

APPLICATION OF MECHANICAL TESTING METHODS FOR EVALUATING HYDROGEN EMBRITTLEMENT OF STRUCTURAL STEELS

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Abstract

Hydrogen is considered one of the primary energy carriers of the future due to the growing demand for clean and renewable energy. Consequently, its global consumption is expected to increase significantly, as indicated by available forecasts. During the production, storage, transportation, and utilization of hydrogen, it interacts with a wide range of structural steels. The presence of elevated hydrogen content in steels can lead to changes in several of their properties. From a practical standpoint, the most critical effect is that it may result in material degradation due to so-called hydrogen embrittlement, potentially causing complete failure of structures, pressure vessels, and similar components. To ensure the long-term reliability and service life of steels operating in hydrogen-containing environments, it is therefore essential to test their resistance to hydrogen embrittlement under various mechanical, thermal, and pressure conditions that accurately simulate real operating environments. The present paper focuses on the application of conventional and unconventional testing methods for evaluating the resistance of structural steels in hydrogen-containing gaseous environments. The test procedures described in the paper can be used for assessment of the resistance to hydrogen embrittlement of materials used in existing gas infrastructure as well as for optimizing the manufacturing processes for components operating in high-pressure hydrogen environments, or their heat treatment, in order to achieve the desired strength properties, which often also yields significant economic benefits.

Keywords: Structural steels, hydrogen embrittlement, mechanical testing methods, fracture mechanics, fractography

1. INTRODUCTION

During the production, storage, transport, and use of hydrogen, it interacts with structural materials, particularly steel. An increased hydrogen content in steel can significantly affect its properties. From a practical standpoint, it is critical that this can cause material degradation known as hydrogen embrittlement. This manifests itself primarily through a reduction in plastic properties during tensile tests, or through changes in the yield stress and tensile strength, changes in the fracture mechanism, and a decrease in resistance to brittle fracture. The most serious consequence is the formation of cracks, which can lead to catastrophic failure of structures or pressure vessels, with significant human, environmental, and economic impacts [1–3].

Various types of mechanical tests can be used to evaluate the susceptibility of materials to hydrogen embrittlement, such as tensile tests, disk fracture tests, wedge-hole loading (WOL), fracture toughness tests, and fatigue crack growth tests. Tests can be static (e.g., controlled fracture test) or dynamic (e.g., fatigue test) [4, 5]. Mechanical tests for evaluating hydrogen embrittlement can be performed in-situ or ex-situ. In-situ tests are performed by simultaneously exposing specimens to a hydrogen-containing atmosphere and a load (mechanical, thermal, etc.). Ex-situ tests, on the other hand, are performed by applying a load to specimens

pre-saturated with hydrogen in an air environment or an inert/controlled atmosphere. The advantage of ex-situ tests is their lower operational complexity. Ex-situ experimental tests have certain limitations, as the results obtained through this approach are subject to a significant degree of uncertainty; this is because, under real operating conditions, hydrogen, mechanical stress, and temperature act on the material simultaneously, whereas in ex-situ laboratory tests, these factors are applied sequentially. Another significant aspect is the fact that when a sample is exposed to the atmospheric environment during measurement, hydrogen diffusion from the material may occur due to its limited dimensions. This phenomenon significantly reduces the reliability of ex-situ tests and complicates the interpretation of their results. Under laboratory conditions, samples can be saturated using two different methods: electrochemically, usually at room temperature, or by saturation with a gaseous hydrogen phase [6–8].

The aim of this paper is to present methods for evaluating hydrogen embrittlement using tensile tests, specifically through electrolytic hydrogenation and in-situ loading of test specimens in a high-pressure hydrogen environment. The tests were conducted on a pipe made of X52 steel, which is a typical example of structural materials used in the manufacture of gas pipelines.

2. EXPERIMENTAL METHODS

In electrolytic saturation, the test specimen is connected as the cathode, and the anode is made of platinum or stainless steel. The direct current applied to the electrolyte causes it to decompose, producing hydrogen ions—protons. The most used electrolyte is a diluted solution of H_2SO_4 in H_2O , or solutions of methanol with H_2SO_4 and NaOH , together with hydrogen inhibitors such as NaAsO_2 and AsO_2 . The potential difference created by the power source causes the positive hydrogen ions to move from the electrolyte to the test specimen, i.e., the cathode (a negatively charged electrode attracting a positive charge). This flow of charged particles causes a layer of hydrogen to form on the surface of the test specimen. As with any other process, the entropy of the system increases and manifests itself through diffusion, i.e., the spontaneous penetration of hydrogen into the metal structure. The diffusion rate is influenced by temperature and the concentration of hydrogen at the surface itself. Cathodic hydrogenation is a stable method because hydrogen atoms are rapidly delivered to the metal surface, resulting in a homogeneous distribution of hydrogen concentration. A major advantage of this method is the wide range of test parameters. During the experiment, it is possible to vary the types of electrolytes, current densities, and hydrogenation times, as well as to adjust the hydrogenation temperature and modify the test specimens in various ways [9].

