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Research on the airtight helium leak detection test device of fully-wrapped carbon fiber reinforced cylinders with a plastic liner for the on-board storage of compressed hydrogen

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Abstract: This article aims at the discussion on the airtight helium leak detection test of CHG type IV plastic inner composite gas cylinder. An equipment for test the airtight helium leak detection test was built. And the properties of the airtight helium leak detection of a CHG type IV plastic inner composite gas cylinder was carried out. The results show that at the beginning of the test, the permeability fluctuates while after about 600 min, the test results tend to be stable.

1. Introduction

Hydrogen energy has a high calorific value, which is more than three times that of petroleum, and is an ideal substitute for fossil fuels [1-2]. The combustion of hydrogen energy does not produce any harmful or greenhouse gases after use. It is widely recognized by society and is known as the ' ultimate energy ' in the 21 st century. Hydrogen energy vehicles and supporting hydrogen refueling stations are the main downstream of the hydrogen energy industry chain [3-6]. Vigorously promoting hydrogen energy vehicles is of great significance for achieving the goals of ' carbon peak ' and ' carbon neutralization '.

As an important storage medium for hydrogen in hydrogen energy vehicles, the main existing hydrogen storage methods include compressed gaseous hydrogen storage, low-temperature liquefied hydrogen storage, hydride solid-state hydrogen storage, organic liquid hydrogen storage and composite hydrogen storage. The most industrialized hydrogen storage method is high-pressure hydrogen storage using on-board gas cylinders. At present, the common hydrogen storage pressure is 35MPa, but due to the endurance mileage requirements, the hydrogen storage pressure of the vehicle-mounted gas cylinder to be promoted in the future should reach at least 70MPa[7-8]. However, high pressure means high energy and high potential danger. In addition, under such high pressure, if the metal liner is still used with carbon fiber winding, the increase of metal wall thickness will be very unfavorable to the lightweight demand of the vehicle. It is necessary to use non-metallic liner composite cylinders.

However, the failure mechanism of non-metallic (plastic) liner gas cylinders is complex, and the isotropic performance of plastics is different from that of metal materials [9]. The most important thing is that the gas cylinder has low resistance to external impact damage and poor air tightness. Under high pressure environment, the gas is easy to penetrate from the non-metallic inner liner. In particular, the size of hydrogen molecules is small, and it is easy to penetrate from the inside to the outside of the bottle, so it is difficult to detect.

In this study, the airtight detection device of vehicle-mounted hydrogen energy composite gas cylinder based on helium mass spectrometry was built. And the Helium micro-penetration properties of fully-wrapped carbon fiber reinforced cylinders with a plastic liner for the on-board storage of compressed hydrogen was studied.

2. Design and construction of the airtight detection device

For the plastic liner composite gas cylinder, due to the low resistance to external impact damage and poor air tightness of the cylinder, the gas is easy to penetrate from the non-metallic liner to the outside under high pressure environment. In the test, not only the gas leakage at the valve should be considered, but also the leakage of the cylinder body should be considered. In particular, the size of the hydrogen molecule is small and it is easy to penetrate from the inside to the outside of the cylinder. The traditional immersion method cannot fully measure the gas tightness of the cylinder.

According to GB GB/T 42612-2023 “Fully-wrapped carbon fiber reinforced cylinders with a plastic liner for the on-board storage of compressed hydrogen as a fuel for land vehicles”, the helium mass spectrometry method should be used to test the airtightness of such gas cylinders. And the vacuum chamber needs to be built for testing.

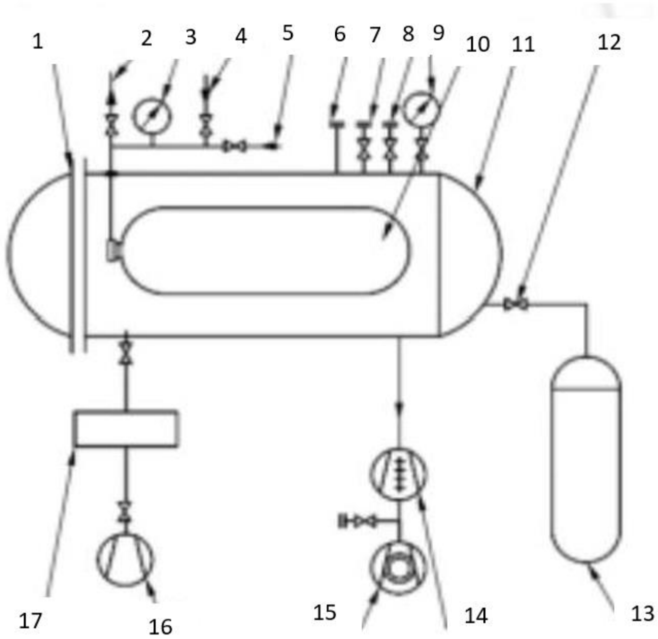

Figure 1: The schematic diagram of the vacuum chamber.

