USE OF EXISTING STEEL PIPELINE INFRASTRUCTURE FOR GASEOUS HYDROGEN STORAGE AND TRANSPORT: A REVIEW OF FACTORS AFFECTING HYDROGEN INDUCED DEGRADATION

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ABSTRACT: The transition of the existing natural gas grid infrastructures towards transport and storage of hydrogen gas plays a prominent role in the global decarbonization of the energy landscape. Hydrogen absorption into pipeline steels may lead to a ductility decrease, particularly in the presence of stress concentrations. Hydrogen lowers the fracture resistance of steels, which may render them to become susceptible to crack extension under static loading. Furthermore, due to fluctuations in gas pressure and applied loads on the pipeline structure, hydrogen assisted fatigue crack growth could take place (even at relatively low hydrogen gas partial pressures). Therefore, the use of existing pipeline systems (initially not designed) for pressurized gaseous hydrogen transport requires prior confirmation of their fitness-for-service. Mechanical (load level and cycle frequency), material (microstructure, chemical composition and the presence of welds), and environmental variables (gas pressure, gas composition and temperature) influence the severity of hydrogen embrittlement by gaseous hydrogen. Investigations have indicated that the take-up of hydrogen by pipeline steel may be mitigated by the addition of gas impurities, i.e. inhibitors, to the gas mixture. The current work presents a literature overview of the relevant aspects in this consideration, indicating ample scope for further research in all abovementioned aspects.

Keywords: hydrogen gas, natural gas pipelines, hydrogen embrittlement, fatigue, inhibitors

1. INTRODUCTION

An increasing awareness of the greenhouse effect and the resulting global warming promotes an alteration of the energy system. Europe has the ambition to become the first climate neutral continent by 2050, i.e. European Green Deal [1], by implementation of hydrogen as an energy carrier in the renewed energy landscape. There are various ways to produce hydrogen in a carbon-friendly manner. For instance, electricity from renewable energy sources (e.g. wind, solar) can be used to produce hydrogen gas, following the power-to-gas (P2G) principle, which can hence be stored temporarily ("green hydrogen"). Moreover, hydrogen combustion does not lead to CO_2 formation. The gas systems may, as such, store surpluses of green electricity which would otherwise be lost. An efficient logistic system for hydrogen storage and transport is essential for the successful implementation of hydrogen as an energy carrier. Purpose-built hydrogen pipelines have been in service for many years. These hydrogen pipelines are operated at relatively low pressures under static loads and are not meant for long-distance and high volume hydrogen transportation; their materials have been selected with the deliberate aim of hydrogen transportation. However, as the initial capital and time cost of new hydrogen suited pipeline construction is high, the timely transition to a sustainable energy-based economy necessitates the incorporation of the existing natural gas infrastructure into hydrogen logistics.

Using this gas infrastructure would enable transportation and storage of significant volumes of hydrogen (or hydrogen mixtures with natural gas). However, hydrogen gas transport in natural gas pipeline steels brings numerous complications.

Enriching natural gas with hydrogen gas will lead to direct contact of gaseous hydrogen with the pipeline networks and the associated installations, which were designed specifically for contact with natural gas. As such, hydrogen may be taken up by carbon steels, potentially triggering a variety of degradation modes in otherwise high performance steels. This phenomenon is generally referred to as hydrogen embrittlement (HE). Hydrogen may cause significant losses in ductility particularly in the presence of stress concentrations [2–6]. Additionally, hydrogen is known to potentially increase the growth rate of existing cracks under cyclic pressure, deteriorate the fracture toughness and, in excess quantities, give rise to hydrogen induced cracks. Consequently, the service life of pipelines can be decreased in comparison to service with natural gas.

The chemical composition, heat treatment and weldability requirements for structural steels are more stringent for hydrogen transport compared to natural gas systems due to the occurrence of hydrogen embrittlement phenomena. Moreover, hydrogen is highly flammable with a flammability range in air of 4-75 vol% compared to 5.28-15 vol% for methane. Furthermore, hydrogen exhibits a lower ignition energy than methane, i.e. 0.018 mJ vs. 0.28 mJ [7]. The explosive character of hydrogen may lead to severe safety hazards in case leakage or failure of a pipeline by hydrogen induced damage occurs. For the reasons mentioned above, hydrogen embrittlement is acknowledged as one of the main challenges in a hydrogen-based energy system, challenging the long-term safety, durability, and performance of existing pipeline systems.

