FREEZE OF NOZZLE/RECEPTACLE DURING HYDROGEN FUELING

Hiraki, W.[1] and Mitsuishi, H.[2]
[1,2] FC-EV Research Division, Japan Automobile Research Institute,
1328-23 Takaheta, Osaka, Shirosato, Ibaraki 311-4316, Japan
[1]whiraki@jari.or.jp, [2] hmituisi@jari.or.jp

ABSTRACT

We conducted a fueling test with hydrogen gas for a safety evaluation of the nozzle/receptacle at a controlled temperature and humidity. Test results confirmed that the nozzle/receptacle froze under specific conditions. However, freezing did not cause apparatus damage nor hydrogen leakage. The nozzle/receptacle is thus able to fuel safely even if the nozzle/receptacle is stuck due to ice. In addition, we quantified the water volume that causes freezing.

1.0 Introduction

We examined and tested the fueling method complying with SAE TIR J2601[1] used for fueling hydrogen into fuel-cell vehicles safely and efficiently at commercial hydrogen stations in Japan. When hydrogen gas is admitted into the fuel tank, the gas temperature inside the tank rises due to adiabatic compression[2]. Therefore, precooled fueling[3], [4] by cooling hydrogen gas to -40℃ is necessary to fully charge with hydrogen gas the fuel tank within a short time. In stations where demonstration tests of -40℃ precooled fueling are conducted, the water condensed in the nozzle/receptacle is sometimes frozen and the nozzle becomes impossible to disconnect. In such cases, besides the nozzle being unable to be disconnected, the ice generated at the nozzle/receptacle joint may influence the air tightness of the seal. Therefore, it is necessary to understand nozzle/receptacle freezing and to verify its safety before introducing -40℃ precooled fueling to commercial hydrogen stations. Furthermore, data useful for establishing the test method must be collected based on the assumption that an evaluation test method for freezing of the nozzle/receptacle will be proposed to ISO 17268[5], the standard for hydrogen fueling connectors.

In this study, we conducted connection-disconnection tests with the nozzle/receptacle assuming introduction of -40℃ precooled filling, checked for hydrogen leaks, when the nozzle/receptacle froze, and quantified the volume of moisture that influences freezing in order to develop a test method for ISO 17268.

2.0 Safety Evaluation Test

2.1 Nozzle/receptacle

The nozzles used for the test are depicted in Fig. 1 and the receptacle, in Fig. 2. Both Nozzle A and B, which are commercially available, were used. Each nozzle has a receptacle-seal structure complying with SAE J2600[6]. A check valve is installed inside the nozzle, and a safety mechanism is provided to prevent connection of the nozzle to the receptacle if there is pressure in the upstream side of the nozzle and to prevent disconnection of the nozzle if there is any residual pressure.

