generally used to compare the pressure vessels are Cm and Cv defined as:

- Cm: weight performance: mass of H₂ stored divided by the mass of the vessel (%wt)
- Cv: volume performance: mass of H₂ stored divided by the external volume of the vessel (g/l)
- cost

The safety requirements are of course the same for all of them. Fig. 7 shows the technical performances as a function of service pressure for the today technology of type III and type IV vessels. The performances are given with 10% of incertitude. Note that the Cm for type I and type II vessels are respectively 1 and 1,5 % at 200 bar (the weigh is ~60-70kg to store 10 Nm³ of hydrogen).

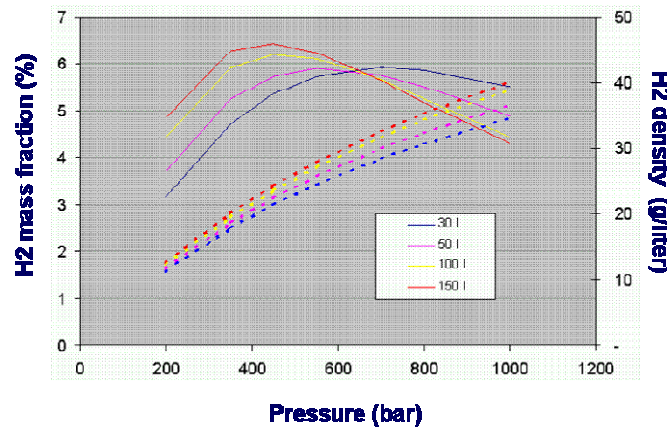


Figure 7. Cm and Cv as a function of the pressure (types III and IV)

1.5 Compressed gas storage – Conclusion

The main features (state-of-the-art) of the 4 pressure vessels are summarized in Table 2.

Each pressure vessel type can be used in the hydrogen energy supply chain. This depends mainly on the application type of the fuel cell. The main developments today concern type III and type IV at very high pressure. Even with the today design, some improvement has to be performed for the 700 bar pressure in order to fulfill the hydrogen energy standards.

Table 2. Main features for H₂ pressure vessel types in 2006

Type I	Type II	Type III	Type IV
Technology mature: ++ Pressure limited to 300 bar (⇒ density: -) Cost performance: ++ Weight performance: -	Technology mature: + Pressure not limited (⇒ density: +) Cost performance: + Weight performance: 0	Technology mature for P ≤ 350 bar; 700 bar under development. Cost performance: - Weight performance: +	Technology mature for P ≤ 350 bar; 700 bar under development. Cost performance: - Weight performance: +

2.0 CRYOGENIC VESSELS FOR THE STORAGE OF LIQUID HYDROGEN

2.1 Introduction (comparison of efficiency/grows storage)

Cryogenic vessels have been commonly used for more than 40 years for the storage and transportation of industrial and medical gases. The advantage of storing gases in such form is obvious: in a volume of 1 litre of liquid, about 800 litres of gas can be stored. This represents a clear advantage compared to the transportation of such gases in compressed form, which is done today at pressures of 200-300 bar (less gas per volume unit) and require thick walls (and heavy vessels) to resist the high pressure.

The disadvantage is, of course, that the gases need to be refrigerated down to very low temperatures to be in liquid form, especially for liquid hydrogen. The temperature gas/liquid equilibrium for different gases under a pressure of one atmosphere are given in Table 3. For gases being stored at such low temperatures, it is necessary to use high efficiency (vacuum) insulated vessels.

Table 3. Boiling temperatures at atmospheric pressure of different gases

Gases	Kr	O ₂	Ar	Air	N ₂	Ne	H ₂	He
Boiling temperature	- 153	- 183	- 186	- 191	- 196	- 246	- 253	- 269

2.2 Different types of cryogenic vessels

Cryogenic vessels can be differentiated either by the type of insulation used or the type of applications.

For example, cryogenic vessels used for gases requiring low temperature for liquefaction are normally vacuum insulated and composed of an inner pressure vessel and an external protective jacket (see Fig. 8). To reduce the thermal conductivity of the space between the inner vessel and the outer jacket, perlite (powder structure) or super insulation (wrapping with layers of aluminium film) are used. For gases such as carbon dioxide or nitrous dioxide, due to the relatively high liquefaction temperature, non-vacuum insulated vessels are used. The insulation of the vessels normally consists of a thick layer of polyurethane.