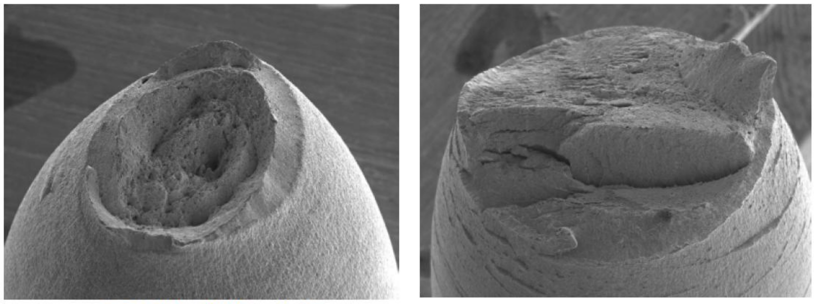
Despite a long history of investigation of hydrogen embrittlement [8], hydrogen still induces unpredictable failures in a great number of applications, e.g. storage tanks, fuel cells, nuclear power plants, wind turbines, (sulfide) stress corrosion cracking, welds, etc. [9–12]. Due to the complexities involved in the physics of hydrogen assisted material degradation, consensus lacks on which (and to what extent) pipeline steels are susceptible to the adverse effects of hydrogen. Mechanical, environmental and material variables can all influence the severity of hydrogen embrittlment by gaseous hydrogen. Such variables include loading rate, load cycle frequency, gas pressure, gas composition, material microstructure and composition, and the presence of welds (associated with different microstructures, geometrical stress concentrations, the potential presence of weld flaws and residual stresses) [2]. Understanding the influence of hydrogen on pipeline materials is of primordial importance prior to implementation of the existing pipeline infrastructure for hydrogen/natural gas mixture transport. Hydrogen degrades materials in various manners, of which hydrogen assisted cracking (HAC) and related hydrogen accelerated fatigue crack growth are of interest when considering gaseous hydrogen uptake in pipeline steels. HAC occurs in the presence of hydrogen and a mechanical stress. Diffusible hydrogen is the major player in facilitating crack growth. The phenomenon can manifest itself in the presence of a minor amount of hydrogen, i.e. for some pipeline steels even less than 1 wppm [13] can cause embrittlement. Hydrogen assisted cracking is associated with a loss in global plasticity, which manifests itself as fracture of a material at subcritical stress levels (i.e. before reaching the ultimate tensile stress of the material) due to embrittlement of highly stressed regions ahead of cracks or notches caused by an increased hydrogen concentration [13]. A variety of mechanisms can take place by which hydrogen degrades the material's mechanical behavior. Hydrogen-enhanced decohesion [14,15], hydrogen-enhanced localized plasticity [16–19], adsorption-induced dislocation emission [20,21], and hydrogen-enhanced vacancy formation [21] are proposed in literature as main mechanisms to explain hydrogen assisted cracking. Although there are clear differences between these mechanisms and in spite of the vast amount of research carried out, the question of which mechanism is governing hydrogen assisted crack initiation and propagation (for a specific material) is still vividly debated, e.g. [22–24]. Often, results need to be explained by using a combination of different mechanisms [25–27]. The dominant mechanism is determined by the alloy/environment combination and, therefore, by parameters such as the metallurgical condition of the material, the hydrogen content and the exposure environment, temperature and loading conditions [28,29]. All mentioned theories share the belief that hydrogen in its dissociated, dissolved state is responsible for embrittlement, quite independent of the initial source of the hydrogen, as long as kinetic barriers do not prevent the entry of hydrogen. Fatigue is a material failure mode due to cyclic loading and is arguably the most important failure mechanism in structures subjected to cyclic stresses, such as pipeline structures. Hydrogen assisted cracking can increase the fatigue crack growth rate (FCGR) in structural metals which are subjected to stress cycling. Hydrogen assisted fatigue crack growth takes place under cyclic loading even at relatively low hydrogen gas partial pressures (<1 MPa) and is considered to be the main mechanism of material degradation in pipeline steels transporting hydrogen gas. Absorbed hydrogen diffuses to regions with high triaxial stress (e.g. the crack tip of a certain defect) and locally influences the resistance to an external or internal load.

It is clear from the above that the inclusion of hydrogen in the energy transition requires prior confirmation of the fitness-for-service of the pipeline systems for pressurized gaseous hydrogen transport. Moreover, codes and standards are in the process of being adapted to take into account the repurposing of natural gas pipelines to hydrogen gas transportation (e.g. ASME B31.12 [30,31]). This work presents a literature overview on hydrogen induced degradation in pipeline steels. First, the impact of hydrogen on the mechanical properties of pipeline steels under various loading scenarios is discussed. Furthermore, the influence that material and environmental characteristics exhibit on the hydrogen embrittlement susceptibility is debated. The influence of these parameters should be well-understood to better quantify the risks of introducing hydrogen into the natural gas pipeline system.

2. HYDROGEN EMBRITTLEMENT OF PIPELINE STEELS

The current section aims to elucidate the effect of hydrogen on the mechanical properties and failure behavior of pipeline steels under different loading scenarios in a high-pressure hydrogen gas environment. A distinction is made between quasi-static and cyclic load testing. A quasi-static load refers to a load that is constant in time or changes very slowly (sufficiently slow to retain a steady state hydrogen distribution in the material). Quasi-static loads in pipelines originate from the gas pressure of the transported gas or internal residual stresses in the material. Cyclic loading is associated with fluctuations in gas pressure and applied loads on the pipeline structure when used to transport and store pressurized hydrogen gas. Hydrogen assisted fatigue crack growth takes place under cyclic loading and is considered to be the main mechanism of hydrogen degradation in pipeline steels transporting hydrogen gas. Moreover, impact loading is shortly discussed at the end of the section.

