



Tensile testing in high-pressure gaseous hydrogen using the hollow specimen method

Tomás Freitas,^{*} Florian Konert, Jonathan Nietzke, Zephanja Krzysch, Thomas Böllinghaus, Thorsten Michler, Ken Wackermann, Heiner Oesterlin, Mohamed Tlili, Peter Ruchti, Denise Beitelschmidt, Stephan Elsen-Humberg, Timo Koenigs, Thomas Systemans, and Oded Sobol

Received: 20 March 2024 / Accepted: 16 July 2024

Metallic materials, predominantly steels, are the most common structural materials in the various components along the hydrogen supply chain. Ensuring their sustainable and safe use in hydrogen technologies is a key factor in the ramp-up of the hydrogen economy. This requires extensive materials qualification, however, most of the accepted; and standardized test methods for determining the influence of gaseous hydrogen on metallic materials describe complex and costly procedures that are only available to a very limited extent worldwide. The hollow specimen technique is a simple, rapid, and economical method designed to overcome the limitations of the current methods for the qualification of metallic materials under high-pressure hydrogen gas. However, this technique is not yet standardized. The TransHyDE-H2Hohlzug project is presented in this article, along with the main steps required to optimize the hollow specimen technique. This includes closing knowledge gaps related to the specimen geometry, surface quality, and gas purity in dedicated working packages, thus contributing to a comprehensive standardization of the technique for tests in high-pressure hydrogen gas.

Introduction

The hydrogen storage and transport infrastructure must meet rigorous safety standards to ensure the safe and widely accepted use of hydrogen as an energy carrier. Metallic materials are commonly used as

structural materials for such infrastructure due to their favorable properties; however, they are also susceptible to hydrogen embrittlement (HE).¹ This phenomenon can lead to the degradation of certain mechanical

Tomás Freitas, Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany; tomas.grimault-freitas@bam.de
 Florian Konert, Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
 Jonathan Nietzke, Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
 Zephanja Krzysch, Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
 Thomas Böllinghaus, Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
 Thorsten Michler, Fraunhofer Institute for Mechanics of Materials IWM, Freiburg, Germany
 Ken Wackermann, Fraunhofer Institute for Mechanics of Materials IWM, Freiburg, Germany
 Heiner Oesterlin, Fraunhofer Institute for Mechanics of Materials IWM, Freiburg, Germany
 Mohamed Tlili, Fraunhofer Institute for Mechanics of Materials IWM, Freiburg, Germany
 Peter Ruchti, ZwickRoell Testing Systems GmbH, Fürstenfeld, Austria
 Denise Beitelschmidt, Netzsch-Gerätebau GmbH, Selb, Germany
 Stephan Elsen-Humberg, RWE Power AG, Essen, Germany
 Timo Koenigs, RWE Power AG, Essen, Germany
 Thomas Systemans, Deutscher Verein des Gas- und Wasserfaches e.V. (DVGW), Bonn, Germany
 Oded Sobol, Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany

^{*}Corresponding author

doi:10.1557/s43577-024-00776-9

Impact statement

The hydrogen economy is considered a key solution for achieving climate neutrality in Europe, as it plays a crucial role in the decarbonization of sectors such as transport, industry, power, etc. Ensuring the safety and reliability of infrastructure is crucial for the ramp-up of the hydrogen economy. Therefore, it is necessary to meticulously study the materials and components used for infrastructure under conditions that closely resemble in-service conditions. The currently standardized methods are limited as they do not precisely replicate in-service conditions, and when they do, they are often complex, costly, and not easily accessible. This article presents the hollow specimen technique, a simple, and economical method developed to address the limitations of current standardized methods. The results from this work will contribute to the standardization of this technique for tests in high-pressure hydrogen gas. This will enable a faster evaluation of materials for hydrogen applications by industry and academia, thereby contributing to the growth of the hydrogen economy.

properties of the materials, and, in some cases, to premature and even catastrophic failure.

Selecting materials for new components and parts for hydrogen applications must consider HE so that premature failure is avoided during their service life. For components that are already in use, ensuring their feasibility and safety in hydrogen applications is vital as it is seen as a key factor in scaling up the hydrogen market. Europe's existing natural gas infrastructure is a good example as it is expected to be repurposed for hydrogen.² The indispensable basis for such feasibility and safety assessments is the material properties, which must be measured as close as possible to real operating conditions. However, the material testing methods required for such qualification are not sufficiently standardized today. The most common methods available for qualifying materials for hydrogen applications are seen in **Figure 1**.

The *in situ* electrochemical test method (Figure 1a) is based on an electrochemical cell in which the specimen serves as the cathode and a counter-electrode acts as the anode, both immersed in an electrolyte. Stress is applied to the specimen, while hydrogen is cathodically generated on the surface.

The *ex situ* precharging test methods (Figure 1b–c) consist of performing mechanical tests on specimens that have been previously charged with hydrogen. Depending on the source of hydrogen, the precharging method can be classified as electrochemical (Figure 1b) or thermal (Figure 1c) precharging.³

Ex situ and *in situ* electrochemical charging methods (Figure 1a–b), although simple and affordable, have the disadvantage of causing high fugacity on the material's surface. This can result in rapid hydrogen concentration gradients, which can induce blistering, surface cracking, and phase transformations.^{4–7} In addition, electrochemical charging is generally limited to temperatures below 100°C as most electrolytes used

are water-based. In materials with low hydrogen diffusivity (e.g., austenitic stainless steels [ASSs]), such temperatures may not allow for a rapid and uniform distribution of hydrogen throughout the specimen. Thermal precharging (Figure 1c), on the other hand, can be performed at higher temperatures and varying pressure, which increases the diffusion rate. This can allow for a homogeneous distribution of hydrogen in the material within days or weeks.³ However, this technique may not be suitable for materials with high hydrogen diffusion coefficients, such as ferritic steels. This is because a significant amount of the absorbed hydrogen could effuse out of the specimen after charging, and before or during the mechanical test. This can lead to inaccurate results, especially when performing fatigue tests or tests at slow strain rates.