Some cryogenic vessels are used for the storage of gases at the production site, others at the end-user site. Storage vessels used at the production site are not covered by this paper or the scope of ISO technical committee ISO/TC 220, Cryogenic vessels.

Some cryogenic vessels are used for the transportation of gases. The most common are cryogenic trailers used to refill the stationary vessels at end-user sites. Large containers are also transported by road, railroad or sea. All these types of vessels are called “large transportable cryogenic vessels”.

Some other small cryogenic vessels (less than 1 000 litres water capacity) are also filled and transported by companies involved in the supply of industrial or medical gases to the end users.

A large number of cryogenic vessels are being used around the world (see Fig. 8). Table 4 gives the number of such vessels by type.



Figure 8. Cryogenic vessels: schematic and cryogenic trailer

Table 4. Number of different types of vessels being used in the world

Type of vessels	Units					
	Vacuum insulated			Non vacuum insulated		
	Australia	Europe	USA	Australia	Europe	USA
Static vessels	2 000	40 000	50 000	200	20 000	20 000
Small transportable vessels (no more than 1000 L)	3 000	100 000	250 000	-	-	-
Large transportable vessels	200	5 000	5 000	40	1 000	1 000

2.3 Reducing the wall thickness of the vessels

Modern methods like “cold stretching” or “use of cold properties” are still not fully accepted in North America and Japan. These modern methods of designing and manufacturing stationary cryogenic vessels considerably reduce the wall thickness of the vessels. This method of reducing the price of cryogenic vessels by limiting the quantity of expensive materials used (such as stainless steel) is now widely used in Europe.

The principle and detail information on the cold stretching method is given in the ISO 21009-1, Cryogenic vessels – Static vacuum insulated vessels – Part 1: Design, fabrication, inspection and test.

However, all efforts were made to produce efficient ISO standards for stationary cryogenic vessels in an expedient manner. ISO 21009-2, Cryogenic vessels – Static vacuum insulated vessels – Part 2: Operational requirements, is already available, while ISO 21009-1, Cryogenic vessels – Static vacuum-insulated vessels completed and waiting to be issued in the coming months.

2.4 Transport of liquid hydrogen

In order to reduce the volume required to store a useful amount of hydrogen –particularly for vehicles-liquefaction may be employed. Since hydrogen does not liquefy until it reaches -253°C (20 degrees above absolute zero), the process is both time consuming and energy intensive demanding. Up to 40% of the energy content in the hydrogen can be lost (in comparison with 10% energy loss with compressed hydrogen). The advantage of liquid hydrogen is its high energy/mass ratio, three times that of gasoline. It is the most energy dense fuel in use (excluding nuclear reactions fuels), which is why it is employed in all space programmes. However, energy/volume ratio remains low compared to gasoline. Liquid hydrogen is difficult to store over a long period (product loss by vaporisation), and the insulated tank required may be large and bulky.

Liquid hydrogen road transport is carried out using trucks which can exceed a capacity of 60 000 litres. Delivery is achieved either in vacuum insulated containers or by transferring the product to stationary vessels depending on the required quantities.

In the USA there are several pipelines for liquid hydrogen with lengths of up to 40 km.

The intercontinental transport of hydrogen will probably be carried out in liquid form using ships. For this purpose, specialized ships with appropriate tanks and port facilities are being designed. A realization of these ideas will however not take place until the trade in hydrogen reaches an appropriately large scale.

2.5 Material issues

Hydrogen embrittlement (HE)

When the gas is at room temperature, the requirement of section 1.4 applies. For the liquid at low temperature, the specific considerations are:

HE effect is normally attained at ambient temperatures (see Fig. 9) and can often be neglected for temperatures above $+100^{\circ}\text{C}$. In the case of unstable austenitic stainless steels commonly used for cryogenic vessels, the maximum HE effect is attained at -100°C , but can be neglected for temperatures below -150°C (see Fig. 10).

Compatibility of metals and alloys with low temperature

The use of metal at low temperatures entails special problems which must be resolved. Consideration must be given, in particular, to changes in mechanical characteristics, expansion and contractions phenomena and the thermal conduction of the various materials. However, the most important matter to be considered is certainly that of brittleness, which can affect certain metallic items of equipment when they are used at cryogenic temperature. In what follows, we shall only deal with ferritic steels, stainless steels and aluminium alloys, which are the main materials used at low temperatures (see Fig. 11).