The resistance of a material against a quasi-static load is described by distinct mechanical properties, i.e. tensile properties (yield strength, tensile strength, ductility) and fracture resistance properties (fracture toughness). The effect of gaseous hydrogen on the yield and tensile strength of pipeline steels is minor [2,32,33]. On the contrary, the ductility of pipeline steel, which is typically expressed in terms of reduction in area at fracture (RA) or elongation to fracture, is often significantly reduced in the presence of gaseous hydrogen. For smooth specimens, the RA in hydrogen gas can be reduced with values ranging from 20% to up to 50% when compared with tensile test results in air [2,34]. Fig. 1 shows the fracture surfaces of smooth tensile test specimens, in an inert helium and hydrogen gas environment [35]. The hydrogen embrittlement is evident, illustrated by the difference in RA. With notched specimens the reduction in RA was seen to be even more prominent, with reductions as high as 80% relative to the RA when tested in air [2]. Notched specimens are associated with an increased local hydrostatic stress compared to smooth specimens, which makes them more sensitive to HE [3,36]. Moreover, literature results suggest that the RA reduction increases with increasing strength, suggesting a trend of increasing susceptibility to hydrogen embrittlement with higher pipeline steel grades [33] (Fig. 2).



5.5 MPa (800 psi) He, $1x10^{-4}$/sec, 78% RA with a standard “cup and cone” fracture feature and good “necking” showing good ductility.

5.5 MPa (800 psi) H_2, $1x10^{-4}$/sec, 42% RA with reduced “necking” and poorer ductility.

Figure 1: Loss of ductility in hydrogen shown on an API 5L X70 smooth tensile specimen. Reprinted with permission from Ref. [35].

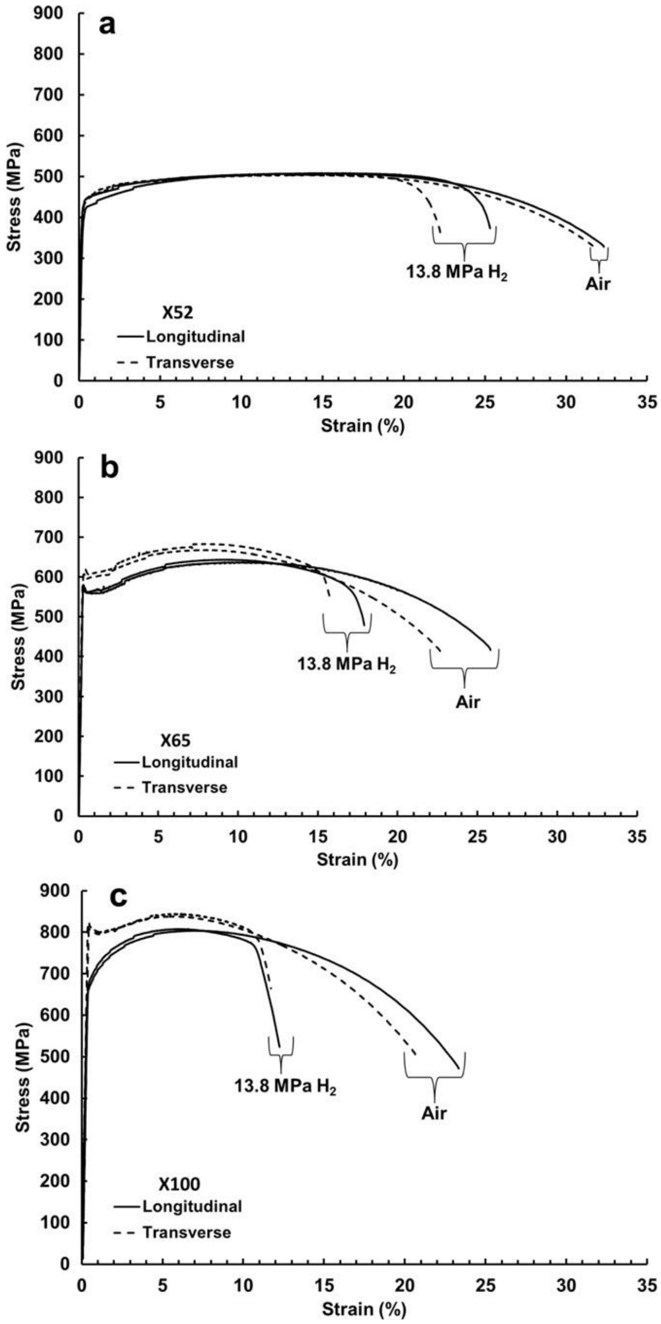


Figure 2: Stress-strain curves for three pipeline steel grades, API 5L X52, X65, X100. Reprinted with permission from Ref. [33].

Despite the apparent influence of hydrogen observed in tensile test results, such results can only provide a qualitative indication of the susceptibility to hydrogen embrittlement. Tensile test methods are referred to as screening tests for metals in gaseous hydrogen environments. They have the advantage of relatively easy execution and short duration, however, they may not always capture the important variables that are pertinent to a given application. Therefore, the results are used for comparative purposes to generate a pass/fail result or to rank materials by susceptibility to hydrogen embrittlement [37]. In practice no structure is free of (crack-like) defects, which may be introduced during manufacture, fabrication or subsequent service. Evaluation and control of the initiation and growth of such defects is of primordial importance. Therefore, to determine the criticality of a certain defect on the pipeline's safety a more quantitative method is required, i.e. fracture toughness testing, which involves the application of a dynamic load on a pre-cracked specimen in pressurized hydrogen gas at room temperature.