When testing a material's resistance to hydrogen embrittlement, two different approaches can be used: either the test specimen for the tensile test is saturated with hydrogen for a predefined period of time and the test is then conducted in air according to standardized methods, or the tests are performed in situ in a high-pressure hydrogen environment. From an operational standpoint, if the testing laboratory's capabilities allow, it is preferable to use the latter approach, in which the test specimen is subjected to the simultaneous effects of tensile stress and high-pressure hydrogen. Test specimens are loaded in an autoclave, which is purged with nitrogen prior to sample exposure to displace air and subsequently evacuated to a pressure below 130 Pa. Hydrogen at the required pressure is then introduced from a pressure cylinder, and the test itself is performed using the slow strain rate test (SSRT) [10].

When conducting the in-situ SSRT test in a high-pressure hydrogen environment, a 0.55-liter autoclave was integrated into the MTS 100 kN servo-hydraulic testing machine. The procedure involved, in the first phase, clamping the test specimen into the autoclave's working chamber along with a longitudinal strain gauge. After evacuating and filling the autoclave with hydrogen, the test specimen was lightly loaded three times and then unloaded to break any oxide film on its surface that might impede hydrogen penetration into the material. Prior to the actual start of the test, it was exposed to hydrogen for approximately 30 minutes. The test was then performed at a constant crosshead rate approximately one and a half orders of magnitude lower than that used in the tensile test conducted in air. The total duration of a single test is then, depending on the material's

resistance to hydrogen embrittlement, measured in hours. The same test is then performed in air. From the tensile test diagrams, the values of tensile strength, yield strength, elongation, and reduction of area are subsequently determined. The effect of high-pressure hydrogen is then expressed by the so-called hydrogen embrittlement exponent (HEE) for selected parameters, e.g., for elongation in the form of equation (1).

$$HEE_A = \frac{A_{air} - A_{hydrogen}}{A_{air}} \cdot 100 \% \quad (1)$$

where:

HEE_A - hydrogen embrittlement equivalent based on elongation (1)

A_{air} – elongation in the air (%)

$A_{hydrogen}$ – elongation in hydrogen (%)

The prepared samples were hydrogenated in a 0.05 mol% H_2SO_4 solution with a small amount of KSCN added at a concentration of 1 g/l. KSCN is added to prevent atomic hydrogen from recombining into H_2 , which would significantly reduce the efficiency of the hydrogenation. Two different current densities, 1 $mA \cdot cm^{-2}$ and 5 $mA \cdot cm^{-2}$, were used for hydrogenation. A tensile test was then performed immediately after removing the samples from the electrolyte to prevent hydrogen diffusion from the sample. In this case as well, the same mechanical property parameters were evaluated as in the case of hydrogenation with gaseous hydrogen in an autoclave.

For comparison, tensile tests were performed on cylindrical test specimens with a diameter of 6 mm in air, in a hydrogen gas environment at a pressure of 10 MPa, and after electrolytic hydrogenation at two different current densities.

