Research on the Rupture of Certain CNG-II Gas Cylinders during the Leak-Before-Break Test

Shutao Xionga, Song Huanga*, Bin Lib, Zhongguo Zhaoc

National Compress Natural Gas Cylinder Quality Supervision and Inspection Centre, Chongqing Special Equipment Inspection and Research Institute, Chongqing, China

*Corresponding author e-mail: amwmmswc_huang@163.com, a410011646@qq.com, b13594034267@189.cn, c489279151@qq.com

Abstract. We are studying the Leak-before-break (LBB) test, which is an important test for the safety performance verification of CNG-II bottles, and it is a necessary link for new product identification. We ruptured during the LBB test of a CNG-II gas cylinder. Through the chemical composition analysis, metallographic observation, mechanical performance test, scanning electron microscope and other tests of the failed gas cylinder, we found that the cylinder failure was due to the curved part. There are oxide slag inclusions. Under the fatigue test under the action of alternating load, slag inclusions are used as the fatigue source, which accelerates the crack growth and eventually causes the cylinder to fail.

1. Introduction

CNG cars are cars with compressed natural gas as their fuel, and their main energy storage container is CNG gas cylinders. Internationally, CNG gas cylinders for vehicles are divided into four models, including CNG-I (metal gas cylinder), CNG-II (hoop-wrapped composite cylinder), CNG-III (Full-wrapped composite cylinders) and CNG-IV type (full-wrapped gas cylinders with plastic liner), of which CNG-II gas cylinders occupy 60% of CNG gas cylinders, and are currently the most widely used gas cylinders. Both GB 24160-2009 [1] and ISO 11439: 2013 (E) [2] standards stipulate that each new CNG cylinder must be type tested before production. The type test is to simulate the service in order to verify whether the product can be used normally. Environmental testing is an essential part of new product identification. Only by passing the type test can the product be formally put into production [3]. In the cylinder properties test, the Leak-before-breakage test is an important test. It is a service situation that simulates the repeated charging and deflation of a gas cylinder under a high pressure environment [4]. A pressure cycle is performed between the test pressures, and the cycle is tested at a specified cycle rate to failure or more than 45,000 times, and the cylinder does not burst. This test simulates the situation of overpressure filling [5]. The gas cylinder must meet the requirements of allowing leakage failure and not blasting failure, so as to protect the safety of people and property in actual use, which has great practical significance [6].

This article describes a CNG-II gas cylinder with parameters of 356mm in diameter and a nominal working pressure of 20MPa. During the LBB test, the cylinder end ruptured and failed, and the cylinder end was lifted open (as shown in Figure 1). If this phenomenon occurs in actual use, it will cause huge losses to people and property, and do not meet the requirements of manufacturing...
standards[7]. This article analyzes the failure behavior of the cylinder to find out the cause of the failure, so as to avoid the occurrence of this cause in the next cylinder production.

![Failure of cylinder](image)

**Figure 1.** Failure of cylinder

2. **The test method**
Several samples from different parts of the cylinders were selected for macro observation, chemical composition, optical microstructure, tensile properties and scanning electron microscope. The spectroscopic methods were utilized as follows.

2.1. **Chemical composition**
Chemical composition samples were cut from the cylinder near the failure area and tested by x-ray fluorescence spectrometry (PDA 7000).

2.2. **Optical microstructure**
For microstructural examination, specimens near the failure area with no crack were cut. The samples were mounted and polished. After preparation, the samples were etched by nital 2% solution for 15 s. Then, the cross-section was observed by optical microscope (Aximumer.A1m, Zeiss).

2.3. **Mechanical properties**
Tensile properties test was used for exam the mechanical properties of the gas cylinder. Three specimens near the failure area were chosen and tested by an electronic universal testing machine (SANS) with the speed of $5\times10^4$s$^{-1}$.

2.4. **Scanning Electron Microscope (SEM)**
The fracture of the gas cylinder were investigated by SEM (SU-3500, Hitachi) equipped with an energy dispersive X-ray spectrometer (EDS).
3. Test results

3.1. Macro image of the failure

Figure 2. The appearance of the breach

Figure 2 is a macro picture of the gas cylinder break. It can be seen that the fracture has the presence of a zigzag step, has no metallic luster, and has obvious brittle fracture characteristics.