In the *in situ* autoclave technique (Figure 1d), a standardized solid specimen is typically placed into a pressure vessel (autoclave), together with the measuring equipment. This technique allows a more representative simultaneous interaction between the stress/strain evolution inherent to the particular mechanical test, the activation of the material characteristic deformation mechanism(s) and hydrogen uptake. In addition, a variety of common standardized tests such as tensile,^{8–11} crack growth,¹² and fatigue^{13,14} can be performed. At the pressures required to simulate real operating conditions of hydrogen technologies, the use of the autoclave technique poses a substantial danger due to the high volume of hydrogen involved. As such, this testing method demands extensive safety measures. This leads to high costs, which limits the availability of testing equipment and providers of the qualification tests.

A simple and cost-effective alternative to the autoclave technique and the focus of this work is the use of the hollow specimen technique (**Figure 2**). In this method, an axial hole is drilled in a tensile specimen, which is then filled with

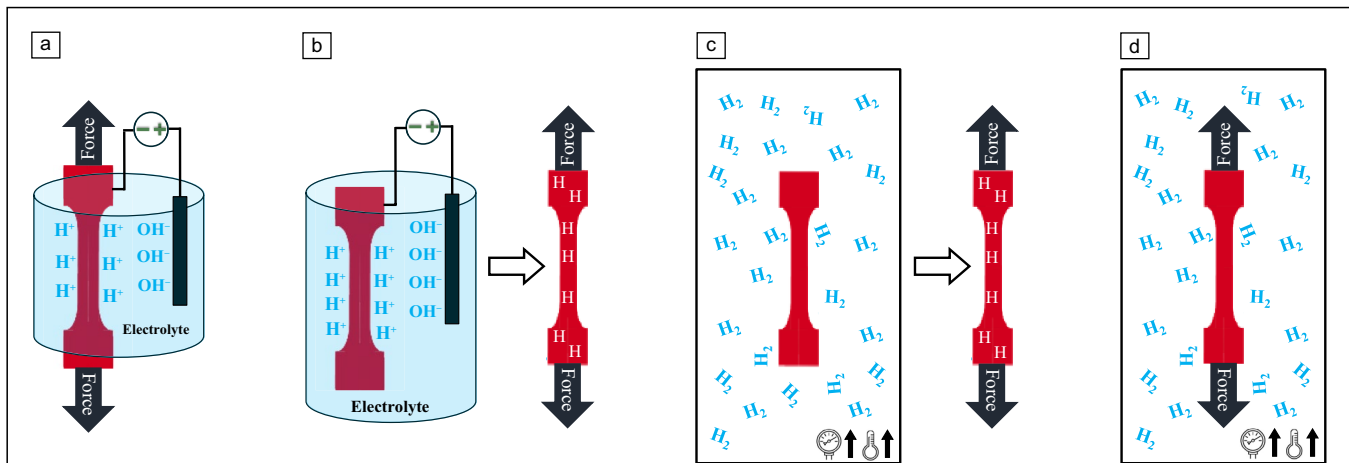


Figure 1. Simple representation of the most common methods for mechanical tests of hydrogen charged specimens: (a) *In situ* electrochemical charging, (b) *ex situ* electrochemical precharging; (c) *ex situ* thermal precharging; (d) *in situ* conventional autoclave technique (the symbols indicate the flexibility in testing conditions).

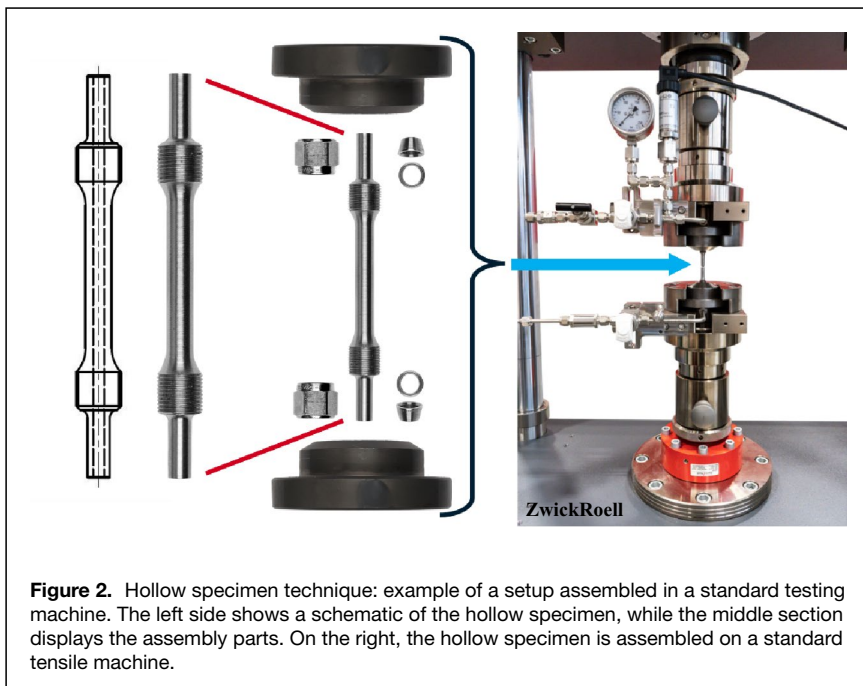


Figure 2. Hollow specimen technique: example of a setup assembled in a standard testing machine. The left side shows a schematic of the hollow specimen, while the middle section displays the assembly parts. On the right, the hollow specimen is assembled on a standard tensile machine.

hydrogen gas, sealed, and placed in a standard testing machine for testing. This method is explored with more detail in the next section.

The H2HohlZug project is presented in this article. It is part of the TransHyDE flagship project,¹⁵ which focuses on the assessment, development, and demonstration of several technologies for the transport and storage of hydrogen. The Bundesanstalt für Materialforschung und prüfung (BAM) coordinates the H2HohlZug project in collaboration with the Fraunhofer Institute for Mechanics of Materials (IWM), RWE Power AG, ZwickRoell SE, and the Deutscher Verein des Gas- und Wasserfaches e.V. (DVGW). The aim of the project is to validate the hollow specimen technique for quasi-static tensile testing to determine material properties under gaseous hydrogen, thus providing academia and industry with a rapid and cost-effective testing method.

