Topics

Progress of Slow Strain Rate Tensile Testing Method under High Pressure Hydrogen Using Hollow Specimen Proposed to ISO/TC 164 (Mechanical Testing of Metals)

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In 2020, we proposed to ISO/TC 164 (Mechanical testing of metals) a test method for evaluating the strength and ductility of metallic materials in high-pressure hydrogen by filling the specimen with high-pressure hydrogen in a hollow space, which is simple and easy to use without using an expensive high-pressure hydrogen container, and by changing the test temperature as desired. After a vote in 2021, it was adopted and ISO/TC 164/SC 1/WG 9 (Hollow tensile testing in hydrogen) was created and the author is serving as convener. So far, the WG has held two WG meetings via web conference, prepared a draft CD, and had a CD ballot for TC 164/SC 1. A summary of the discussions and progress made by the working group will be presented.

1. Introduction (background)

To use hydrogen as an energy source, the challenges are to reduce the cost of production, transportation, and storage, and to improve the strength and ductility properties of materials that may deteriorate when they are subjected to loading in hydrogen environment. Conventional material testing methods for evaluating the effects of hydrogen on material properties in high-pressure hydrogen environments involve placing specimens in a high-pressure hydrogen container. Hence, the equipment was expensive and not easy to implement, and was only available in a limited number of testing laboratories, making it difficult to change the temperature of the specimens in the high-pressure vessel. One of the uses of hydrogen energy is fuel cell vehicles. A method of filling fuel cell vehicles with pre-cooled high-pressure hydrogen gas when filling the fuel tanks with hydrogen gas, and a method of rapidly vaporizing liquefied hydrogen to fill with high-pressure hydrogen were considered. However, a new evaluation method has been demanded because it is difficult to obtain material properties in a high-pressure hydrogen environment at low temperatures using conventional material testing methods.

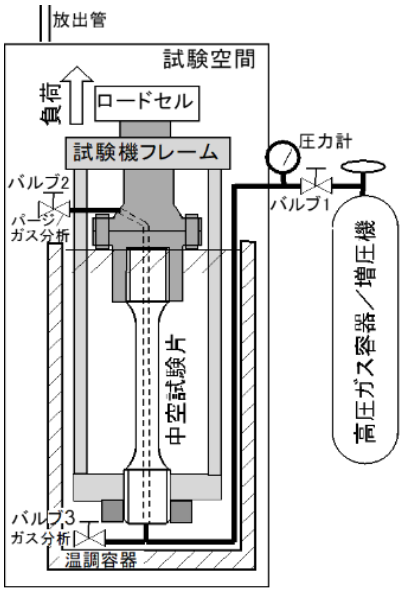

Fig 1. Basic diagram of high-pressure hydrogen environment test method using hollow specimen.

Therefore, we developed a hollow specimen method in which high-pressure hydrogen is filled inside the hollow of the specimen as shown in Figure 1[1]. The advantages of this test method are not only its low cost, since no high-pressure vessel is required, but also its ease of testing in high-pressure hydrogen environments from low to high temperatures, since no seal is needed between the high-pressure vessel and

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the load-loading rod. This method is expected to be implemented in many testing institutions because of the various advantages listed below.

(1) A pressure booster is not required up to the cylinder pressure (15 MPa), and a pressure gauge, valve, piping, fittings, and test space are all that are needed. (2) If the pressure is less than 1 MPa, you don't have to worry about the High Pressure Gas Safety Law, even less than 1 MPa is effective for screening materials. (3) If the volume in the specimen and in the enclosed piping is less than 1 cm^3, the amount of gas used is less than 1 liter at atmospheric pressure even at 100 MPa testing.

The data obtained by the hollow test method is used to obtain special approval from the High-Pressure Gas Safety Institute of Japan (KHK) for the components used in the manufactured equipment, and as the basis for the standards related to the General High Pressure Gas Safety Regulations. The fact that this method is also used to certify hydrogen-compatible materials, and that it has been implemented by private testing laboratories and overseas, has led to requests for standardization from companies wishing to start the hollow test method.

Table 1. Background of ISO proposal for Hollow Specimen Tensile Test Method.