Generally, for metallic materials, ductility (characterized by elongation or constriction), and toughness (characterized by impact strength, notch effect strength or crack growth resistance) decrease when temperature is lowered. In the case of ferritic or martensitic steels, toughness drops rather suddenly in a relatively narrow temperature range; the failure mode, which was ductile, becomes brittle.

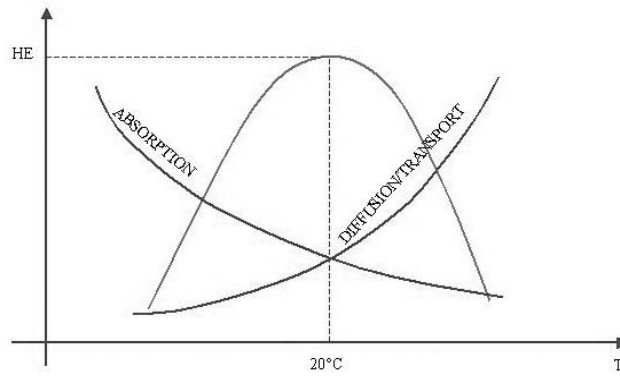


Figure 9. Influence of temperature. Principle

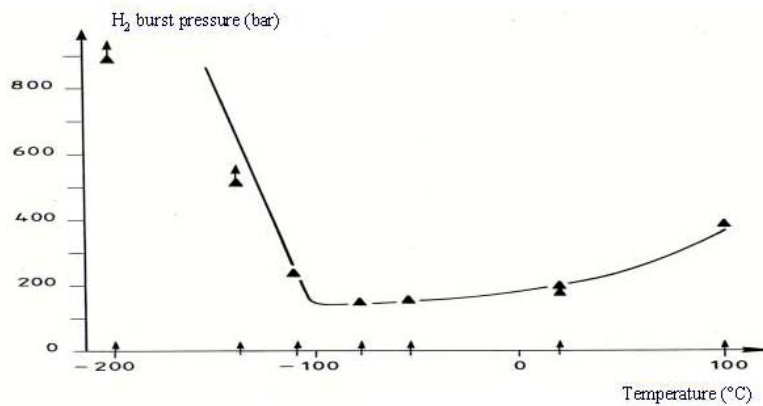


Figure 10. Influence of temperature for some stainless steels

Various types of tests are used to characterize the brittleness of materials due to cold. The most commonly used is the Charpy impact test. This test consists of breaking with an impact testing machine, under well defined conditions, test pieces with a U shaped notch in their middle (see Fig. 12). The absorbed energy is determined, from which strength is calculated. These tests are carried out at different temperatures. Down to -196°C , they can be performed after immersion of the test pieces in a bath set at the desired temperature to bring them to that temperature. The test piece is then rapidly put on the pendulum ram impact testing machine for rupture, while respecting tolerances stipulated in the standards (plus or minus 2°C). For tests performed at lower temperatures, in particular at the temperature of liquid hydrogen or helium, it is necessary to use test pieces « bound » with a special insulation and to monitor the rise in temperature (see Fig. 12) during the impact strength test after immersion in an appropriate bath.

The test results are shown on curves giving the energy needed for the failure of the material, or « strength » of the material, versus its temperature. We noted the temperature or « transition point » of the material, which is the average temperature within the temperature range at which the strength of the material rather suddenly drops. The more and more brittle mode of breaking can also be characterized by an examination of the aspect of the break of test pieces. When temperature drops, « crystallinity » increases; this crystallinity affects about 50 % of the surface of the break at transition temperature and 100 % of the surface in the brittleness range; On examination with a scanning

electron microscope (SEM), the brittle parts (crystalline area) have an aspect of a « cleavage » brittle break with « rivers » whereas the ductile part have a ductile aspect with « cups ».