The following sections present the state of the art regarding the hollow specimen technique, the various steps in the validation of the technique for the qualification of metallic materials, together with initial results of the project.

State of the art of the hollow specimen technique

Material properties obtained from quasi-static tensile tests form the basis of any component design. In hydrogen atmospheres, these properties are obtained almost exclusively with solid tensile specimens.^{7–10} The solid tensile specimens are used in the conventional autoclave technique and the results allow one to assess the influence of high-pressure hydrogen gas environments on the mechanical properties of the tested materials, as described in ASTM G129¹⁰ or ASTM G142.¹¹

From a chronological point of view, hollow specimens have only rarely been used to evaluate high-pressure hydrogen gas environments. The first works date back to the 1950s,¹⁶ in which the deformation behavior was not comparable to that of solid specimens due to the geometry adopted for this type of specimen. This meant that mechanical properties commonly used to assess the effect of hydrogen, such as elongation at break and reduction of area (RA), could not be accurately obtained.

In recent years, policies promoting the transition to renewable energy sources, such as hydrogen, have been adopted. An example of such a policy is the Japanese technical standard (General High-Pressure Gas Safety Ordinance) for hydrogen refuelling stations, responsible for supplying high-purity hydrogen to fuel cell

vehicles equipped with a 35 MPa hydrogen container, that was issued in 2005.^{17,18} In 2010, the technical standard was updated, and the maximum storage pressure of the onboard containers was increased to 70 MPa.^{19,20} These policies highlighted the need for experts to develop new, reliable, and economical techniques to assess the effect of hydrogen gas on materials.

The early 2000s saw a significant development of the hollow specimen technique.^{21–28} This simple test method can be used to evaluate the mechanical properties of materials under high-pressure hydrogen gas. This is done by enclosing high-pressure gas into a mechanically drilled hole along the axis of a round tensile test specimen and then placing the specimen in a standard testing machine (Figure 2) for testing. Such a method entails higher specimen production costs than conventional round tensile specimens used in the autoclave technique due to the inner axial hole machined through the specimen's length. However, the reduced risks associated with the lower volume of hydrogen used during the test (by up to some orders of magnitude), the fact that it can be carried out in a standard laboratory and that various pressure–temperature combinations are relatively easy to replicate, result in a significantly lower overall cost and more accessible technique when compared to the autoclave technique. A proof of concept of this test method was successful not only in tensile tests,^{21–27} but also in fatigue tests,^{23,24,26,28} for ferritic,^{26,27} martensitic,^{21,23,26} and austenitic stainless steels,^{22–28} and nickel-based alloys.^{23,26} The effect of temperature^{21–28} over the (20–373) K range was also successfully assessed. Similar to conventional 300 series of ASS specimens, a minimum tensile ductility was



found at around 200 K.^{23,25,26} The influence of pressure also follows the general trend seen with conventional solid specimens, where tests in higher pressure environments yielded more severe effects of hydrogen.^{22,26} In an initial attempt to standardize the method, the influence of the inner hole and inner surface roughness on relative reduction of area (RA) and other properties in slow strain rate tests (SSRTs) was investigated.^{22,26,27} A smaller hole diameter was found to be preferable as the accuracy of the results compared to conventional solid specimens decreased with increasing hole diameter. Tensile and yield strength were not affected by the hole nor the inner pressure, whereas RA decreased slightly in the hollow specimen in a reference atmosphere. Surfaces with lower inner hole roughness, obtained by axially polishing the specimens, were proposed as the standard.²⁷

The hollow specimen technique has also been evaluated in recent years.^{29–32} A tensile test setup featuring hollow specimens loaded internally with pressurized gas was developed and tested with an X60 pipeline steel.²⁹ The study concluded that the setup is capable of being used for the characterization of the HE susceptibility of specific regions of a material. This setup was later modified to be compatible with *in situ* fatigue testing and was also found to be successful in investigating the effect of hydrogen on fatigue crack initiation and growth in X60 steel and its girth weld.³⁰ The hollow specimen technique has been successfully used to investigate the effect of high-pressure hydrogen on the mechanical properties of materials. However, the number of publications using this design is still limited. A comparison of the results obtained with this novel technique with those obtained using a well-established benchmark method is necessary to gain wider acceptance. In this case, the established benchmark method is the autoclave technique using conventional specimens. For this reason, tensile properties obtained in high-pressure gaseous hydrogen using conventional solid specimens (used in the autoclave technique) and hollow specimens of X60 pipeline steel³¹ and ASS³² were compared. Both yield and tensile strength were comparable between the specimen geometries. However, the RA showed differences between conventional and hollow specimens. When tested in a reference atmosphere, the RA of conventional solid specimens was higher than that of hollow specimens. In gaseous high-pressure hydrogen, the RA of conventional solid specimens was lower than that of hollow specimens.^{31,32}

The initial studies have shown that it is, in principle, possible to assess hydrogen effects in metallic materials using the hollow specimen technique. The technique is expected to be used worldwide as the preferred technique for evaluating the tensile and fatigue properties of structural materials for severe environments. However, further studies are still required to address the open questions that have been raised during the initial studies.

The high potential of the hollow specimen technique has led to an ISO standardization process for this method, initiated by Japan in August 2021. The substantive work is being done by a dedicated working group in ISO TC 164/SC 1 (Uniaxial

Testing), in which BAM and Fraunhofer IWM are strongly involved. Several influencing factors such as specimen geometry, surface quality, or gas purity prevent the technique from being standardized for hydrogen testing. These are addressed in the H2HohlZug project, in which the main objective is to systematically fill the knowledge gaps and contribute to a standard for the hollow specimen technique for hydrogen testing.

In the following section, four key steps are discussed and supported by preliminary results: (1) optimization of the specimen geometry, (2) influence of the hole surface quality, (3) influence of the gas purity, and (4) reproducibility and repeatability of the findings (round-robin tests).