Fig. 1 shows the schematic diagram of the vacuum chamber. 17 parts could be seen in the device. The appellation of each part is shown in Table 1.

Table 1: The appellation of each part of the vacuum chamber.

No.	Name
1	Fast open-door of the vacuum chamber
2	Inflatable and exhaust pipelines
3	Pressure gauge
4	Helium filling pipeline
5	Nitrogen filling pipeline
6	Mechanical explosion-proof interface
7	Air exchange port
8	Standard leak joint
9	Pressure gauge
10	Testing Cylinders
11	Vacuum chamber
12	Nitrogen flush valve
13	Nitrogen supply
14	Vacuum pump
15	Mechanical pulp
16	Leak detection pump
17	Helium leak detector

While started to test, this device was used to test by following steps:

(1) Open the sealing door on the airtight test tank, and the operator transports the cylinder filled with the test gas to the fixed position in the airtight test tank, then close the sealing door.

(2) Vacuumize the vacuum chamber.

(3) Open the inlet valve and outlet valve, close the test mouth, and discharge nitrogen and the Helium from the nitrogen tank, until the cylinder is filled with test gas.

(3) Close the inlet and outlet valves, start timing and hold the cylinders. Then measured the helium concentration in the vacuum chamber.

(4) Open the inlet valve and outlet valve and close the test port. Then discharge the permeable gas in the closed test tank.

(5) Close the inlet and outlet valves, open the sealing door and remove the test cylinder.

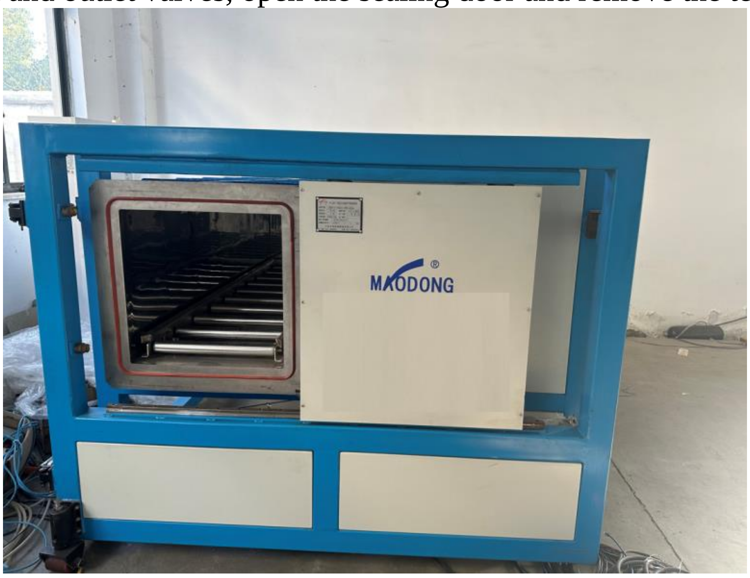

Figure 2: Vacuum chamber object

According to the test principle, we built a vacuum chamber, as shown in Fig.2, and further optimized the device. In this object, a vehicle-mounted helium leakage detection box (vacuum chamber) for hydrogen bottles is designed. The box body is provided with a holding space, and an opening connected with the holding space is arranged on one side of the box body. The cover body is sealedly connected with the box body for opening/closing the opening, the base of the gas cylinder is set in the accommodation space, and two groups of parallel chutes are set on the top of it. There are two sets of moving rollers at the bottom of the inlet and outlet frame. The inlet and outlet frame cooperates with the chute through the moving roller, so that the inlet and outlet frame can be moved to the top of the gas cylinder placement base, and the inlet and outlet frame is used to place the gas cylinder in and out of the box. A vacuum tube, a leak detection probe and an inflatable tube are also arranged on the box. By optimizing the structure of the explosion-proof box, a closed holding space is provided to place the gas cylinder. During the detection process, the air in the holding space is discharged through the vacuum tube, so that there is no external factor affecting the detection value in the detection process, which can effectively solve the problems existing in the existing technology.