Fracture resistance properties of pipeline steels are significantly degraded in hydrogen gas when dynamic loading tests are performed [2,34,38–41]. The fracture resistance properties of pipeline steel are characterized by fracture toughness tests, where a fatigue precracked specimen is subjected to a slowly rising displacement or a slowly rising load (dynamic load). The critical stress intensity factor K_{Ic} is commonly used as a measure of fracture resistance. The elastic plastic J-integral method is frequently used to measure the fracture toughness of carbon steels according to ASTM E1820 [42], where a rising load is applied to the specimen. If a material exhibits stable crack growth the J value can be quantified as fracture toughness K_{Jc} [34]. Table 1 gives fracture toughness values in 6.9 MPa hydrogen gas for a range of pipeline steel grades. The fracture toughness in hydrogen was observed to be from 48 % to around 60% of the fracture toughness obtained in an air test, depending on the material [2,34]. It was reported that the reduced fracture toughness remains high with most values above 100 MPa.m$^{1/2}$ (K_{JC}), which is considered sufficiently high for most engineering applications [34]. Xu [34] recognized that the reduction of the fracture toughness does not simply depend on the strength alone, but also on the specific microstructure, and in particular on the micro-alloying elements. An even more pronounced effect was observed on the crack growth resistance, which gives an indication of the resistance to further crack growth upon reaching the critical fracture toughness. The slope of the tearing resistance curve (expressed as dJ/da) measured in hydrogen can be up to 90% lower than the values measured in air or in an inert gas in pipeline steels [2]. In other words, hydrogen results in a lower stress needed for crack propagation (reduced fracture toughness) and a reduced resistance for further crack propagation upon reaching crack growth (reduced dJ/da).

Table 1: Fracture toughness values (K_{JC}) obtained in literature of several pipeline steels in 6.9 MPa hydrogen gas. Reprinted with permission from Ref. [34]. YS represents yield strength.

Material	YS (MPa)	K_{JC} (H2) (MPa $\sqrt{m}$)	$\frac{K_{JC}(H_2)}{K_{JC}(Air)}$
A516	375	113*	68%
A106	297	81**	62%
X42	366	107*	60%
X52 Microalloyed	469	102**	63%
X60 Microalloyed	473	104*	73%
X70 Microalloyed	584	95*	48%
X80 Microalloyed	676	111**	54%

*Data reduction by ASTM E813.
**Data reduction by ASTM E1820.

Moreover, hydrogen can negatively affect a metal's resistance to crack growth under cyclic loading, which could be detrimental to the durability of the pipeline system. Understanding the fatigue behavior of steel pipelines in a hydrogen environment is crucial to determine the structural integrity of the pipeline infrastructure when transporting and storing hydrogen/natural gas mixtures [43–45]. Pipelines are subjected to cyclic loading resulting from daily pressure fluctuations during normal operation and from upset conditions where the pipeline experiences shutdowns resulting in a near-atmospheric pressure condition back up to full operating pressure. S-N curves indicate a significant reduction in fatigue life for low cycle fatigue (short life regime) when exposed to hydrogen gas, but is hardly reduced in high cycle fatigue (long life regime) [46–48]. However, compared to S-N curve determination, the fatigue crack growth rate (FCGR) test method is more appropriate for evaluation of the fatigue behavior of pipeline systems, as the method assumes a pre-existing crack. This is in line with the fact that welds contain small imperfections, which serve as crack initiation sites. FCGR tests relate the crack growth per cycle da/dN to the stress intensity range ΔK. Various recent studies show that the fatigue crack growth rate can increase by one or two orders of magnitude in gaseous hydrogen gas compared to air [2,41,45,46,49–57]. The exact influence is determined by many parameters such as the material, the load frequency, the stress ratio, hydrogen gas purity and pressure. Fig. 3 shows typical FCG results that are obtained from fatigue testing in a gaseous hydrogen environment [45]. FCG acceleration is not prominent in relatively low ΔK regime, where most of FCG life is spent. Therefore, for practical application, the acceleration rate in the low ΔK regime and the transition point are more important than focusing on the acceleration rate in larger ΔK regime.

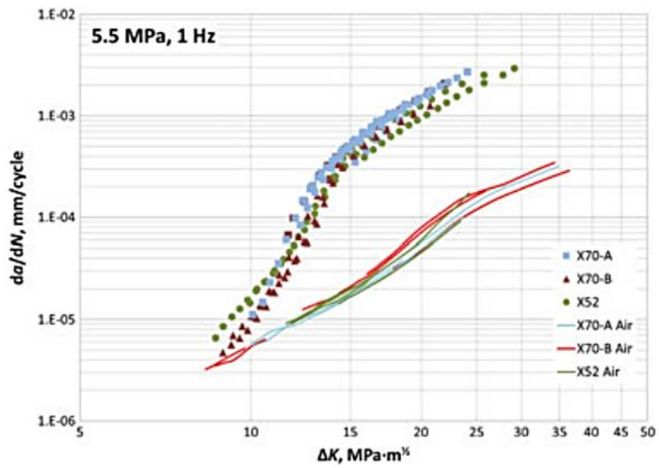
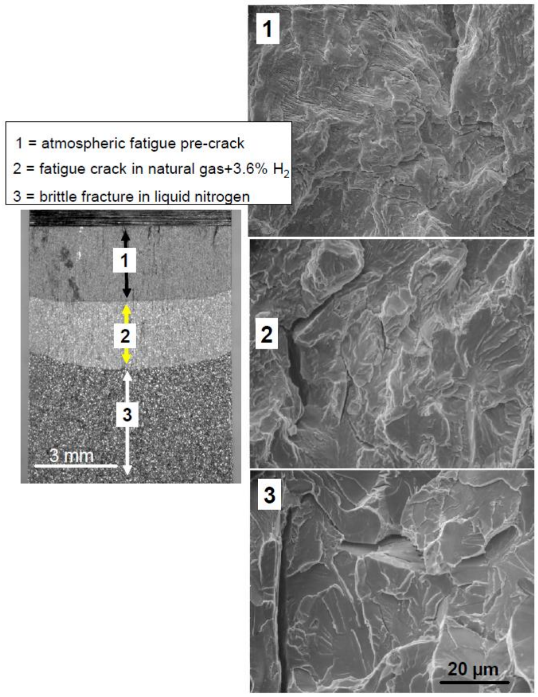