Methodology and discussion

Two representative steels widely used in hydrogen technologies have been selected, namely X5CrNi18-10 (AISI type 304), a corrosion-resistant ASS used for hydrogen infrastructure components,³³ and 42CrMo4 (AISI type 4140), a medium carbon Cr–Mo alloy steel used for hydrogen transport in high-pressure vessels.³⁴ The H2HohlZug project has been divided into four different working packages to address the current research questions related to testing in hydrogen with the hollow specimen technique:

Simulation and validation of the mechanical behavior of hollow specimens compared to conventional solid specimens in SSRT

The elastic–plastic behavior of a hollow tensile specimen is compared to that of a conventional solid tensile specimen with identical external dimensions using finite element methods (3D-FEM) and validated afterwards by experimental results.

Initial experimental and numerical investigations at the Fraunhofer IWM have shown that the stress–strain behavior of a conventional solid tensile specimen ($L_0=30$ mm, $\varnothing 6$ mm) and that of a hollow tensile specimen with identical outer dimensions and an internal hole diameter of 2 mm is identical up to the maximum stress. The differences in mechanical behavior occur mostly after the tensile strength is exceeded and thus have a strong effect on the necking at fracture.

For the evaluation of material degradation by hydrogen, it is precisely the evaluation of the necking at fracture that has proven to be particularly useful, since this parameter is the one most strongly influenced by hydrogen.³⁵ In a reference medium (i.e., without the influence of hydrogen), ductile metallic materials show a maximum stress in the tensile test, the so-called tensile strength. After reaching the tensile strength, the specimen begins to neck (i.e., the specimen cross section is locally reduced). Pores form inside the material, which coalesce and lead to fracture of the specimen once a critical stress is exceeded. This mechanism is influenced by the insertion of an axial hole (**Figure 3**). This is supported by initial experimental results, where slightly lower necking at fracture tended to be measured for a hollow specimen than for conventional solid specimens (in a reference medium).^{27,31,32}

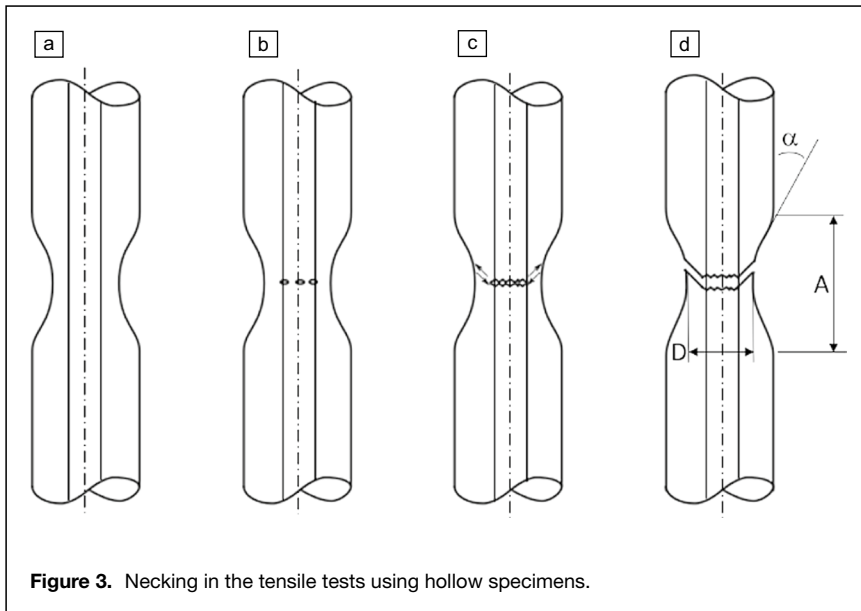


Figure 3. Necking in the tensile tests using hollow specimens.

To better understand this difference, the necking and fracture behavior of a solid and a hollow specimen are simulated using 3D FEM (Abaqus) and validated by experiments. It is planned to proceed with digital image correlation (DIC) of tensile tests on notched specimens to obtain the specific material parameters of the elastic–plastic deformation model. In this way, high-quality simulation results are obtained. The model is then used to simulate tensile tests on specimens with varying geometry (e.g., inner and outer diameter). Finally, the simulation results obtained are compared to the results of tensile tests on specifically manufactured specimens with the same varying geometry.

Another difference between solid and hollow specimens is the application of the gas pressure. While in a conventional solid specimen, the gas pressure is applied externally, in a hollow specimen, the gas pressure is applied at the inner hole. This difference must be taken into account, as the wall should not be mechanically influenced by the internal pressure significantly (i.e., buckling of the specimen due to thin walls), but, at the same time should be feasible in standard testing machines. The two representative steels previously mentioned are used to investigate how the application of the gas pressure affects the necking behavior. Experimental validation is performed with tensile tests in an inert atmosphere.

The investigations described in this section include the analysis of the deformation behavior of solid and hollow specimens in a reference medium (i.e., without hydrogen influence). The aim of these investigations is to approximate the elastic–plastic deformation behavior of the hollow specimen to that of the conventional solid specimen, and thus obtain an optimized specimen geometry. The influence of hydrogen is discussed in the following chapters (Sections “[Influence of different manufacturing and finishing processes on the inner hole surface of the hollow specimen and its response to hydrogen](#) effects” and “[Influence of gas purity and purging process](#)”). This influence should be only minimally dependent on the selected specimen geometry.

effects” and “[Influence of gas purity and purging process](#)”). This influence should be only minimally dependent on the selected specimen geometry.

