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Evaluation of the tensile properties of X65 pipeline steel in compressed gaseous hydrogen using hollow specimens

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Abstract

Hydrogen has great potential on the path towards decarbonization of the energy and transport sectors and can mitigate the urgent issue of global warming. It can be sustainably produced through water electrolysis with potentially zero emissions, and efficiently used (e.g., in fuel cell systems). Despite its environmental advantages, hydrogen-metal interactions could result in the degradation of the mechanical properties of several structural materials. In order to determine the magnitude of the material degradation in relation to hydrogen exposure, extensive material testing is required. The standardized procedure for in-situ testing for the quantification of the impact of compressed gaseous hydrogen (CGH_2) relies on the utilization of an autoclave around the tested specimen. Such test set-up is complex, expensive, time-consuming and requires special equipment, trained personnel, and strict safety procedures. A relatively recent method to circumvent these issues and provide affordable results consists of using hollow specimens, thus applying the hydrogen pressure inside rather than outside the specimen. It allows to reduce the volume of hydrogen by several orders of magnitude and to perform the tests more efficiently and in a safer manner. This study focuses on evaluating the tensile properties of X65 vintage pipeline steel tested in a high-pressure hydrogen environment using hollow specimens. Tests are performed in 6 MPa H_2 and Ar at the nominal strain rate of 10^{-6} s^{-1} to evaluate the reduced area at fracture and the elongation loss. The effect of surface finishing on crack initiation and propagation is investigated by comparing two different manufacturing techniques. In this way, this study provides insights into the applicability of a novel, reliable, and safe testing method which can be used to assess the hydrogen-assisted ductility loss in metallic materials.

Keywords: Hydrogen embrittlement; In-situ tensile tests; Hollow specimens; Compressed gaseous hydrogen; Pipeline steel

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1. Introduction

The European Commission has indicated hydrogen as a clean, reliable, and versatile energy carrier with the potential to decarbonize several industrial sectors, road, air, maritime transport, and power production. The *Hydrogen Roadmap Europe* (FCH JU, 2019) has led to national and regional strategies to guide the increasing utilization of hydrogen-based technologies. Despite its unquestionable advantages, hydrogen can permeate most metallic materials and degrade their mechanical properties, eventually leading to sudden component failures and hazardous releases to the environment. Although hydrogen embrittlement (HE) has been investigated for decades, a unified theory to explain its underlying mechanism is still under debate.

It is generally believed that hydrogen embrittlement results from the synergistic interaction of a hydrogenated environment, a susceptible material, and mechanical loads (Lee, 2019). In fact, hydrogen atoms can diffuse through the metal lattice thanks to their size and tend to accumulate at microstructural features, such as vacancies, dislocations, grain boundaries and inclusions. In this sense, chemical composition and microstructure, along with temperature, strongly influence hydrogen diffusivity (Campari et al., 2023). Even if it is well understood that diffusible hydrogen is responsible for HE degradation, the specific mechanism with regard to low-carbon steels is still under debate and, most likely, depends on the combination of hydrogen-enhanced decohesion (HEDE) and hydrogen-enhanced localized plasticity (HELP) (Djukic et al., 2019).

In industrial practice, only a few standards consider hydrogen embrittlement in the design, inspection, and maintenance of existing and new components for the hydrogen value chain (Campari et al., 2023b). The lack of a unified regulatory framework often results in over-conservative design criteria and the use of a limited variety of high-performance materials. Hence, new standards are required to regulate the production and utilization of hydrogen technologies. These include also the existing natural gas infrastructure, in which the injection of hydrogen is actively examined. The development of technical guidelines for hydrogen compatibility of materials requires an extensive testing campaign. In this context, slow strain rate tensile (SSRT) tests are used as a screening methodology to assess the hydrogen impact on the tensile properties of metals. These tests must be conducted under realistic environmental conditions to assess the H_2 influence and should be reliable, fast, affordable and safe.