3.2. Routine analysis of materials around the breach

In order to verify whether there is a problem with the matrix material, a good matrix material was selected near the breach for component analysis, metallographic analysis, and mechanical property testing.

The chemical composition analysis results are shown in Table 1. It can be seen that the main alloy elements are Cr and Mo, the barrel material type is 30CrMo steel, the impurity elements P and S are also within the standard (GB 24160) range, and the chemical composition does not show abnormality.

Table 1. Chemical composition of materials in different positions /%

| No. | C     | Si    | Mn    | P     | Cr   | Mo   | S     | Ni   | Cu   |
|-----|-------|-------|-------|-------|------|------|-------|------|------|
| 1   | 0.25  | 0.25  | 0.51  | 0.012 | 0.94 | 0.17 | 0.006 | 0.03 | 0.063 |
| 2   | 0.24  | 0.25  | 0.52  | 0.012 | 0.93 | 0.17 | 0.007 | 0.03 | 0.064 |
| 3   | 0.23  | 0.24  | 0.52  | 0.011 | 0.93 | 0.17 | 0.006 | 0.03 | 0.066 |
| 4   | 0.23  | 0.24  | 0.51  | 0.011 | 0.92 | 0.17 | 0.007 | 0.03 | 0.061 |
| Ave | 0.24  | 0.25  | 0.51  | 0.011 | 0.93 | 0.17 | 0.007 | 0.03 | 0.063 |

GB 24160 ≤0.37 0.15~0.37 0.40~0.90 ≤0.020 0.80~1.20 0.15~0.35 ≤0.020 -- ≤0.20

Figure 3 shows a photo of the metallographic structure around the breach. It can be seen that the metallographic structure of the cylinder liner material is relatively uniform, consisting of tempered sorbite and ferrite, and fine needle-shaped tempered sorbite is uniform. Distributed on the ferrite matrix, no coarse Weiss structure and band structure were found. The grain size was 10 grades, which is a typical quenched and tempered structure of chromium-molybdenum steel.
Table 2 shows the test results of the mechanical properties of the inner cylinder of the cylinder. A total of 3 samples were taken for testing. The results are shown in Table 2. The mechanical properties of the material are relatively uniform. The tensile strength is greater than 820 MPa, less than 880 MPa, the yield strength is greater than 690 MPa, the yield ratio is less than 0.92, and the elongation is greater than 14%. The results are similar to the quenched and tempered results of general 30CrMo steel.

Table 2. Test values of mechanical properties

| Sample number | Tensile strength Rm/MPa | Yield strength Rea/MPa | Yield ratio | Elongation A/% |
|---------------|-------------------------|------------------------|-------------|---------------|
| 1#            | 823                     | 726                    | 0.88        | 17.5          |
| 2#            | 837                     | 741                    | 0.89        | 18.0          |
| 3#            | 835                     | 744                    | 0.89        | 17.5          |

3.3. Fracture analysis
In order to further analyze the cause of the failure of the cylinder, we performed SEM analysis on the sampling of the breach. The SEM picture is shown in Figure 4. Figure 4a is a picture with a magnification of 20 times. The existence of shell lines can be clearly seen, which shows that the cylinder is fractured by the action of alternating load, which is a typical fatigue fracture characteristic. Further magnification (Figure 4b), it can be seen that there is a clear fatigue crack propagation region between the crack source and the transient fault region.

Figure 4. The shell line at the breach a) 20 times magnification; b) 100 times magnification
We further search for the crack source through the shell line. As shown in Fig. 5, obvious pits can be observed, which the fatigue crack source is. Figure 5 shows the micro-morphology of the crack source area and the crack first-stage expansion area. Figure 5a and Figure 5b are micrographs of the two crack source areas, respectively. Fatigue cracks initiate at the material defects near the inner surface of the inner liner of the cylinder, and then expand in a fan shape around. Figures 5c and 5d are high-magnification images of the crack source area. There is a fan-shaped cleavage zone around the crack initiation defect, and its cross-section is flat, which together form the fatigue core region with the crack initiation defect.