Influence of different manufacturing and finishing processes on the inner hole surface of the hollow specimen and its response to hydrogen effects

Depending on the production method, different roughness and residual stresses can be expected on the inner hole surface of the specimens. Surface condition is a key engineering variable in the absorption of hydrogen into a metal, as the presence of surface defects can affect hydrogen uptake. In principle, electrons around a peak escape more easily than those in a valley,

so greater surface roughness increases the local fluctuation of the electron work function (minimum energy required to completely remove an electron from a metal surface), leading to accelerated corrosion on a rough surface.³⁶ Similarly, an increase in stress concentration sites, such as notches, has been found to be associated to a higher severity of hydrogen effects.^{37–39} The presence of strain-induced martensite caused by residual stresses could also be detrimental and accelerate the embrittlement process of metastable ASS such as AISI type 304.⁴⁰ To assess this, the hole is manufactured by conventional drilling or electrical discharge machining (EDM) and the finishing is done by either reaming or honing. The objective is to examine which surface properties (roughness, residual stresses) are produced by the various manufacturing and finishing methods and how they affect the tensile test result under a hydrogen atmosphere. The following surface conditions are investigated: (a) drilled only; (b) drilled and reamed; (c) drilled and honed; (d) EDM and honed; (e) EDM and reamed. The expected average roughness, R_a , obtained from each process is shown in **Table I**.

The five different methods of producing the inner axial hole were chosen to ensure different average roughness values and residual stresses (drilling versus EDM), and also to ensure a good representation of the more common and easily accessible processes (drilling and reaming) to more complex ones (EDM and honing). Moreover, as this work includes a metastable austenitic grade, the minimization of the effect of residual stresses and phase transformation (γ -austenite \rightarrow α' -martensite) due to the mechanical processing is evaluated by having the inner axial hole produced by drilling and EDM.⁴²

A comparison of the tensile properties obtained in a reference medium and in hydrogen gas with hollow specimens produced using different inner axial hole manufacturing processes was carried out at BAM (**Figure 4**). The reference



Table I. Roughness average (Ra) in μm based on ANSI/ASME B46.1.⁴¹

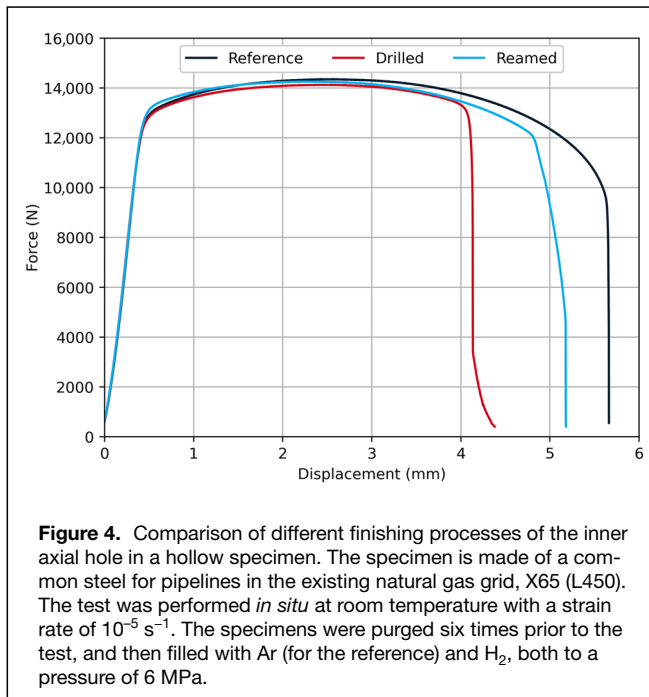
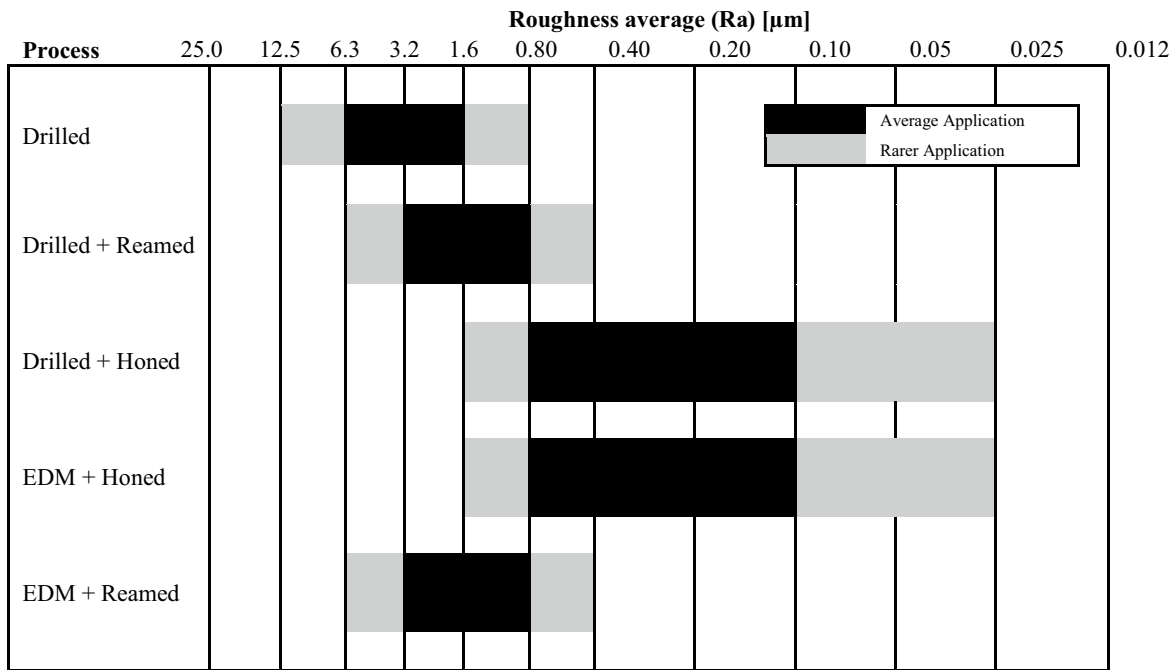


Figure 4. Comparison of different finishing processes of the inner axial hole in a hollow specimen. The specimen is made of a common steel for pipelines in the existing natural gas grid, X65 (L450). The test was performed *in situ* at room temperature with a strain rate of 10^{-5} s^{-1} . The specimens were purged six times prior to the test, and then filled with Ar (for the reference) and H_2 , both to a pressure of 6 MPa.

specimens were tested in an inert atmosphere (argon), which, in principle, should not result in differences in the assessed mechanical properties if different manufacturing processes are used. Preliminary results at BAM support this theory, and are expected to be published at a later stage of this project. In Figure 4, the reference specimen (black curve) was manufactured by conventional drilling, and later

tested in argon. The specimens tested in 6 MPa H_2 were manufactured by conventional drilling only (red curve), and conventional drilling followed by a reaming finish (blue curve). It can be observed that the reamed specimen (blue curve) showed higher tensile ductility when compared to the drilled-only specimen (red curve). The preliminary results give an insight into the possible influence of the surface quality on the results.