The state-of-the-art method for evaluating the effect of hydrogen on tensile properties consists of in-situ tests using a high-pressure autoclave. This standardized technique is suitable for a wide variety of structural materials, which commonly undergo tension while being exposed to hydrogen gas. It allows to adjust independently several parameters to reproduce the operating conditions of the components. Nevertheless, the large amount of pressurized hydrogen imposes significant safety measures, which result in high costs and long overall test duration. A method to circumvent these limitations and provide affordable and fast results consists of using hollow specimens (also known as tubular) instead of solid ones, thus applying hydrogen pressure on the inner surface of a mechanically made hole. Even if at an early stage and not yet standardized, this technique can reduce the volume of hydrogen by several orders of magnitude and allow to perform tests safely and effectively (Michler and Ebling, 2021).

This study aims to investigate the hydrogen influence on the tensile properties of API 5L X65 steel. Hollow specimens were extracted from the base metal of a pipeline for natural gas transport, which was in service for more than 30 years. The samples were tested in H_2 and in a reference environment. While pressure, temperature, and nominal strain rate remained the same for all the tests, two different finishing treatments were used to evaluate the influence of the inner surface roughness on hydrogen-induced degradation. The HE effect was evaluated microscopically by determining the embrittlement index (EI) and the relative elongation loss. Moreover, microstructural analysis and post-mortem fractographic analysis were conducted to characterize the material and to clarify the failure mechanism.

2. Testing techniques in hydrogen

Some methods to conduct tensile tests for evaluating the hydrogen effect on structural materials are already used and standardized. They can be broadly divided into two categories: (i) testing in a hydrogenated environment by applying the mechanical load concurrently with the exposure to H_2 and (ii) testing in the air after precharging with hydrogen. The second method, known as ex-situ testing, is not suitable for carbon and low-alloyed steels since the diffusion of hydrogen atoms (typically greater than 10^{-10} m^2/s) is too rapid for the timescale of SSRT tests, and a significant amount of hydrogen can exit the material between the precharging and the test completion (San Marchi and

Somerday, 2012). Hydrogen charging (both in-situ and ex-situ) can be performed by electrochemical or high-pressure gas charging. In the case of carbon steels, the former is less complicated and can simulate the cathodic protection of subsea pipelines. Charging in gaseous hydrogen, on the other side, simulates the presence of pressurized hydrogen gas in transport and storage equipment (Zhao et al., 2015). The most consolidated technique for in-situ testing with high-pressure hydrogen charging consists of using an autoclave, where various parameters, such as pressure, temperature, oxygen content, and nominal strain rate, can be independently adjusted to reproduce the actual operating conditions of a specific application. This method is well-established, standardized and described in ASTM G142-98 (2022) and ISO 11114-4 (2017). Nevertheless, these tests have high costs and long duration due to the extensive safety regulations required by the use of large amounts of H_2. An alternative method lies in using hollow specimens to apply the hydrogen pressure in the inner hole. This technique has lower costs and shorter test duration and is simple to handle and reproduce; furthermore, it is inherently safer due to the small amount of hydrogen inside the drilled hole. However, the hollow specimen methodology is not yet standardized, and the results cannot be directly compared with those obtained in the autoclave, mainly due to the sample geometry and differences in the fracture mechanics during which necking and fracture occur (Michler and Ebling, 2021).

This technique was also used in 2008 to test the effect of high-pressure hydrogen gas on the tensile properties of 304, 304L, and 316L austenitic stainless steels. Ogata (2008) performed the tests at room and cryogenic temperatures and compared the influence of surface roughness on the test results, finding good agreement with similar tests conducted in an autoclave. Thereafter, a similar test setup with cryostat and refrigerator allowed to test the fatigue performance of 304L stainless steel from room temperature down to 20 K (Ogata, 2010), thus showing the influence of H_2 on the fatigue crack growth rate (FCGR) at higher stress levels. Similar experiments on tensile properties and fatigue resistance were conducted on 304, 304L, 316L, and 630 stainless steels, strain-hardened 316, heat-resistant 660 stainless steel, and Alloy 718 at temperatures down to 20 K and hydrogen pressure up to 70 MPa (Ogata, 2012). In addition, Ogata (2018b) highlighted how the temperature can be changed simply by a refrigerant or a heater, and how no compressors are needed if the test pressure is lower than the pressure of the H_2 bottle. Another advantage is the low amount of hydrogen required, which can be easily handled with low risk and limited maintenance costs. In an attempt to standardize the testing method, Ogata and Ono (2018) evaluated the influence of the inner roughness on SSRT test results by comparing three different surface finishing (i.e., wire-cut, honing, and polishing). It was found that the results with the polished specimens were similar to those with solid ones, and were more sensitive to the change in environmental conditions. Boot et al. (2021) developed a testing machine to evaluate the tensile properties of X60 pipeline steel and its welds, varying the hydrogen pressure in the hole, while Michler et al. (2022) investigated the effects of different gaseous impurities, highlighting the role of purge cycles to reach the desired hydrogen purity within the hole. It turned out that since the volume of hydrogen is smaller, the impact of impurities becomes stronger. Finally, Michler et al. (2023) tested the yield and ultimate tensile strength of several austenitic steels, comparing hollow specimens with conventional ones and obtaining similar results. As shown, the utilization of hollow specimens gained more and more attention in the last decade, and further research aims at defining the differences with other testing techniques.