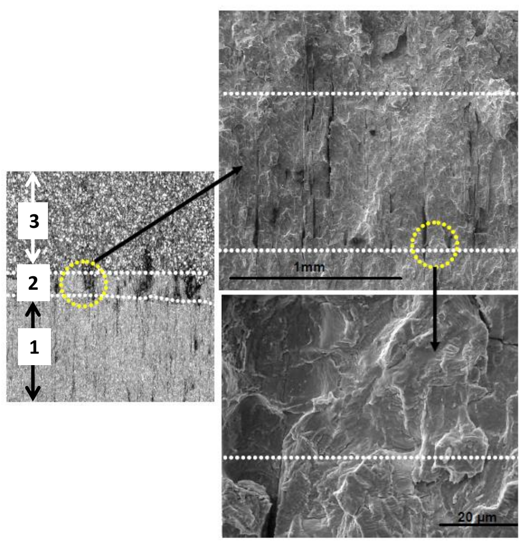
Figure 3: FCGR tests performed on API 5L X52 and API 5L X70 with a cyclic loading frequency of 1 Hz and both in air and in a hydrogen gas pressure of 5.5 MPa. Reprinted with permission from Ref. [45].

ΔK_{th} is a threshold value of ΔK below which a fatigue crack will propagate at negligibly low rates. At ΔK values just above this fatigue threshold, the crack growth rate per load cycle, *da/dN*, strongly depends on ΔK. A decrease in ΔK_{th} is disadvantageous, as a certain crack will propagate from a lower cyclic load level. Multiple sources state that the fatigue threshold ΔK_{th} is seemingly reduced by hydrogen, i.e 10-20 % relative to thresholds measured in air [34,47,58–63]. The hydrogen effect on FCGR is less pronounced than at higher ΔK values. Much of the discussion of hydrogen damage on early stage FCG revolves around the influence of a hydrogen atmosphere on crack closure. Ritchie and Suresh [60] showed that ΔK_{th} values and near-threshold FCGR may be influenced more by reductions in crack closure than by actual hydrogen embrittlement effects for Cr-Mo steels tested in low pressure gaseous hydrogen. It is argued that the observed reduced threshold in hydrogen compared to air can be explained by the dry, low oxygen environment which is provided by hydrogen gas [47]. The higher values of ΔK_{th} for specimens tested in air were attributed to a build-up of oxide on the crack surface due to high levels of plasticity-induced crack closure, which results in a decrease of the fatigue threshold. This theory is experimentally supported by tests showing ΔK_{th} values in He similar to those in H_2, values in moist hydrogen similar to those in air, and the similarity of fracture surfaces in air and hydrogen. At high stress ratios, the effect of hydrogen on the threshold diminishes, providing an additional argument for the reasoning above. Liaw et al. [64] found that the influence of hydrogen on near-threshold crack propagation rates in steels appears to depend on strength level. At low R values, lower-strength steels (<600 MPa) have lower ΔK_{th} values and faster crack growth rates in hydrogen gas, relative to ambient air, while in higher-strength steels they found that the trend is reversed, regardless of *R* values. The authors propose that the threshold fatigue crack propagation kinetics in high strength steels are controlled by the residual moisture content in gaseous environment, which controls the supply of atomic hydrogen required for embrittlement. Moreover, the reversibility of slip in the absence of oxides and adsorbates on the crack surface can be considered as an influencing factor, as lower near-threshold FCG rates were observed when testing in vacuum compared to air [65]. The dislocations moving forward during the loading part of the cycle can reverse during the following unloading part. The high reversibility of slip decreases the real increment of crack propagation in a whole cycle and results in a low crack propagation rate in vacuum. The effects of hydrogen on values of ΔK_{th} appear to vary among different environments, loading parameters, and materials, which makes it difficult to evaluate the role of hydrogen on near-threshold FCG. The above discussion shows the limited amount of data on this effect available at the moment. More tests that could support the proposed theories or add to the discussion would be of value in order to evaluate the role of hydrogen on this critical area of fatigue.

Microstructural analysis of fracture surfaces of FCGR test specimens shows large differences between the fatigue pre-crack in air and the hydrogen assisted fatigue crack (Fig. 4a) [44]. The fatigue pre-crack in air is ductile with striations, while the hydrogen fatigue crack exhibits brittle facets with an overall semi-ductile fracture surface. The final fracture in liquid nitrogen is 100% brittle. Fig. 4b demonstrates that fatigue cracks in 100% natural gas showed a comparable appearance as the fatigue pre-cracks in air.



(a)



(b)

Figure 4: (a) SEM micrographs of fracture surface of (1) fatigue pre-crack in air, (2) hydrogen assisted fatigue crack, and (3) brittle fracture in liquid nitrogen at the end of the test. Brittle facets are clearly visible in (2). **(b)** SEM micrographs of fracture surface of (1) fatigue pre-crack in air, (2) fatigue crack in 100 % natural gas, and (3) brittle fracture in liquid nitrogen at the end of the test [44].