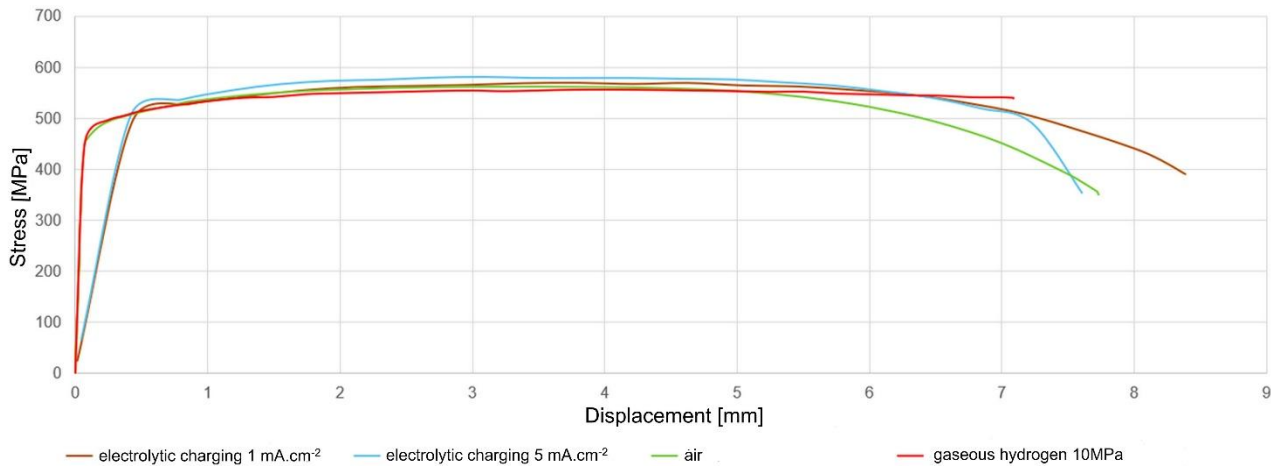


Figure 1 Stress – strain curves for all test conditions

3. RESULTS

Figure 1 compares the tensile diagrams from tests conducted in air, in situ in a high-pressure hydrogen environment, and after 8 hours of electrolytic hydrogenation at current densities of 1 $mA \cdot cm^{-2}$ and 5 $mA \cdot cm^{-2}$.

The stress - strain curves do not completely overlap because the tests were performed on two different testing machines and under different configurations. However, an evaluation of the data reveals that approximately similar strength properties were achieved under all test conditions. As for the plastic properties, earlier neck

formation can be observed in the case of the sample after electrolytic hydrogenation at a current density of $5 \text{ mA}\cdot\text{cm}^{-2}$ (blue curve), which is associated with a reduction in elongation.

A summary of the resulting mechanical properties, along with the calculated HEE values for ductility reduction, is presented in **Table 1**. The measured results clearly show that the analysed X52 steel pipe resists the effects of high-pressure hydrogen relatively well, with ductility remaining virtually unchanged. Exposure to electrolytic hydrogen causes a slight decrease in plastic properties during saturation at lower current densities; as the current density increases, the decrease in ductility becomes more pronounced, reaching approximately 8%. The increase in the yield strength of samples subjected to electrolytic hydrogenation may be due to the higher test speed, which is, in a sense, necessary for this type of testing to eliminate hydrogen from the test specimen.

Table 1 Calculated values of mechanical properties

Test condition	Tensile Strength (MPa)	Yield Stress (MPa)	Elongation (%)	HEE _A (1)
Air	561	478	27.4	N/A
Gaseous hydrogen, 10 MPa	556	477	27.6	0
Electrolytic charging, $1 \text{ mA}\cdot\text{cm}^{-2}$	569	527	27.2	0.73
Electrolytic charging, $5 \text{ mA}\cdot\text{cm}^{-2}$	583	537	25.1	8.39

The fractured specimens were then examined under a JEOL JSM-5510 scanning electron microscope equipped with an X-max 20 energy-dispersive X-ray analyser from OXFORD Instruments. The evaluation was performed primarily in secondary electron (SE) mode, and in several cases also in backscattered electron (BSE) compositional contrast mode. The aim of the analysis was to identify any changes in the fracture mechanisms on the test specimens. A comparison of the fracture surfaces of the samples after electrolytic hydrogenation and after in-situ testing in high-pressure hydrogen is shown in **Figures 2 and 3**.

While samples tested under air exhibited exclusively transcrystalline ductile fracture, certain changes in the fracture mechanism were observed in the hydrogenated samples. In samples after electrolytic hydrogenation at a current density of $5 \text{ mA}/\text{cm}^2$, transcrystalline ductile fracture predominated, with a smaller proportion of cleavage fracture in the so-called fish-eye regions. This morphology is typical for this state of the material. After loading the samples in high-pressure hydrogen, the occurrence of transcrystalline cleavage fracture can be observed to a lesser extent on the fracture surface