3. Materials and methods

The material investigated is a grade API 5L X65 pipeline steel manufactured in 1982 by Fukuyama Steel Works and used for natural gas transport since 1985. This carbon steel is the structural material of roughly 7% of the European pipeline network (Pluvinage, 2020). The pipe is longitudinally welded and produced through the UOE method. The material is extracted from the base metal of the inner pipe (with an outer diameter of 770 mm, wall thickness of 26 mm, and pipe length of 1000 mm) in the longitudinal direction. The chemical composition of the steel, obtained by the OES Spectrotest (SPECTRO Analytical Instruments GmbH), and the nominal composition, provided by the manufacturer, are given in Table 1.

The microstructure of the steel is also shown in Fig. 1 a) at ×500 magnification. It mainly consists of polygonal ferrite and pearlite in banded appearance. A microstructural feature of this material is the presence of plate-like bainitic bands, which can be responsible for an anisotropic behavior of the mechanical properties. The phase volume fraction has been estimated using the grey scale color coding to distinguish between different phases. The fraction of ferrite, corresponding to the white-colored areas in Fig. 1 b), is approximately 83%, while the fraction of pearlite and bainite,

Table 1. Measured and nominal chemical compositions of the API 5L X65 vintage pipeline steel

C	Mn	P	S	Cr	Cu	Mo	Ni	Nb	V	Ti	Si
0.07	1.53	0.013	< 0.002	0.02	< 0.01	0.01	0.01	0.033	0.076	0.009	0.25
< 0.1	< 1.6	< 0.025	< 0.015	< 0.25	< 0.25	< 0.05	< 0.25	< 0.05	< 0.1	< 0.02	< 0.6

corresponding to the black-colored areas, is around 17%. The average grain size, determined through the average grain intercept (AGI) method, is 4.2 μm (Alvaro et al., 2021). The material investigated has nominal yield and tensile strength equal to 526 MPa and 627 MPa, respectively. In addition, the mechanical properties have been obtained through slow strain rate tensile tests conducted in air at room temperature and with a nominal strain rate of $2.5 \cdot 10^{-4}$ s^{-1}. The measured yield and tensile strength are 518 MPa and 590 MPa, respectively.

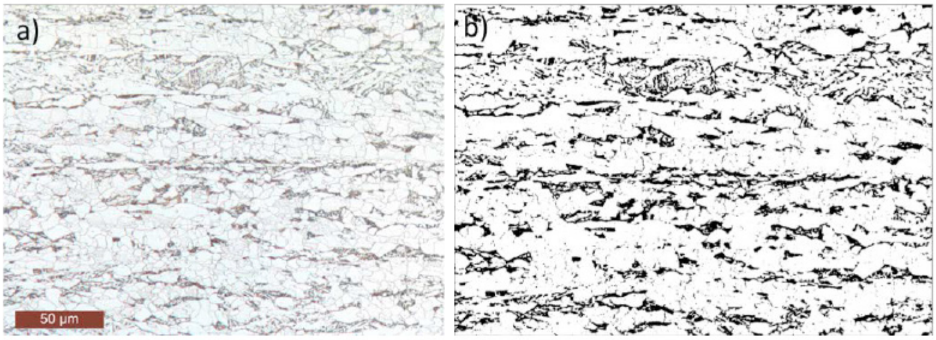

Fig. 1. Optical micrographs of the API 5L X65 microstructure in longitudinal direction