Models can be devised which predict the FCG in hydrogen gas and, therefore, could estimate the remaining lifetime of an existing pipeline [31,66]. The models would be empirically based on the general trends observed from FCGR tests and should be calibrated for each particular material. Further, the pipeline lifetime can be estimated conservatively by using an upper bound of an FCGR database gathered from experiments, using different pipeline steel grades and loading conditions. This method is proposed in a recent modification of the ASME B31.12 code concerning the fatigue lifetime of pipelines transporting gaseous hydrogen [51,67]. Fig. 5 shows data of four pipeline steels (two X52 & two X70) with gas pressures up to 21 MPa in green. The equivalent fatigue crack growth rates in air are displayed in blue. The red line represents the approximate upper bound of the data and is implemented in the new B31.12 code.

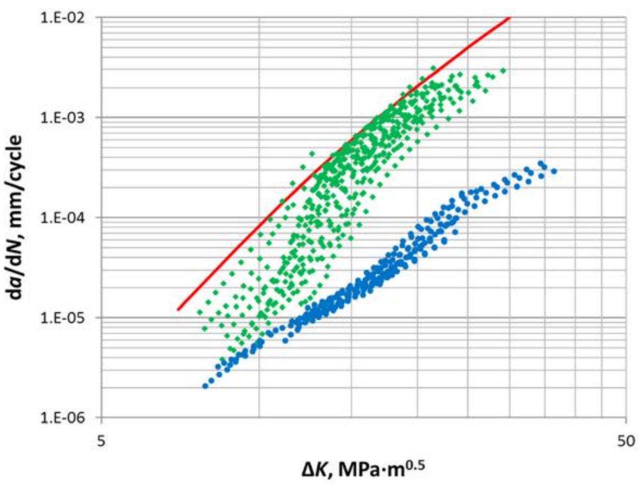


Figure 5: FCGR data from pipeline steels in hydrogen gas pressures up to 21 MPa, shown in green. The equivalent data in air is shown in blue and the red line can be seen as an approximate upper bound of the hydrogen crack growths. Reprinted with permission from Ref. [51].

Impact load testing, e.g. Charpy notch toughness testing, is widely used, especially in pipeline applications, as it gives a first indication of the fracture toughness. Multiple authors (e.g. [32,68]) have performed Charpy impact tests on hydrogen charged pipeline steel and found no significant change of the upper shelf impact energy, nor of the ductile to brittle transition temperature. This can be explained by the very high loading rates associated with impact tests, which do not allow for hydrogen to diffuse to the critical locations in order to have a harmful effect on the structural integrity. For this reason, impact tests such as the Charpy impact test cannot be used for assessing hydrogen embrittlement in pipeline steel. These results emphasize the importance of selecting the appropriate set of tests to address hydrogen effects on pipelines.

3. INFLUENCE OF MECHANICAL VARIABLES ON HYDROGEN ASSISTED FATIGUE CRACK GROWTH

3.1 Loading frequency

The effect of loading frequency on fatigue crack growth rate in hydrogen gas environments is of particular interest for the hydrogen embrittlement problem in pipelines and understanding the fatigue crack growth behavior at low frequencies is particularly relevant. Nevertheless, FCGR data at these low testing frequencies are rare (e.g. [51,69]) due to the extremely long time required for a fatigue test at this frequency.

In inert environments FCG is generally independent of the loading frequency. On the contrary, various studies indicate a negative correlation between frequency and FCGR in a hydrogen gas environment [44,45,51,53,54,66]. Hydrogen induced damage is time dependent as hydrogen needs to absorb and diffuse to the crack tip in order to deteriorate the material. As lowering the loading cycle frequency extends the exposure time of the material to the hydrogen gas environment, more hydrogen atoms are able to absorb and diffuse over a longer distance to the crack tip region within each loading cycle. Literature reports that a critical frequency f_c might exist, below which hydrogen saturation occurs and the measured FCGR is no longer affected by the load frequency [66]. Yu et al. [70] measured a critical frequency for API 5L X60 pipeline steel of 1.04 x10^{-3} Hz (σ_{ys}=414 MPa, T=303 K, υ= 0.31, partial volume of hydrogen= 2 x10^{-6} m^3/mol). To evaluate the fatigue behavior in pipelines transporting hydrogen gas, FCGR tests should either be performed at a frequency below f_c or the data should be corrected for using a higher test frequency to accelerate the test.

Slifka et al. [51] investigated the effect of loading frequency and hydrogen pressure for a vintage (1964) and a modern (2011) API 5L X52 steel and two modern API 5L X70 steels. Fig. 6 shows the crack growth rates per cycle for a fixed ΔK = 14 MPa√m, and loading frequencies of 1, 0.1 and 0.01 Hz. The bar charts show that the API 5L X52 vintage steel has the smallest sensitivity to loading frequency, while the others show some modest sensitivity. As such, the quantitative effect of loading frequency was observed to be material dependent, which could be explained by a difference in hydrogen diffusivity originating from the variations in microstructure. The authors note that for the four tested steels, the frequency effect can be conservatively taken into account in a phenomenological model using a power law with an exponent of -0.1. In other words, they suggest that testing at low frequencies (i.e. below 1 Hz) is not required, since the crack growth rate at a frequency f can be estimated from the crack growth rate at a certain tested frequency f_0 using following equation:

$$\left(\frac{da}{dN}\right)_f = \left(\frac{da}{dN}\right)_{f_0} * \left(\frac{f}{f_0}\right)^{-0.1} \quad \text{(Eq. 1)}$$

Since Eq. (1) was based on test results of four materials, its generic conservative nature is yet to be demonstrated. Other steel microstructures may have greater sensitivities to cyclic loading frequency, which could be evaluated by experimental testing [51]. Fig. 6 also shows that the hydrogen gas pressure has a far larger effect on the FCG than the load frequency [53].