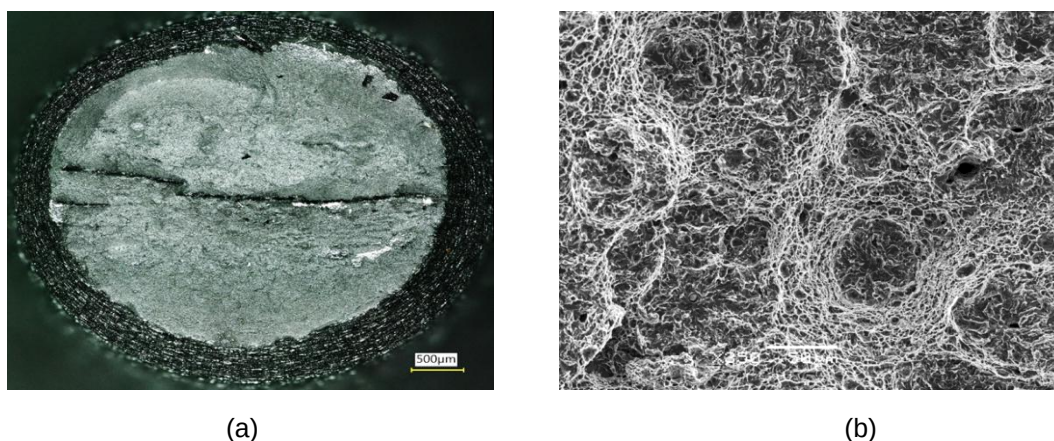


Figure 2 Overall view on test specimen loaded in electrolytic hydrogen at current density $5 \text{ mA}\cdot\text{cm}^{-2}$ (a) and transcrystalline ductile fracture with occurrence of “fisheyes” (b)

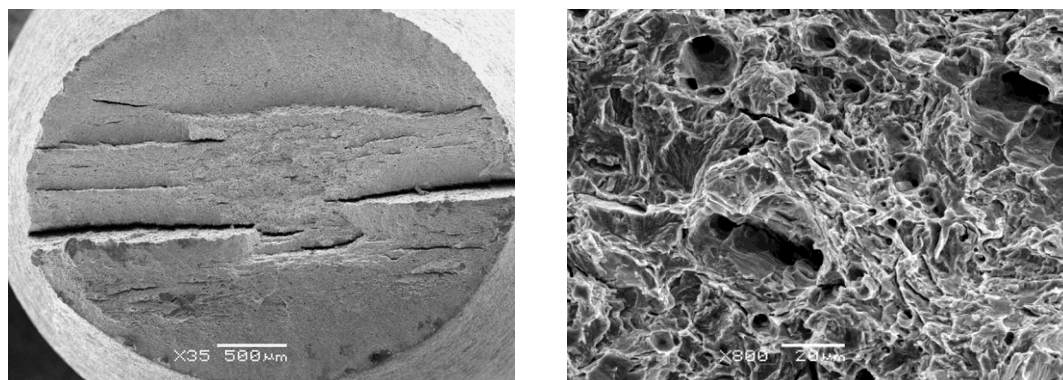


Figure 3 Overall view on test specimen loaded in gaseous hydrogen (a) and transcrystalline morphology with a lower portion of cleavage fracture (b)

4. CONCLUSION

Based on the results obtained, it can be concluded that structural steel X52, which is used in the gas transport system and has been evaluated exhibits good resistance to hydrogen embrittlement. This is achieved thanks to its ferritic-pearlitic structure, in which hydrogen diffusivity is not particularly high, as is the case with acicular ferrite. The material is sufficiently strong due to alloying with manganese and microalloying with vanadium, titanium, and niobium. The low content of elements that form carbides or non-metallic particles, on which hydrogen can adsorb, also contributed to the good resistance. The material became partially embrittled after hydrogenation, but after a fivefold increase in current density from 1 mA/cm² to 5 mA/cm², the brittleness did not increase significantly.

Fracture surfaces of the hydrogenated samples showed the presence of cleavage fractures, which, in the case of electrolytic hydrogenation, were concentrated in areas known as "fisheye" defects. An important factor in a material's resistance to hydrogen embrittlement is its microstructure, as the presence of inclusions typically leads to significant degradation of plastic properties. The selection and heat treatment of materials for the manufacture of gas system components must therefore be chosen regarding both their strength properties and the degree of degradation resulting from exposure to high-pressure hydrogen, as well as the risk of brittle fracture due to long-term operation.

Based on the results, the method used to evaluate the resistance of steels to hydrogen embrittlement must be selected primarily regarding the environment to which the material is exposed during service. It can be stated that electrolytic hydrogenation represents a less favourable condition for the material and thus provides conservative results, whereas methods based on in-situ loading of samples in a high-pressure hydrogen environment reflect the actual operational load and the simultaneous effects of tensile stresses and the environment. Evaluation based on SSRT testing represents only one of many methods for assessing a material's resistance to hydrogen embrittlement. Other methods based on fracture mechanics principles describe brittle fracture behavior in the presence of a sharp defect (crack), which further accentuates the undesirable effect of hydrogen on the material.

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