The outer geometry of the specimens has been machined by turning, and the inner part has been manufactured through two different techniques to evaluate the effect of surface finishing on crack initiation and propagation. Five specimens have been drilled, thus obtaining a rougher inner surface with a nominal average roughness (R_a) and a ten-point height of irregularities (R_z) of 1.5 μm and 8.5 μm, respectively. In contrast, the remaining five specimens have been reamed after drilling, thus obtaining a smoother inner surface with R_a and R_z equal to 0.1 μm and 1.4 μm, respectively. The tests have been conducted with hydrogen gas of quality 5.0 (i.e., purity of 99.999% and maximum oxygen content of 2 ppm), while argon has been used as a reference gas. The gas pressure of 6 MPa has been applied in the hole of the specimens at room temperature. The desired oxygen content was obtained by purging six times between 1 and 6 MPa both in hydrogen and argon, as described by Michler et al. (2022). All the tests have been conducted at a nominal strain rate of 10^{-6} s^{-1}, corresponding to a displacement rate of $2.54 \cdot 10^{-5}$ μm/s. The equipment required for the hollow specimen technique was integrated with a conventional machine for SSRT tests in a standard laboratory environment at room temperature. The drawing of the hollow specimen is shown in Fig. 2.

The reduced area at fracture (RA) is the most relevant parameter to quantify the hydrogen effect on tensile properties. The values of RA were used to calculate the embrittlement index through the following formula:

$$EI = \frac{RA_{Ar} - RA_{H_2}}{RA_{Ar}} \cdot 100 = \frac{[(A_i - A_f)\ /\ A_i]_{Ar} - [(A_i - A_f)\ /\ A_i]_{H_2}}{[(A_i - A_f)\ /\ A_i]_{Ar}} \cdot 100 \tag{1}$$

where RA_{Ar} and RA_{H_2} are the reduced area at fracture in argon and hydrogen, respectively, and A_i and A_f are the initial and the final fracture areas, respectively. The area was measured through a digital optical microscope Keyence VHX-5000. Finally, the fractography was performed through a scanning electron microscope FEI Quanta 650 FEG with a high voltage of 20 kV.

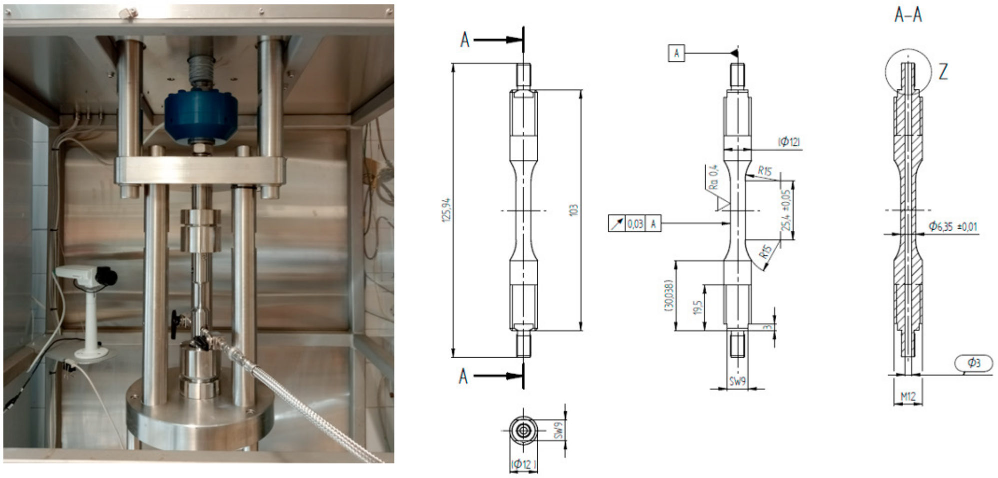

Fig. 2. SSRT test machine and drawing of the hollow specimens

4. Results and discussion

The influence of hydrogen on the mechanical properties has been evaluated in terms of embrittlement index and elongation loss by comparing the test results in hydrogen and argon. Fracture area, embrittlement index, elongation at failure, and elongation loss are reported in Table 2 for each test. In addition, the force-displacement curves are plotted in Fig. 3. Tests conducted in H_2 gas and Ar are marked with red and blue lines, respectively.