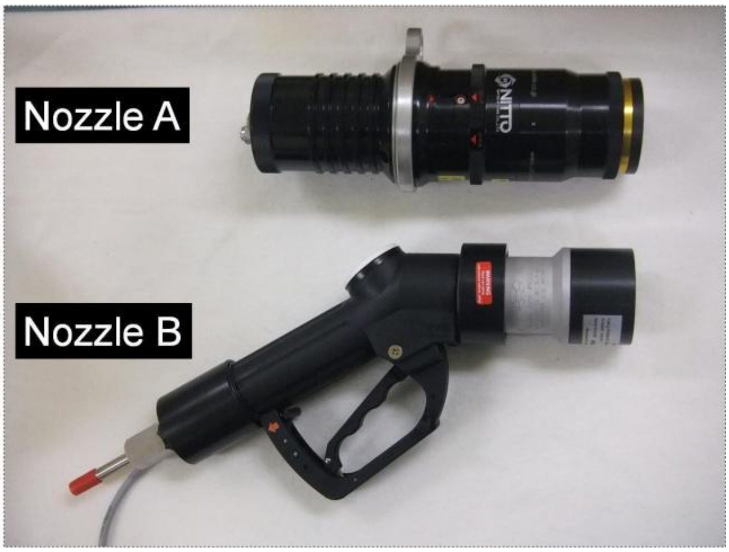

Figure 1. Nozzle A and Nozzle B

Figure 2. Receptacle

2.2 Experiment setup and test procedures

Experiment setup is presented in Fig. 3. The nozzle and receptacle were placed in a constant-temperature, constant-humidity bath ("constant-temperature bath"). Three receptacles were installed to conduct hydrogen-fueling tests repeatedly with high efficiency at the same initial temperature. The hydrogen gas temperature inside the piping and the surface temperature of the nozzle/receptacle were measured using a T-type thermocouples. The constant-temperature bath was filled with nitrogen gas to assure safety. A cycle of hydrogen fueling was conducted at a gas pressure of 35MPa and a gas flowrate of 0.67kg/min (2kg/3min). After discharging the hydrogen gas remaining in the nozzle/receptacle ("depressurizing"), the possibility of manual disconnection of the nozzle was checked. If it was impossible to disconnect the nozzle from the receptacle, the nozzle was judged to have frozen. A cycle of hydrogen fueling and nozzle disconnection was repeated until the nozzle froze and became stuck. To propose this as the standard test method, a maximum of five consecutive cycles were conducted under the same conditions, taking into consideration the overall test duration. If the nozzle/receptacle stuck and could not be disconnected, disconnection was attempted again after 3min. Hydrogen leakage was checked with a gas detector (Type PE-2DC, made by Shin-Cosmo Electric, lower detection limit 20ppm). The gas detector common to all set at the top of the incubator for all receptacles. Damage to the O-ring was evaluated by inspecting the interior of the receptacle after the nozzle was disconnected.

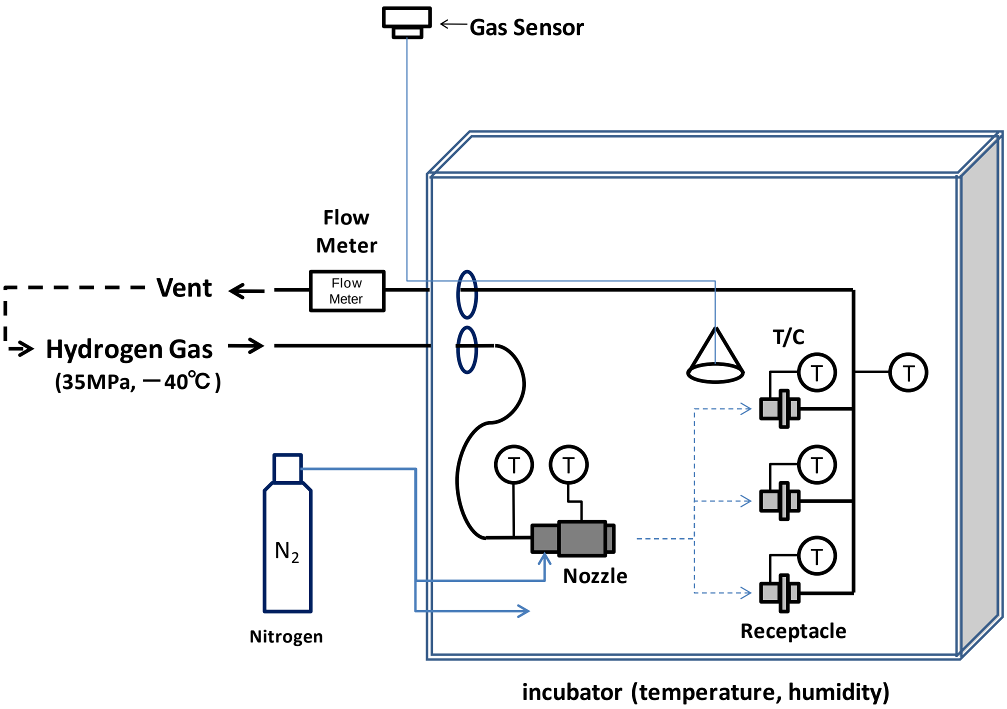

Figure 3. Experiment setup

2.3 Range of tested parameters

The test conditions are listed in Table 1. Tests were conducted while changing the test conditions as follows: the test temperature was changed to -10℃, 15℃, and 30℃, and the relative humidity was changed to 60% and 95%. Tap water was used to add moisture, and tests were conducted with no moisture added, moisture added to the nozzle alone, moisture added to the receptacle alone, and moisture added to both the nozzle and receptacle. A commercially-available spray was used to add moisture. The moisture-adding position for the nozzle joint is indicated in Fig. 4. To spraying into the nozzle charging port, the port was covered so that no moisture entered the interior of the nozzle pin, where hydrogen gas flows. The spray was applied to three points, marked × in Fig. 4, so that the moisture would attach there. To add moisture to the outer surface of the nozzle, the spray was applied to the surface of the sleeve from three directions perpendicular to the axis from a distance of 100mm so that the moisture would attach there. To add moisture to the receptacle, the spray was applied from three directions in the same manner as for the outer surface of the nozzle so that the moisture would attach to the surface of the joint. To remove moisture, the nozzle was wiped and dried at 40℃ in a constant-temperature bath.