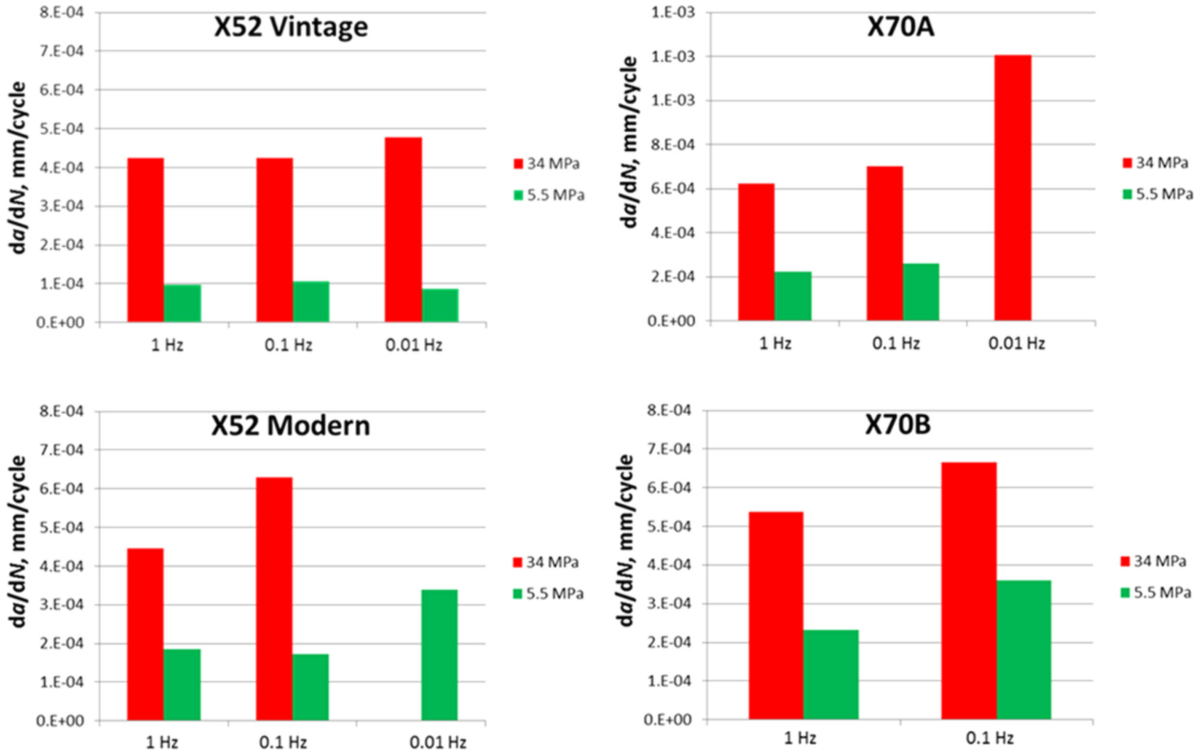


Figure 6: Crack growth per cycle at a fixed ΔK of 14MPa√m of four pipeline steels. The effect of both the hydrogen gas pressure and the loading frequency is evident. Note that at low frequency, not all tests are run due to time constraints. Reprinted with permission from Ref. [51].

3.2 Amplitude

Typically, load cycles with a fixed amplitude are applied during fatigue tests in laboratories. However, in industrial applications, it is often the case that a sequence of random load cycles are applied to the structure. This can have a large influence on the crack growth behavior. The phenomenon of crack growth retardation due to stress overloading is well known for classical cyclic loading in air. An overload (i.e. a peak load) causes a large plastic zone size, which induces a large plasticity-induced crack closure effect, effectively leading to retardation (i.e. a reduction in the FCGR). Although this mechanism is well established for steels in an inert environment, hydrogen diffusion could potentially influence the crack growth retardation. Xing et al. [71] argue that due to a larger ΔK and K_{max}, hydrogen accumulation in front of the crack tip will increase, eliminating the beneficial effect of overloading retardation. This idea was motivated by experimental tests using electrochemically charged specimens, where the crack growth rate increased rather than retarded for an increasing overload. Whereas overloading tends to cause crack growth retardation in air, underload in combination with so-called 'minor cycles' can lead to crack growth acceleration in air. Minor cycles followed by an underload might represent the pressure fluctuations during pipeline operation, i.e. near-static pressure conditions with occasional pressure drops (underload). Since the stress ratio R ($=K_{min}/K_{max}$) is large for minor fluctuations at a near-to-steady pressure level, the contribution to the fatigue crack growth rate of these minor cycles is small according to the logarithmic Paris' law using a fixed maximum stress intensity factor K_{max}. However, preliminary experiments have demonstrated that the crack growth of a specimen loaded with a certain amount of minor cycles followed by an underload can be significantly larger than the tests without any minor cycles, indicating a non-negligible contribution of the minor cycles [71]. The phenomenon of crack growth acceleration due to minor cycles and underload is potentially even more accelerated due to hydrogen. Yu et al. [72] observed a crack growth acceleration of factor 3 and 5, for an uncharged and an electrochemically hydrogen charged specimen (and equal other conditions), respectively. This acceleration factor depended heavily on the number of minor cycles introduced per underload cycle. There is a lack of data on the behavior of variable amplitude fatigue loading of pipeline steel in a gaseous hydrogen environment. Despite this, the existing data concerning electrochemically charged specimens indicate that the effect of variable amplitude fatigue loading cannot simply be neglected and is to be further investigated.