All drilled samples tested in 6 MPa H_2 showed a loss in elongation compared to those tested in 6 MPa Ar. The values range from 5.90% to 41.93% (i.e., 21.97 ± 18.33%), indicating significant scattering. Similarly, greater fractured areas were measured in H_2, thus resulting in embrittlement indexes from 14.88% to 38.67% (i.e., 27.76 ± 12.02%). The same effect was observed for the reamed samples tested in hydrogen. In this case, the elongation loss ranges from 7.33% to 25.80% (i.e., 16.45 ± 9.24%) and the embrittlement index from 6.39% to 23.75 % (i.e., 13.37 ± 9.17%). In this sense, not only the average elongation at failure of reamed specimens is higher compared to that of the drilled ones, but also the scattering is lower. Therefore, it is concluded that a smoother inner surface with fewer notches and grooves results in lower susceptibility to HE. Surface defects might act as crack initiation sites in hydrogenated environments, constituting prefered paths for hydrogen uptake and diffusion, thus accelerating the specimen's failure (Michler and Ebling, 2021). In contrast, both the elongation at failure and the embrittlement index of the reference samples are similar for drilled and reamed specimens. Hence, surface finishing has a negligible influence on the tensile properties of X65 steel in inert environments.

Table 2. Results of the SSRT tests in hydrogen and argon

ID	Type	Environment	Fracture area [mm^2]	Embrittlement index [%]	Elongation at failure [mm]	Elongation loss [%]
AR_D_1	Drilled	6 MPa Ar	7.98	-	5.99	-
AR_D_2	Drilled	6 MPa Ar	8.46	-	6.17	-
H2_D_1	Drilled	6 MPa H_2	13.09	29.73	4.98	18.07
H2_D_2	Drilled	6 MPa H_2	14.56	38.67	3.53	41.93
H2_D_3	Drilled	6 MPa H_2	10.86	14.88	5.72	5.90
AR_R_1	Reamed	6 MPa Ar	8.13	-	6.12	-
AR_R_2	Reamed	6 MPa Ar	8.04	-	6.01	-
H2_R_1	Reamed	6 MPa H_2	9.73	9.96	5.08	16.23
H2_R_2	Reamed	6 MPa H_2	12.01	23.75	4.50	25.80
H2_R_3	Reamed	6 MPa H_2	9.14	6.39	5.62	7.33

The behavior of the tensile curves is unchanged before the onset of necking for both drilled and reamed specimens, as shown in Fig. 3. For this reason, it is concluded that the ultimate tensile strength (UTS) is not influenced by the hydrogen atmosphere. This result is in accordance with the findings of Michler et al. (2021, 2022, 2023) made for comparable steels. In contrast, for the samples tested in hydrogen, the plastic behavior changes after the necking occurs in comparison to the samples tested in argon. This leads to a decrease in elongation at fracture which is discussed above and is also shown in Fig. 3. This effect describes the loss of ductility and is also reported by Lee et al. (2011) and Wang et al. (2022) for similar steels in the presence of hydrogen.

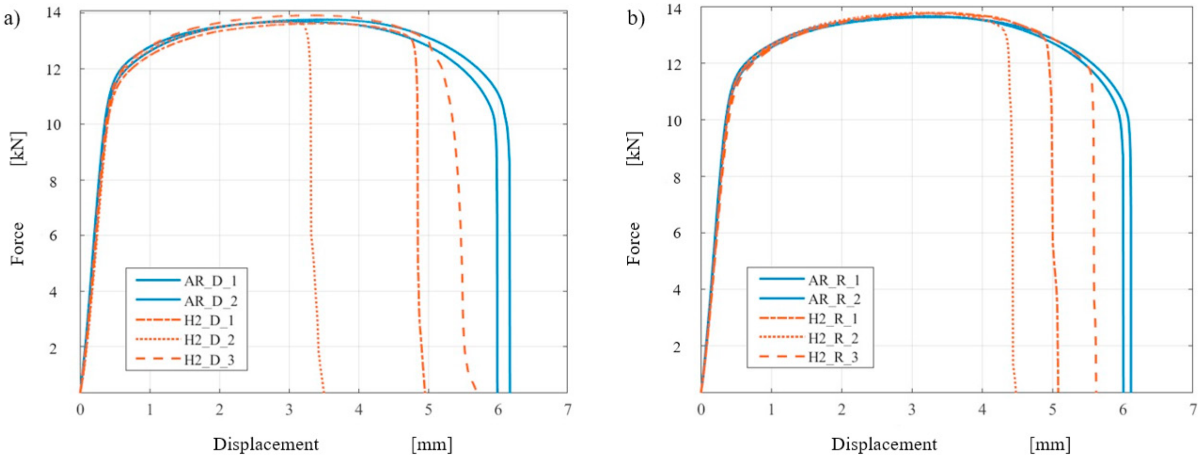

Fig. 3. Force – displacement curves for a) drilled specimens in Ar and H_2, and b) reamed specimens in Ar and H_2