Table 1. Range of tested parameters

Temperature [℃]	-10, 15, 30
Humidity [%RH]	60, 95
Water added	None, Nozzle, Receptacle, Both

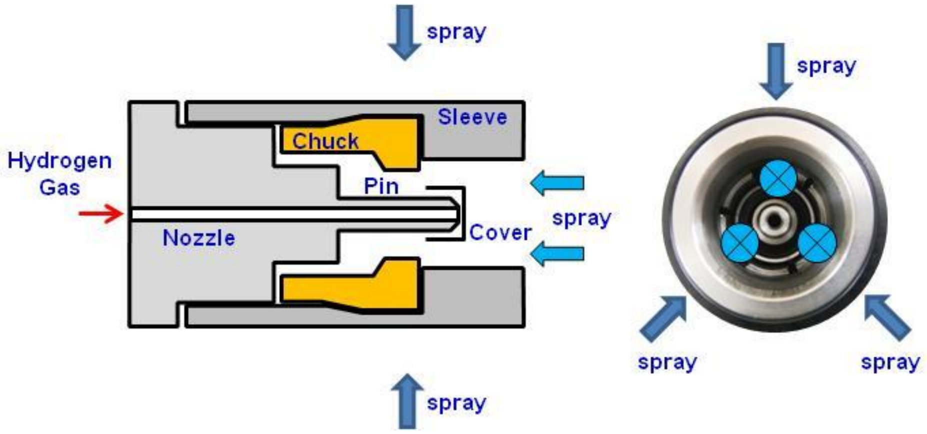

Figure 4. Water spraying position

2.4 Test Results and discussion

2.4.1 Safety when nozzle froze

Changes in pressure, temperature and hydrogen concentration during the Nozzle A fueling test when freezing which is impossible to disconnect the nozzle from the receptacle occurred after the second fueling cycle are depicted in Fig. 5. The pressure rose to 35MPa immediately after fueling began and stayed at that level until the fueling ended. Furthermore, the surface temperature of the receptacle drops to around -20℃ when it is fueled with -40℃ precooled gas. After fueling, the pressure dropped to 0MPa and the receptacle temperature began to rise. After each fueling cycle, the disconnection was checked for hydrogen concentration. Although a small volume of hydrogen gas remained in the joint section when the nozzle/receptacle was disconnected, hydrogen leakage due to lack of air tightness, possibly due to a damaged O-ring, was not detected in either the non-frozen or the frozen case.

Next, during the fueling test with Nozzle B, changes in pressure, temperature, and hydrogen concentration when the case nozzle froze after the fifth fueling cycle are depicted in Fig. 6. As in the case of Nozzle A (Fig. 5), a small volume of hydrogen gas remained in the joint section when the nozzle/receptacle was disconnected, but no hydrogen leakage due to the lack of air tightness, possibly caused by a damaged O-ring, was detected in either the non-frozen or the frozen case. Therefore, hydrogen did not leak from Nozzle A or Nozzle B even if the nozzle/receptacle were frozen or stuck.

Furthermore, when the nozzle was repeatedly connected/disconnected visual inspection of the O-ring of the receptacle after being disconnected did not reveal any abnormality due to freezing, such as damage to or dropping of the O-ring (Fig. 7). Therefore, this test confirmed that freezing did not cause hydrogen leakage or any damage to the O-ring.