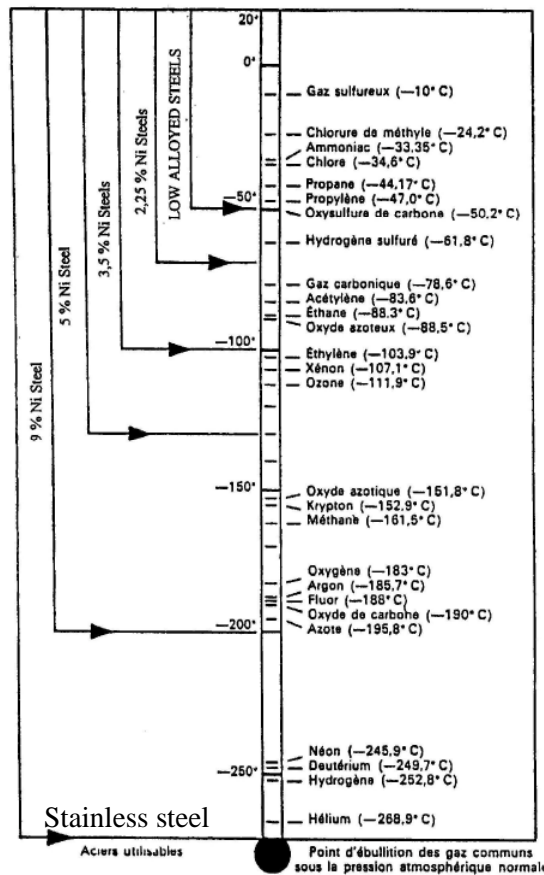


Figure 11. Possibility of using steel for the different cryogenic gases

Other changes in mechanical characteristics

In contrast to toughness and ductility, a lowering of temperature will bring about an increase in the yield point, hardness, the modules of elasticity, breaking strength and the fatigue limit of metals and alloys.

Contraction – Thermal conduction

It is important to consider relative contractions due to a lowering of temperature, in particular for assemblies made of different materials. As an example, it can be seen that the main employed stainless steels contract in appreciably the same way.

In addition, consideration is to be given to the possible transmission of cold by metal parts which are not brittle, while taking into account the thermal conductivity of materials and their specific heat, to parts, whether or not metallic, which are, for their part, brittle at low temperature. To avoid this, appropriate insulation is used.

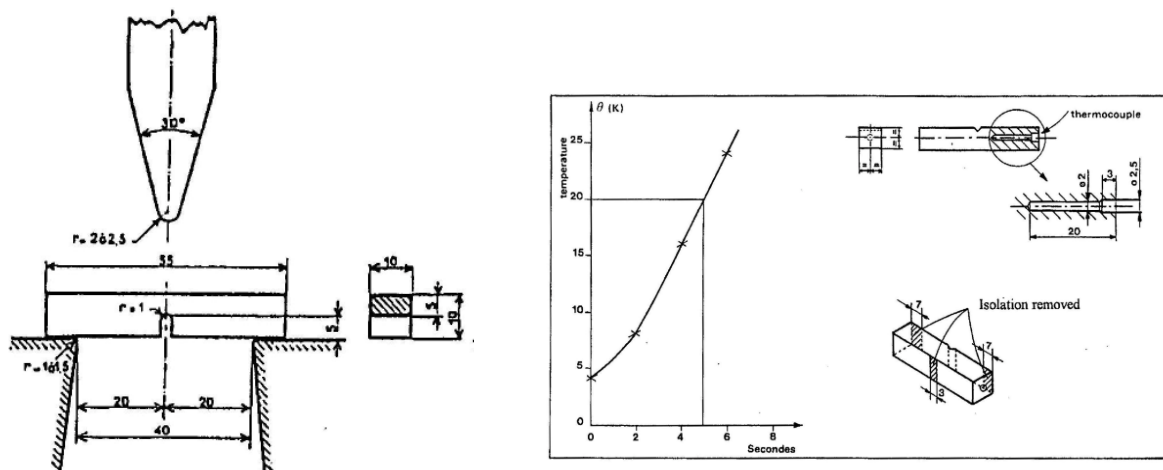


Figure 12. Charpy test – Variant for test at liquid helium temperature

Cryogenic vessels – Conclusion

In conclusion, and as regards cryogenic equipment, materials which are not brittle at minimum operating temperature should be used (nickel ferritic steels down to -200°C or stabilized austenitic steels and aluminium alloys which can be used down to absolute zero).

In the case of materials known not to be brittle when cold, care is nevertheless to be taken with respect to their chemical composition, their heat treatment and their welding.

However, for some applications, it is possible to lighten structures by taking advantage of certain mechanical characteristics of alloys at low temperature.

Finally, consideration is to be given to the contraction of metallic materials when temperatures are lowered and the risks of cold conduction.

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