The fractographic analysis of the specimens supports the findings of the tensile tests. Fig. 4 a) shows SEM image with a general view of the reamed reference sample, and the fracture surface in higher magnification is shown in Fig. 4 b). The fracture surface is characterized by an elliptical shape which indicates an anisotropic plastic deformation due to the banded microstructure. The surface topography is inclined and dimples indicating ductile microvoid coalescence are clearly visible in Fig. 4 b). Similar behavior has been observed for the drilled specimens tested in argon.

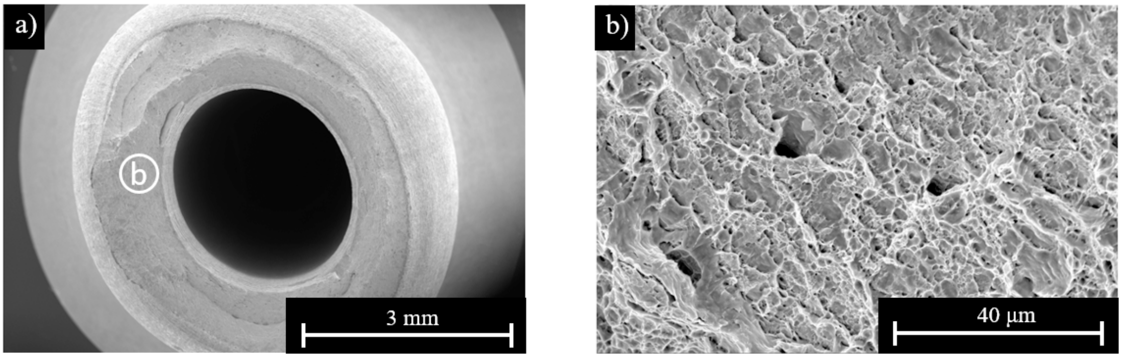

Fig. 4. Post-mortem analysis of the reamed specimen tested in 6 MPa Ar a) in a general view and b) in a higher magnification

The elliptical shape indicating the anisotropic deformation is also observed for the specimens tested in hydrogen gas. The fracture surfaces of the drilled and reamed samples are shown in Fig. 5 a) and b), respectively. The images of higher magnification of the fracture surfaces show the presence of three distinct areas: quasi-cleavage (QC), microvoid coalescence (MVC), and transition region. The region close to the inner wall is mostly characterized by a transgranular brittle fracture. The surface characterized by the QC region is shown in Fig. 5 c) and f) for drilled and reamed specimens, respectively. In contrast, the outer area is characterized by dimples and is fairly similar to the fracture surface observed for the specimen in argon in Fig. 4 b). It is assumed that the crack propagation is not concentric to the longitudinal axis of the sample. As a result, the crack penetrates the wall at a local spot, resulting in hydrogen release. As hydrogen is released from the inner volume of the specimen, further hydrogen uptake at the crack tip is not possible. This leads to the ductile behavior of the material and MVC at the outer surface. The MVC area is shown in Fig. 5 e) and h) for drilled and reamed specimens, respectively. Finally, a transition zone exists between the QC and the MVC regions and has a morphology with mixed features, as shown in Fig. 5 d) and g) for drilled and reamed samples, respectively.