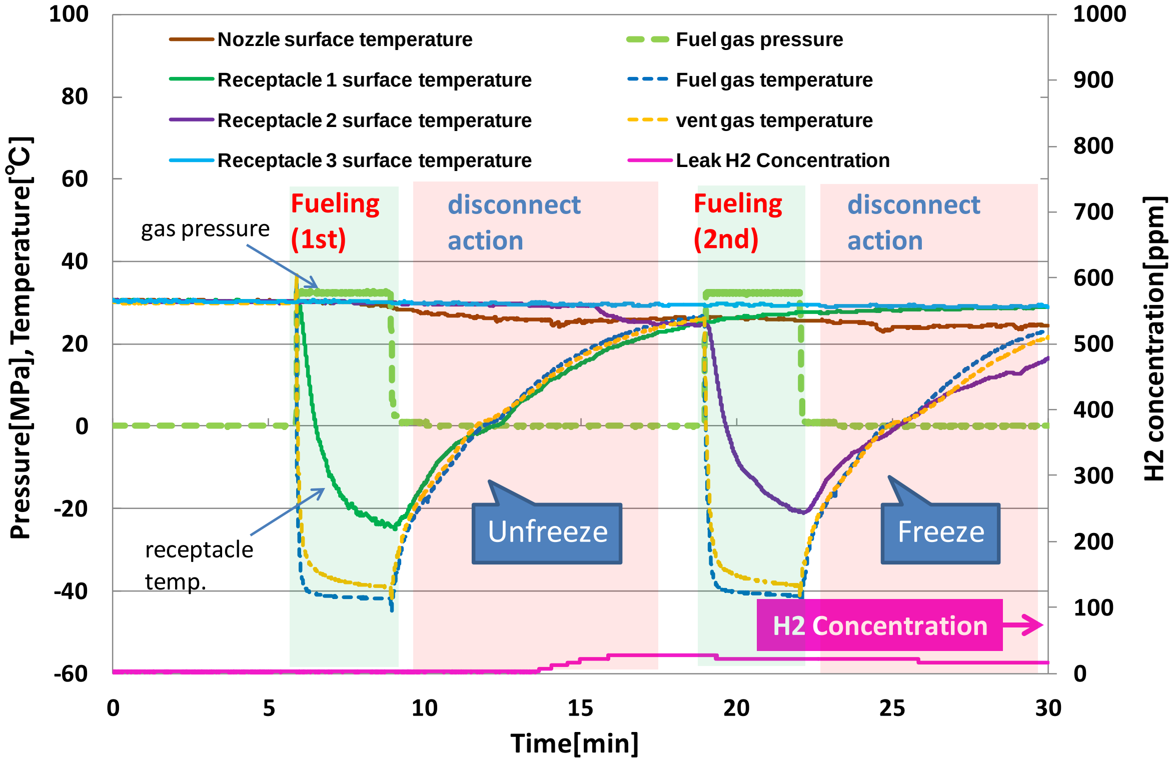

Figure 5. Hydrogen concentration during tests (nozzle A, 30℃, 95%RH)