Year/Month	Events
2017/08	A hollow tensile specimen method in high-pressure hydrogen was proposed to the Japanese Standards Association, the secretariat of ISO/TC 164 (Mechanical testing of metallic materials).
2020/10	The proposal was explained at the TC 164/SC 1 (uniaxial tests) meeting and a new proposal document (Form 04 and reference draft) was submitted to the SC 1 secretariat (France).
2021/02	Voting opens for new proposal NP7039
2021/05	Adopted after voting (for: 12 countries, against: 1 country, abstentions: 9 countries, elected experts: 5 countries (Japan, Germany, UK, Korea, China))
2021/09	ISO/TC 164/SC 1/WG 9 (Hollow Tensile Testing in Hydrogen) was launched. The duration is three years. Served as secretary with convener.
2021/12	The first meeting of ISO/TC 164/SC 1/WG 9 was held on the web, and the WD was revised and requested to comment again by the end of February 2022.
2022/09	The 2nd meeting of ISO/TC 164/SC 1/WG 9 was held via web and WD was revised again.
2022/09	At the TC 164/SC 1 meeting, the WD title change and proposed schedule were approved.
2022/11	The revised WD was submitted to SC 1 as a draft CD.
2022/12	Voting begins for CD 7039.

2. ISO proposal for hollow test method

2.1 Background to the proposal

ISO/TC 164 (Mechanical testing of metals) was selected as the submission site for this test method. Because we considered this test method to be a general material test method that allows not only high-pressure hydrogen to be filled in the hollow, but also various corrosive gases to be filled. TC 164 includes SC1 (uniaxial tests), SC2 (ductility tests), SC3 (hardness tests), and SC4 (toughness and fatigue tests), and we selected SC1, which is responsible for tensile and creep tests.

A summary of the history from the proposal to the establishment of the WG is shown in Table 1.

2.2 Discussion at ISO/TC 164/SC 1/WG 9

The main discussions in the first WG are as follows. Many of the opinions adhered to this test method as a screening test for hydrogen compatibility evaluation of materials.

(1) Since ASTM G142 and ISO 11114-4 are available for tensile testing in hydrogen, a new standard is not necessary.

(2) Evaluation of hydrogen compatibility by tensile properties is easily misleading. There is no standard that selects materials based on tensile properties, and fracture toughness K_{IH} is necessary to determine the limit of use considering crack propagation when the pressure vessel is used.

(3) If screening, it is valuable and should be clearly stated in the scope and text. This draft says test method, but implies a selection method.

(4) Since the selection of hydrogen-compatible materials is an urgent issue, it would be good if they could be used for screening.

(5) Agreement with the results of a conventional method on a solid specimen is not required.

(6) The easier and more flexible the better. Do not include vacuum (turbo) pumps as they are expensive.

These discussions led to the following agreements.

1. The WG members agreed to submit the draft CD for ISO/AWI 7039 to SC1 for balloting no later than June before.

2. It was agreed to distribute the revised WD of the clarified version by the end of 2021 and return comments by

the end of February 2022.

A huge number of comments were received by the end of February 2022, but it took a long time to respond to them because many of the revisions stuck to screening from the title. In the end, the term "in hydrogen" was omitted so that screening would not have to be used and could be used for the evaluation of gases other than hydrogen, since there is no precedent for the use of screening in the standards under the jurisdiction of TC164.

The main discussion in the second working group, in addition to the discussion of the text, was to revise the title to "Tensile testing — Method for evaluating changes of properties in a high-pressure gaseous environment using hollow test piece ". A CD vote was taken to make WD a CD.

3. Summary (Prospects)

After many comments and discussions in the working group, the ISO proposal, "Slow Strain Rate Tensile Test Method Using Hollow Specimen in High Pressure Hydrogen Environment," has become "Evaluation Method for Property Changes in High Pressure Gas Environment Using Hollow Specimen," which can be applied to other than hydrogen gas, and has advanced to CD balloting. However, it is anticipated that it will take time to receive a vast number of comments and to discuss the issue in order to reach a consensus, although there is the time limit of enacting the standard within three years from the establishment of the working group.

References

1) T. Ogata：Journal of the Japan Institute of Metals, **72**, 125-131 (2008).