The influence of the different manufacturing and finishing processes is investigated by means of tensile tests at room temperature under reference and hydrogen atmospheres. A sufficient number of specimens are tested per variant to obtain reliable statistical significance. The characterization of the surface and the demonstration of the influence of different preparation methods will be carried out using a profilometer, light optical microscopy, and scanning electron microscopy (SEM). **Figure 5** shows an example of the cross section of a fractured specimen analyzed by SEM. The specimen (X5CrNi18-10, AISI 304 grade) was tested *in situ* at room temperature in 10 MPa gaseous hydrogen using the hollow specimen technique.

If relevant, other characterization methods will be considered (e.g., electron backscatter diffraction [EBSD]) to characterize the microstructure or x-ray microtomography ($\mu\text{-CT}$) to assess the fracture behavior of the specimens.⁴³

Influence of gas purity and purging process

The gas purity is another topic of interest as it can affect the measured value of the material properties to be determined. Several investigations regarding the effects of common impurities in natural gas pipelines can be found in the

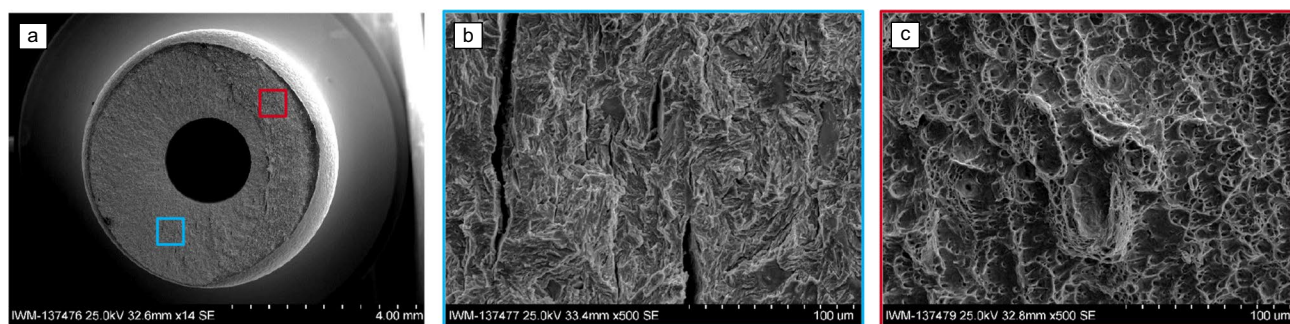


Figure 5. Fractography of a X5CrNi18-10 (AISI 304) grade hollow specimen tested *in situ* at room temperature in 10 MPa gaseous hydrogen. (a) Overview of the fracture surface showcasing areas of interest in blue and red; (b) blue region at higher magnification showing hydrogen-assisted fracture features and (c) red region at higher magnification showing ductile fracture features.

literature. In particular, small amounts of oxygen in hydrogen have been found to significantly reduce the damaging effect of hydrogen.^{31,44–47} Contrary to oxygen molecules, sulfur-containing molecules are considered to promote hydrogen embrittlement.⁴⁸ Specifications for gas purity can be found in standards for materials characterization under pressurized hydrogen.⁴⁹ In hollow specimens, gas purity is particularly important since the ratio of specimen inner surface area to gas volume is greater than in solid specimens tested in an autoclave. Therefore, smaller absolute amounts of impurities are expected to have a greater impact on the measurement result.

In this project, the gas composition and the various parameters of the purging process that determine the gas purity during the experiment are varied. The different gas compositions include, for example, H₂ 5.0 (≥99.999% hydrogen), H₂ 6.0 (≥99.9999% hydrogen), H₂ + CO_x or H₂ + C_xH_y, and the parameters of the purging process include the number of purging cycles, the pressure difference of the purging cycle (e.g., 15 MPa → 1 MPa), and the application of a prevacuum.

Measuring the chemical composition of the gas in a hollow specimen is complicated due to its limited volume. Therefore, the effect of the gas purity is evaluated by tensile tests. The aim is to define the most suitable gas quality, a purging process that provides repeatable and reproducible results, and to determine a maximum permissible storage time for the subsequent test.

Reproducibility and repeatability of the technique (round-robin tests)

The knowledge acquired in sections “Simulation and validation of the mechanical behavior of hollow specimens compared to conventional solid specimens in SSRT,” “Influence of different manufacturing and finishing processes on the inner hole surface of the hollow specimen and its response to hydrogen effects,” and “Influence of gas purity and purging process” is validated by the means of a round-robin. The

project partners, along with European and international partners will be involved in order to validate the applicability of the optimized specimen geometry, the surface properties of the inner hole, and the influence of gas purity and purging cycles on the test results.

The round-robin tests are conducted in two stages. The first stage aims to assess the repeatability of results obtained using the hollow specimen technique. This involves centrally producing a sufficient number of hollow specimens from the selected materials, according to the specifications outlined in sections “Simulation and validation of the mechanical behavior of hollow specimens compared to conventional solid specimens in SSRT” and “Influence of different manufacturing and finishing processes on the inner hole surface of the hollow specimen and its response to hydrogen effects.” These specimens are then shipped to the test laboratories, where they will be tested under the conditions defined in the section “Influence of gas purity and purging process.” The second stage involves each partner producing their own hollow specimens according to the specifications outlined in sections “Simulation and validation of the mechanical behavior of hollow specimens compared to conventional solid specimens in SSRT” and “Influence of different manufacturing and finishing processes on the inner hole surface of the hollow specimen and its response to hydrogen effects.” These specimens are then tested according to the conditions defined in the Section “Influence of gas purity and purging process.” The aim of the second round-robin is to determine whether the hollow specimen technique can be replicated in full. The results obtained in the round-robin tests will be presented for discussion in the standardization committee for the hollow specimen technique, and in the national mirror committees.