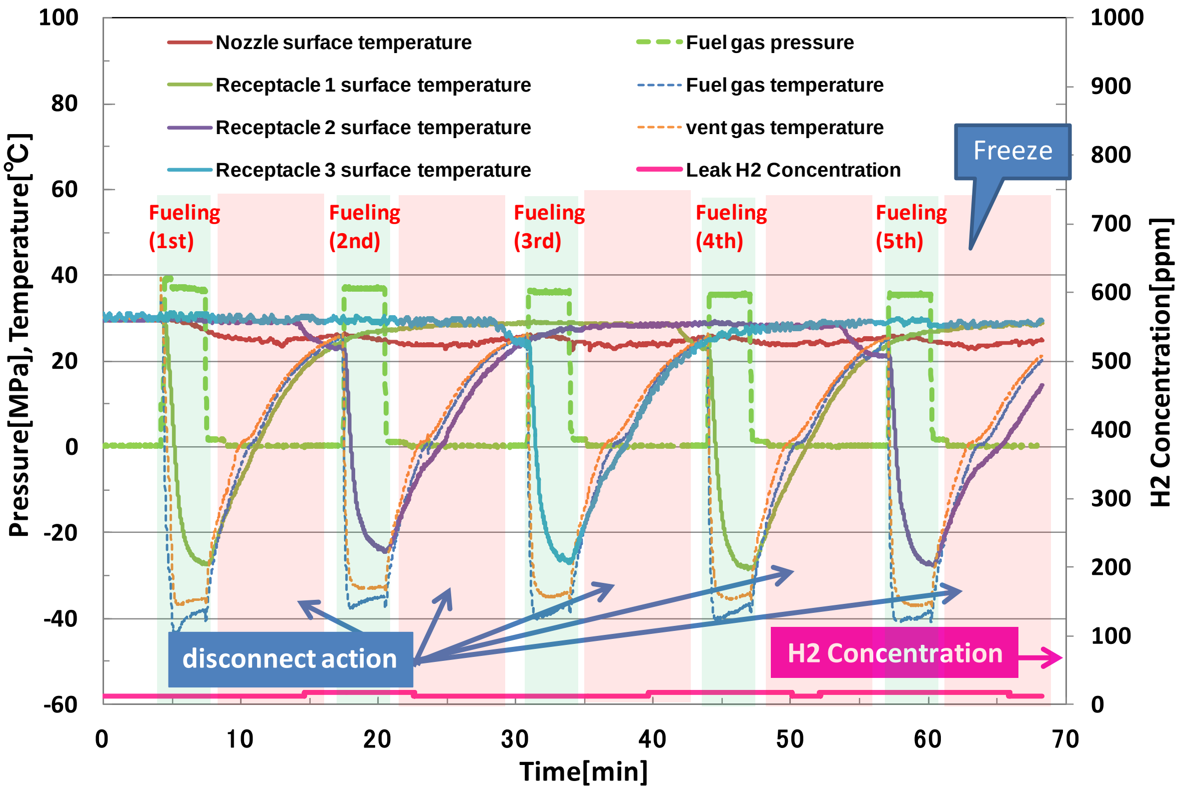

Figure 6. Hydrogen concentration during tests (nozzle B, 30℃, 95%RH)

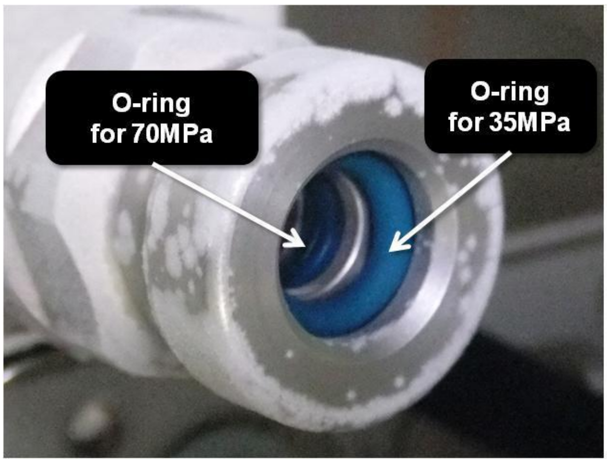

Figure 7. O-ring in receptacle after freezing

2.4.2 Effects of adding moisture

When moisture was added using a spray, it was possible to melt the freezing on the joint by turning the nozzle, but it was impossible to disconnect the nozzle because the sleeve portion of the nozzle was frozen and unable to operate (by sliding). Furthermore, when moisture was added after two cycles of hydrogen fueling and connection-disconnection, the added moisture was frozen at the chuck together with the moisture in the nozzle, so it was impossible to connect again. Thus, if there is moisture, the nozzle cannot be disconnected because moisture in the slide mechanism of the sleeve or the chuck section will freeze. However, we were unable to quantize the influence of moisture on freezing by the spraying method.

3.0 Moisture quantizing test

3.1 Test method

To determine the conditions for freezing and sticking of the nozzle/receptacle, tests for quantizing the volume of moisture required for freezing were conducted using Nozzle A. The constant-temperature bath was filled with nitrogen gas to ensure safety, and one cycle of hydrogen fueling was accomplished at a gas pressure of 35MPa and a gas flowrate of 0.67kg/min (2kg/3min). After depressurizing, we checked if the nozzle could be disconnected manually.

If the nozzle could not be disconnected from the receptacle, we judged that freezing had occurred. Here, we explored a method to evaluate any sticking of the nozzle by one cycle of connection-disconnection.

3.2 Test conditions

The test conditions for the test to determine the nozzle/receptacle sticking conditions were a temperature of 30℃ and a relative humidity of 60% based on the result of the nozzle/receptacle freezing test. The moisture-adding position is indicated in Fig. 8. Deionized water was used to add moisture. The deionized water was measured by a micropipette and, with the nozzle inclined to approx. 30 degrees, water was poured directly into the interior from the nozzle chuck section. After water was added, the nozzle was connected to the receptacle and hydrogen fueling was conducted. The volume of water poured was 500μL, 750μL, or 1000μL considering the capacity of the nozzle chuck section.