3. Experiments and results

In this paper, after the test device is built, a CHG type IV plastic inner composite gas cylinder with nominal volume of 390L was chosen to carry out the gas permeability test. In order to save the test gas, the air in the bottle is exhausted, and the helium is first filled to 1.75 MPa. Then continue to fill nitrogen to 35 MPa, it can be concluded that the volume fraction of helium is 5 %. Then the gas cylinder was static, and the helium concentration in the vacuum chamber was measured at 1min, 5min, 10min, 20min, 30min, 1h, 2h, 5h, 10h, 24h, 36h, 48h, 72h, and the cumulative helium permeability of the gas cylinder at these time points was calculated. The main macroscopic pictures and main parameters of the gas cylinder are shown in Fig.3 and Table 2.

Figure 3: Macro image of test cylinder

Table 2: Main parameters of gas cylinder

Parameters	Values
Diameter	585mm
Volume	390L
Nominal working pressure	35MPa
Length of cylinder	2090mm
Weight of empty cylinder	140kg

Table 3 shows the helium concentration and corresponding release rate detected by mass spectrometry in the vacuum chamber under different standing time of the gas cylinder. It can be seen that on the whole, the gas cylinder began to penetrate from 1 min after standing, and the penetration rate was about 0.5650 ml / (h ·L). After 24 h stabilization, the gas permeation rate was about 0.48 ml / (h ·L), and the overall change was not significant.

Table 3: Helium concentration and leakage rate in vacuum chamber under different standing time of gas cylinder

Time	**Helium concentration(ppm)**	**Volume of helium(ml)**	**Leakage rate (Pa ·m3/s)**	**Leakage rate (ml/(h ·L))**
0min	0.0760	0.166	0	0
1min	0.1402	0.1836	6.813×10^{-3}	0.5650
5min	0.4049	0.9406	6.334×10^{-3}	0.5788
10min	0.8115	2.1037	7.883×10^{-3}	0.6473
20min	1.5161	4.1188	6.934×10^{-3}	0.6337
30min	2.2200	6.1319	6.882×10^{-3}	0.6289
1h	4.0905	11.4814	6.433×10^{-3}	0.5888
2h	7.6925	21.7832	6.112×10^{-3}	0.5585
5h	18.4069	52.4264	5.844×10^{-3}	0.5377
10h	33.9838	96.9764	5.442×10^{-3}	0.4973
24h	79.9744	228.5094	5.343×10^{-3}	0.4883
36h	119.0264	340.1981	5.303×10^{-3}	0.4846
48h	158.5569	453.2553	5.299×10^{-3}	0.4842
72h	237.6627	679.4980	5.296×10^{-3}	0.4840

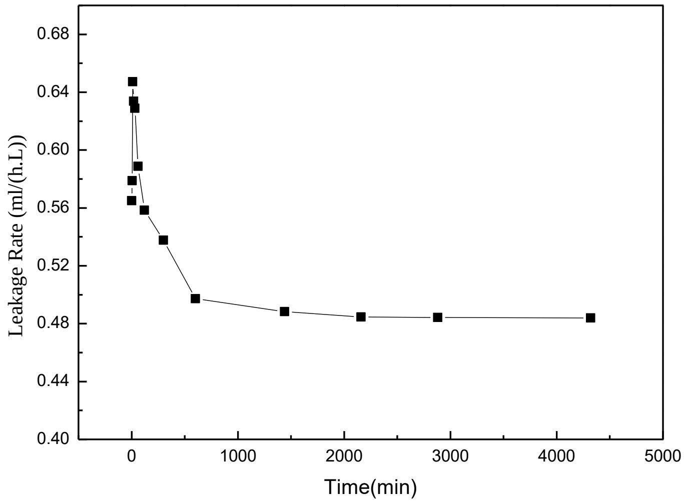

Figure 4: Comparison curve of helium leakage rate and cumulative time

Fig.4 shows the comparison curve of helium leakage rate and cumulative time,.As can be seen, the helium leakage rate of the gas cylinder is compared with the standing time to draw a curve. It can be seen that at the beginning of the test, the permeability fluctuates due to the instability of the leakage rate. Subsequently, the leakage rate began to gradually decrease. After about 600min (10h), the test results tend to be stable.

4. Conclusions

This article aims at the discussion on the airtight helium leak detection test of CHG type IV plastic inner composite gas cylinder.

(1) An equipment for test the airtight helium leak detection test was built.

(2) Properties of the airtight helium leak detection of a CHG type IV plastic inner composite gas cylinder was carried out. The test results show that at the beginning of the test, the permeability fluctuates while after about 600min (10h), the test results tend to be stable.

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