Conclusions and perspectives

The main steps needed to optimize the hollow specimen method and support its standardization for tests in a hydrogen atmosphere are described in detail in this article. This



technique is intended to be applied to evaluate the influence of high-pressure gaseous hydrogen on metallic materials and thus to support the decision-making process of the European hydrogen market ramp-up.

The discussion presented in this work shows how the mentioned factors (specimen geometry, inner surface quality, and gas purity) can influence the results obtained in materials of interest to the hydrogen industry. The geometry of a hollow specimen requires modeling and experimental validation of different inner/outer diameter ratios. On the one hand, the specimen's gauge length must not be significantly deformed by the inner pressure, and, on the other hand, special testing equipment should not be required to perform the tensile tests. Furthermore, as hydrogen absorption is strongly dependent on the surface roughness, it is mandatory to assess the influence of different machining processes. The latter must not only be commercially available, but also be sufficient to provide a suitably fine surface without critical notches (potential stress concentration sites). Finally, the impact of impurities in the inner atmosphere must be assessed. Impurities can stem from the atmosphere prior to submitting the specimen to the intended test atmosphere or can be intentionally added to test in-service conditions.

Overall, along with the potential of the hollow specimen technique for tensile and fatigue tests at ambient conditions, one must consider the potential of applying this test method under different conditions. Considering the reduced safety factors, this method shows promise in high-temperature applications, such as fuel-cell technologies, and in the qualification of materials for the transport of ammonia.

Author contributions

T.F.—Investigation: Lead; Methodology: Lead; Writing—original draft: Lead; Writing—review & editing: Lead. F.K.—Conceptualization: Equal; Data curation: Supporting; Formal analysis: Supporting; Methodology: Lead; Validation: Supporting; Writing—original draft: Supporting; Writing—review & editing: Supporting. J.N.—Conceptualization: Supporting; Formal analysis: Supporting; Investigation: Supporting; Methodology: Equal; Project administration: Supporting; Validation: Equal; Writing—original draft: Supporting; Writing—review & editing: Supporting. Z.K.—Formal analysis: Equal; Investigation: Equal; Validation: Supporting. T.B.—Conceptualization: Equal; Funding acquisition: Lead; Supervision: Supporting. T.M.—Conceptualization: Lead; Funding acquisition: Lead; Investigation: Lead; Methodology: Lead; Project administration: Lead; Supervision: Lead; Validation: Lead; Writing—original draft: Supporting; Writing—review & editing: Supporting. K.W.—Funding acquisition: Lead; Investigation: Lead; Methodology: Lead; Project administration: Lead; Supervision: Lead; Validation: Lead; Writing—original draft: Supporting; Writing—review & editing: Supporting. H.O.—Funding acquisition: Lead; Investigation: Lead; Methodology: Lead; Project administration: Lead; Supervision: Lead; Validation: Lead; Writing—original draft: Supporting; Writing—review & editing: Supporting. M.T.—Formal analysis: Equal; Investigation:

Equal; Validation: Equal; Visualization: Equal. P.R.—Conceptualization: Supporting; Funding acquisition: Equal; Investigation: Supporting; Methodology: Supporting; Project administration: Supporting. D.B.—Conceptualization: Equal; Funding acquisition: Equal; Investigation: Supporting; Methodology: Equal; Project administration: Supporting; Validation: Equal. S.E.—Conceptualization: Supporting; Funding acquisition: Supporting; Investigation: Supporting; Methodology: Supporting; Project administration: Supporting; Validation: Supporting. T.K.—Conceptualization: Supporting; Investigation: Supporting; Methodology: Supporting; Validation: Supporting. T.S.—Conceptualization: Equal; Funding acquisition: Equal; Project administration: Lead; Resources: Equal. O.S.—Conceptualization: Lead; Funding acquisition: Lead; Investigation: Supporting; Methodology: Supporting; Project administration: Lead; Resources: Supporting; Supervision: Lead; Validation: Supporting; Visualization: Supporting; Writing—original draft: Supporting; Writing—review & editing: Supporting.

Funding

Open Access funding enabled and organized by Projekt DEAL. The authors acknowledge funding from the Bundesministerium für Bildung und Forschung, Germany (Award No. 03HY205).

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflict of interest

The authors declare no conflict of interest.

Open Access

This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit <http://creativecommons.org/licenses/by/4.0/>.

References

1. I.M. Robertson, P. Sofronis, A. Nagao, M.L. Martin, S. Wang, D.W. Gross, K.E. Nygren, *Metall. Mater. Trans. B* **46**, 1085 (2015)
2. M.J. Chae, J.H. Kim, B. Moon, S. Park, Y.S. Lee, *Korean J. Chem. Eng.* **39**, 251 (2022)
3. C. San Marchi, "Hydrogen Embrittlement of Stainless Steels and Their Welds," in *Gaseous Hydrogen Embrittlement of Materials in Energy Technologies*, ed. by R.P. Ganglosff, B.P. Somerday (Woodhead Publishing, Sawston, 2012)