![Figure 5. Fatigue crack nucleation area. Fig. 4c is an enlarged photo of the red area in Fig. 4a, and Fig. 4d is an enlarged photo of the red area in Fig. 4b.](image)

We further enlarged the crack source pits. As shown in Figure 6, inclusions can be seen in the pits. Spectral analysis was performed on the pits in the crack source area. The results are shown in Table 3, where pt1 ~ pt4 are Inclusions, pt5 is the matrix. It can be seen that the oxygen content of the inclusions in the pit is high, and it can be considered that the inclusions are mainly oxide slag.
3.4. Discussion about the Failure Behavior

From the above analysis, it can be known that, for the cylinder material itself, the chemical composition, mechanical properties, and metallographic structure are in line with the design requirements and GB 24160-2009 / ISO 11439 standard requirements. It can be inferred that there is no problem in the overall liner material. Failure should be caused by defects in local areas of the material.

Scanning electron microscopy and energy spectrum analysis of the fracture position found that oxide slag was found on the inner surface and near the inner surface of the cylinder. These slag inclusions should be defects of the material itself and formed during steel smelting. The steel pipe containing slag is just used in the curved part. In subsequent fatigue tests, the arc-shaped part of the cylinder is the stress concentration area. Under the action of alternating loads, fatigue cracks are more likely to occur than other parts, and the presence of oxide slag accelerates the fatigue crack growth and eventually leads to cylinder rupture failure.

4. Conclusion

In this paper, through macro observation, chemical composition analysis, metallographic structure observation, mechanical property test, scanning electron microscope and other tests, the rupture phenomenon of a certain CNG-II type cylinder during the Leak-before-breakage test is analyzed. The failure of the cylinder is considered to be due to the slag inclusion of oxides in the arc-shaped part, the slag inclusion is used as a fatigue source under the fatigue test under the action of alternating load, which accelerates the crack propagation, and eventually results in the failure of the gas cylinder.

**Table 3.** Composition table of each energy spectrum position in FIG. 6 (wt %)

|   | C-K | O-K | Si-K | P-K | S-K | Cl-K | Ca-K | Cr-K | Fe-K | Mo-L |
|---|-----|-----|------|-----|-----|------|------|------|------|------|
| pt1 | 13.91 | 21.86 | 1.10 | 0.46 | 0.46 | 2.81 | 59.39 | | | |
| pt2 | 9.40 | 27.13 | 0.84 | | | 0.30 | 1.29 | 61.03 | | |
| pt3 | 9.79 | 17.45 | 0.58 | 0.86 | | 0.53 | 3.49 | 66.22 | 1.09 | |
| pt4 | 14.59 | 24.87 | 0.30 | 0.39 | 0.55 | 0.20 | 0.34 | 1.66 | 57.10 | |
| pt5 | 7.38 | 4.44 | | | | | | 1.38 | 86.80 | |
References

[1] GB 24160-2009. Hoop-wrapped composite cylinder with steel liner for the on-board storage of compressed natural gas as a fuel for automotive vehicles.

[2] ISO 11439-2013. Gas cylinders — High pressure cylinders for the on-board storage of natural gas as a fuel for automotive vehicles.

[3] Lu Hongliang, Xue Xiaolong, Tang Xiaoying, etc. Research progress on safety supervision technology of gas cylinders. China Special Equipment, 5, 2015, pp.26-34.

[4] Liu Zhiyan. Relevant issues in the installation and inspection of taxi CNG cylinder. China Special Equipment Safety, 02, 2015. pp.35-37.

[5] Huang Xiaoyu, Wu Yanfeng, Zhang Yiming, etc. Discussion on periodic inspection of LNG gas cylinders on board vehicles. Chemical Engineering and Preparation, 02, 2015, pp.191-192

[6] Yang Yong. Safety inspection and quality management of CNG gas cylinders for vehicles. Science and Technology Information, 07, 13, 2015, pp.111.

[7] Yao Jian. Discussion on issues related to gas tightness test standards for compressed natural gas (CNG) cylinders for automobiles. Petrochemical Safety and Environmental Protection Technology, 04, 28, 2012, pp.6, 32-34, 58.