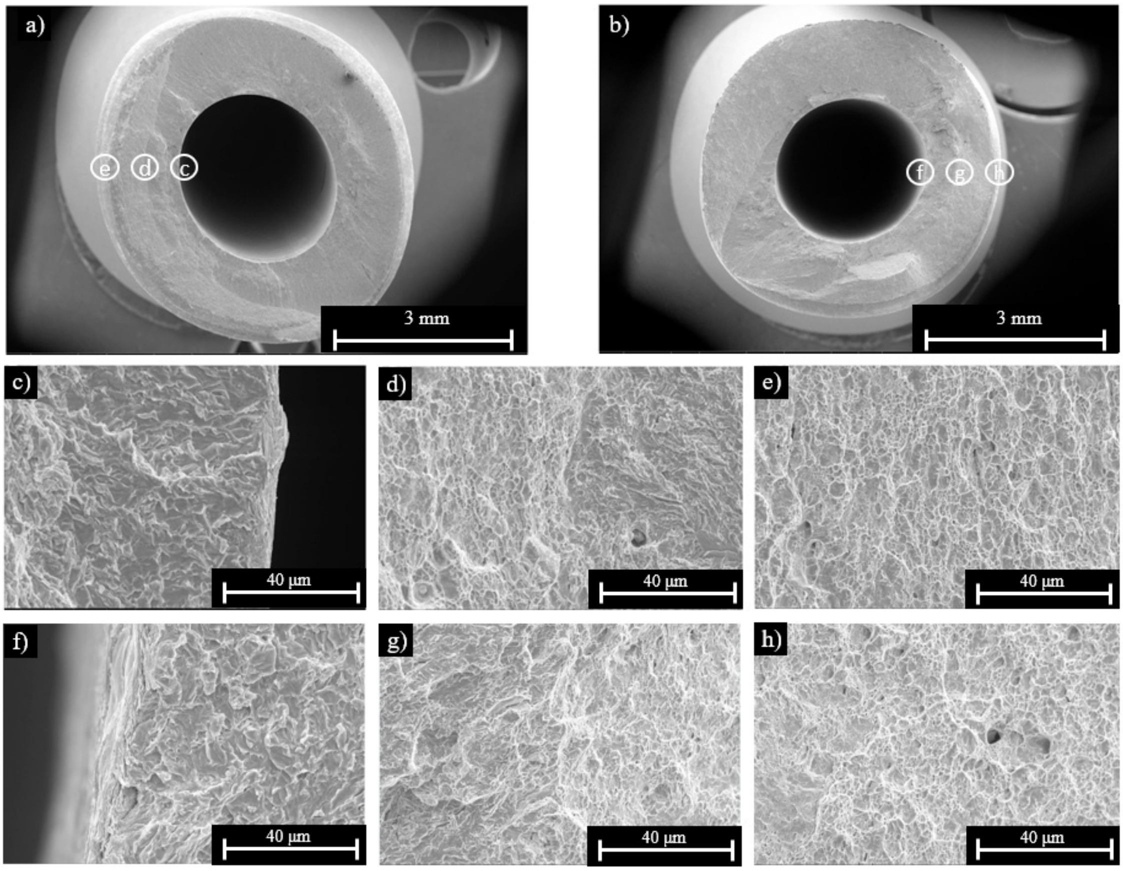

Fig. 5. Post-mortem analysis of a) drilled and b) reamed specimens tested in 6 MPa H_2 in a general view. Higher magnifications of the QC areas of c) drilled and f) reamed specimens, the transition areas of d) drilled and g) reamed, and the MVC areas of e) drilled and h) reamed

5. Conclusions

This study focuses on evaluating the tensile properties of X65 vintage pipeline steel tested in a high-pressure hydrogen environment using the hollow specimen technique. Two different machining techniques were applied and compared to evaluate the effect of inner surface roughness on crack initiation and propagation. The results show how the elongation at failure always decreases in hydrogenated environments; nevertheless, the drilled specimens manifest a more marked reduction in elongation and more scattering of the results. Similar considerations can be made for the embrittlement index, which is significantly higher for drilled specimens. The test results' dependence on the surface finishing does not apply to the specimens tested in an inert environment. Minor machining defects have a high potential of becoming crack initiation sites when they come into contact with pressurized hydrogen gas; therefore, the conditions of the surface must be considered when assessing the HE susceptibility of components for H_2 transport and storage. A direct comparison of hollow (HS) and conventional (CS) specimens is required to evaluate the influence of the specimen geometry on hydrogen-assisted degradation of materials' tensile properties. In addition, a thorough inspection of the hollow specimens' inner surface is necessary to evaluate the number of surface defects and their shape, depending on the specimen type (i.e., drilled or reamed). This study shows that, in principle, it is possible to evaluate the HE susceptibility of materials using hollow specimens. Nonetheless, further research is necessary to make these tests directly comparable with those in the autoclave and to standardize this technique.

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