4. M.C. Tiegel, M.L. Martin, A.K. Lehmborg, M. Deutges, C. Borchers, R. Kirchheim, *Acta Mater.* **115**, 24 (2012)
5. P. Rozenak, D. Eliezer, *Metall. Trans. A* **19**, 723 (1988)
6. E. Minkovitz, D. Eliezer, *J. Mater. Sci. Lett.* **1**, 192 (1982)
7. E. Dabah, V. Lisitsyn, D. Eliezer, *Mater. Sci. Eng. A* **527**, 4851 (2010)
8. ASTM E8/E8M, Standard test methods for tension testing of metallic materials, 2022
9. DIN EN ISO 6892, Metallic materials—Tensile testing, 2019
10. ASTM G 129, Standard practice for slow strain rate testing to evaluate the susceptibility of metallic materials to environmentally assisted cracking, 2021
11. ASTM G 142–98, Standard test method for determination of susceptibility of metals to embrittlement in hydrogen containing environments at high-pressure, high temperature, or both, 2022
12. ISO 11114-4:2017, Transportable gas cylinders—Compatibility of cylinder and valve materials with gas contents—Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement, 2017
13. ASTM E 466, Standard practice for conducting force controlled constant amplitude axial fatigue tests of metallic materials, 2021
14. ASTM E 606, Standard test method for strain-controlled fatigue testing, 2021
15. BMBF, How the TransHyDE flagship project aims to develop a hydrogen transport infrastructure. <https://www.wasserstoff-leitprojekte.de/projects/transhyde>, Accessed June 2023
16. W.T. Chandler, R.J. Walter, *ASTM STP* **543**, 170 (1974)
17. The High Pressure Gas Safety Institute of Japan, General high-pressure gas safety ordinance, Article 7–3, Japan, 2005
18. JARIS001, Technical standard for containers of compressed-hydrogen vehicle fuel devices, Japan, 2004
19. The High Pressure Gas Safety Institute of Japan, General high-pressure gas safety ordinance, Revision to 82 MPa, Article 7–3, Japan, 2010
20. KHK S0128, Technological standard for compressed hydrogen containers for vehicle fuel device with maximum fuelling pressure of 70MPa, Japan, 2010
21. T. Ogata, "Influence of 70 MPa Hydrogen Gas on SUS630 from 77 K to 373 K by Simple Testing Method," in *Proceedings of the ASME 2018 Pressure Vessels and Piping Conference*, Vol. 6B (Materials and Fabrication), (ASME, Prague, Czech Republic, 2018)
22. T. Ogata, *J. Jpn. Inst. Metal. Mater.* **72**(2), 125 (2008)
23. T. Ogata, *AIP Conf. Proc.* **1435**, (2012), pp. 39–46
24. T. Ogata, *IOP Conf. Ser.: Mater. Sci. Eng.* **102**, 12005 (2015)
25. T. Ogata, *AIP Conf. Proc.* **986**, (2008), pp. 25–32
26. T. Ogata, "Simple Mechanical Testing Method to Evaluate Influence of High Pressure Hydrogen Gas", in *Proceedings of the ASME 2018 Pressure Vessels and Piping Conference*, Vol. 6B (Materials and Fabrication) (ASME, Prague, Czech Republic, 2018)
27. T. Ogata, Y. Ono, Influence of roughness of inner surface of simple mechanical testing method to evaluate influence of high pressure hydrogen gas, in *Proceedings of the ASME 2019 Pressure Vessels and Piping Conference*, Vol. 6B (Materials and Fabrication) (ASME, San Antonio, 2019)
28. T. Ogata, *AIP Conf. Proc.* **1219**, (2010), pp. 25–32
29. T. Boot, T. Riemslag, E. Reinton, P. Liu, C.L. Walters, V. Popovich, *Metals* **11**(8), 1242 (2021)
30. L.E. Faucon, T. Boot, T. Riemslag, S.P. Scott, P. Liu, V. Popovich, *Metals* **13**(3), 563 (2023)
31. T. Michler, F. Ebling, H. Oesterlin, C. Fischer, K. Wackermann, *Int. J. Hydrogen Energy* **47**, 34676 (2022)
32. T. Michler, T. Freitas, H. Oesterlin, C. Fischer, K. Wackermann, F. Ebling, *Int. J. Hydrogen Energy* **48**(65), 25609 (2023)
33. B.P. Somerday, C. San Marchi, "Hydrogen Containment Materials", in *Woodhead Publishing Series in Electronic and Optical Materials, Solid-State Hydrogen Storage*, ed. by G. Walker (Woodhead Publishing, Sawston, 2008), pp. 51–81
34. A. Zafra, L.B. Peral, J. Belzunce, C. Rodriguez, *Int. J. Press. Vessels Pip.* **171**, 34 (2019)
35. G.R. Caskey Jr., *Hydrogen Compatibility Handbook for Stainless Steels*, (University of Texas North Libraries, 1983)
36. W. Li, D.Y. Li, *Acta Mater.* **54**(2), 445 (2006)
37. R.J. Walter, W.T. Chandler, *Mater. Sci. Eng.* **8**(2), 90 (1971)
38. D. Hardie, S. Liu, *Corros. Sci.* **38**(5), 721 (1996)
39. M. Wang, E. Akiyama, K. Tsuzaki, *Mater. Sci. Eng. A* **398**, 37 (2005)
40. M. Martin, S. Weber, C. Izawa, S. Wagner, A. Pundt, W. Theisen, *Int. J. Hydrogen Energy* **36**(17), 11195 (2011)
41. ASME B46.1-2019, Surface texture (roughness, waviness, lay), 2020
42. A. Roehsler, Hydrogen Effects in Austenitic Stainless Steel Microstructures Validated by ToF-SIMS and EBSD, PhD thesis, Otto-von-Guericke-Universität Magdeburg, (2021)
43. F. Konert, F. Wieder, J. Nietzsche, D. Meinel, T. Böllinghaus, O. Sobol, *Int. J. Hydrogen Energy* **59**, 874 (2024)
44. B.P. Somerday, P. Sofronis, K.A. Nibur, C. San-Marchi, R. Kirchheim, *Acta Mater.* **61**, 6153 (2013)
45. H.J. Cialone, P.M. Scott, J.H. Holbrook, K. Sieradzki, N. Bandyopadhyay, *Adv. Hydrogen Energy* **4**, 1855 (1984)
46. R. Komoda, M. Kubota, A. Staykov, P. Ginet, F. Barbier, J. Furtado, *Fatigue Fract. Eng. Mater. Struct.* **42**, 1387 (2019)
47. S.W. Ooi, P. Yan, R.H. Vegter, *Mater. Sci. Technol.* **35**, 12 (2019)
48. H. Fujii, T. Fujishiro, M. Sagara, Y. Masaki, T. Hara, *ISIJ Int.* **60**, 739 (2020)
49. ANSI/CSA CHMC1–2014, Test methods for evaluating material compatibility in compressed hydrogen applications—Metals, 2014 □

Publisher's note

Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.