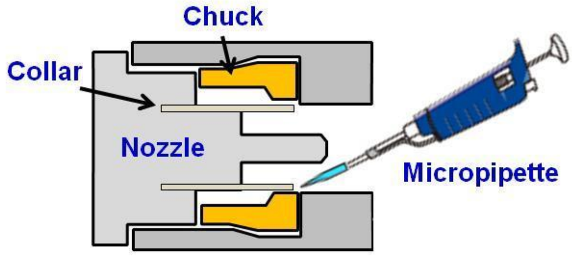

Figure 8. Water supply position

3.3 Test results and considerations

The results as to whether the nozzle can be disconnected after adding moisture to the nozzle to extract the nozzle/receptacle sticking conditions are listed in Table 2. The nozzle could be disconnected when 500μL or 750μL of water was poured in, but not when 1000μL was poured in. If the nozzle cannot be disconnected because sliding is disabled, the cause can be assumed to be that moisture entered the clearance related to sliding, was frozen by -40℃ gas, and filled critical spaces. In this test, however, we were unable to clarify where in the nozzle moisture was frozen.

Table 2. Test results

Water added [μL]	Disconnection		
	Rotate(Sleeve part)	Slide(Sleeve part)	Disconnect
500	possible	possible	possible
750	possible	possible	possible
1000	possible	impossible	impossible

4.0 Conclusions

We conducted nozzle/receptacle connection-disconnection tests assuming -40℃ precooled fueling. We then checked for hydrogen leaks when the nozzle froze and tried to quantize the volume of moisture that influences freezing. The findings were as follows.

・When moisture enters the nozzle/receptacle, the sleeve and chuck sections of the nozzle may stick due to freezing and thus the nozzle becomes impossible to disconnect. However, no hydrogen leakage or damage to the O-ring due to freezing was detected.

・The nozzle could be disconnected when 500μL or 750μL of water was poured in. When 1000μL of moisture enters the nozzle, sticking occurred due to freezing and the nozzle became impossible to disconnect.

The above results confirmed that the possibility of hydrogen leakage or damage to the apparatus is low and that it is safe for the nozzle/receptacle to freeze at the hydrogen station. However, freezing at the hydrogen station will prolong fueling time. We recommend identifying precisely what portion may prevent disconnection of the nozzle if moisture enters it and freezes for establishing an evaluation test method on freezing of the nozzle/receptacle for ISO 17268, the standard for hydrogen-fueling connectors. We will examine other freezing conditions to explore their detailed phenomena and will suggest an evaluation method in the future.

ACKNOWLEDGEMENTS

This study is a summary of the “Hydrogen Using Technology Research and Development Project,” under consignment by the New Energy and Industrial Technology Development Organization (NEDO).

REFERENCES

1. SAE SAE TIR J2601 -Fueling Protocols for Light Duty Gaseiys Hydrogen Surfaace Vehicles, SAE Fuel Cell Standard Committee, 2010.
2. C.J.B. Dicken, and W. Mérida, Measured effects of filling time and initial mass on the tenperature distribution within a hydrogen cylinder during refueling, Journal of Power Sources, 165, 324-336(2007)
3. Y. Matsuno et al.: The Temperature Reached in the Tank During Gas Filling and Discharging Cycle for Compressed Hydrogen Tanks for Vehicles, Collection of papers of the Society of Automotive Engineers of Japan, Vol.42, No.3, p.773-776(2011)
4. Y. Matsuno et al.: Temperature Behavior in the Tank During Rapid Filling to Compressed Hydrogen Tanks for Vehicles (Report No.5) - Verification of the safety of rapid filling with precooled hydrogen gas, Autumn Lecture Meeting, Society of Automotive Engineers of Japan, pre-preinted collection No.111-12, p.1-4(2012)
5. ISO 17268 -Gaseous Hydrogen Land Vehicle Fueling Connectors, 2000.
6. SAE J2600 -Compressed Hydrogen Surface Vehicle Refuelling Connection Devices, SAE Fuel Cell Standard Committee, 2002.