4. INFLUENCE OF MATERIAL CHARACTERISTICS ON HYDROGEN EMBRITTLEMENT SUSCEPTIBILITY

4.1 Strength

Literature provides contradictory statements concerning the effect of material strength on the sensitivity to hydrogen embrittlement. Numerous studies [36,47,73–76] state that steel grades with higher strengths exhibit a higher hydrogen embrittlement susceptibility (Fig. 7). The strength level is stated to be an overriding factor for the hydrogen embrittlement susceptibility, i.e. over microstructural effect and alloying elements for the studied low-alloy steels. Currently, standardized acceptance criteria for pipeline steels and welds in hydrogen applications (ASME B31.12) are based on a maximum allowable hardness of the pipeline material [67]. Such trend imposes structural designs for hydrogen gas service which not only specify a minimum yield strength, but also a maximum yield strength for managing hydrogen embrittlement [73]. However, this relation does not necessarily apply to fatigue crack growth resistance. Existing literature is somewhat contradictory, but generally fails to confirm a correlation between strength and hydrogen-assisted FCG for pipeline steels [31,77,78]. Microstructure and chemistry seem to dominate the FCG behavior in gaseous hydrogen over material strength [34,35,41,49,58,79–81]. It is, therefore, not correct that all increases in strength by definition lead to inferior hydrogen resistance [82,83]. Steels of similar strengths are often fabricated following different heat treatment schedules and thus exhibit different microstructures, which will not generally behave similar in a hydrogen gas environment.

This is a very interesting observation from an economic point of view, as it implies that higher strength pipeline steels are not necessarily more susceptible to hydrogen related degradation than pipeline steels of a lower grade when fatigue loads are concerned. Consequently, the strength correlation must be regarded as a rule of thumb. Established applicability criteria for pipeline steels and welds (ASME B31.12) exposed to gaseous hydrogen are essentially based upon hardness, thereby oversimplifying the microstructural effects on embrittlement susceptibility and introducing unnecessary conservatism in fitness-for-service assessment of the existing network. Nevertheless, codes and standards are regularly being updated in order to better take into account the repurposing of natural gas pipelines to hydrogen gas transportation (ASME B31.12 and code case KD10). It is vital to further understand the hydrogen embrittlement resistance in thermo-mechanically processed microstructures in order to help guide future microstructural design and alloy application for pipelines in hydrogen gas environments.

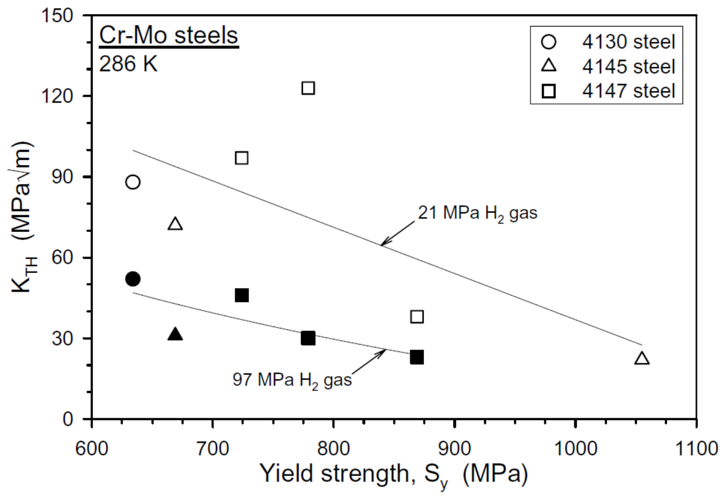


Figure 7: Effect of yield strength on the threshold stress intensity factor (K_{TH}) for hydrogen-assisted fracture for low alloy steels [75].

4.2 Microstructure

As introduced above, the microstructure strongly influences the hydrogen susceptibility of a material. Each microstructural component, i.e. phases and microstructural defects, exhibits a different behavior when exposed to hydrogen, adding to the complexity of hydrogen related studies. Generally, absorbed hydrogen atoms diffuse into the steel and are trapped at reversible and/or irreversible trap sites, such as grain boundaries, dislocations and other metallurgical defects, such as non-metallic inclusions, precipitates and bands of hard microstructures [74,84]. The trapping characteristics, i.e. nature and morphology of traps, of microstructural features will strongly influence the hydrogen embrittlement susceptibility of materials, as the presence of traps strongly affects the kinetics of hydrogen transport. Reversible trapping and related diffusible hydrogen are regularly considered as the main culprits in hydrogen assisted degradation phenomena [85–90]. There are two main microstructures in today's pipeline steels. The majority of pipeline steels are used in the as-hot-formed or normalized conditions. These conditions deliver a microstructure consisting of a mixture of pearlite and ferrite for the lower grade pipeline steels (≤ API 5L X70). Pipeline steel grades with a minimum yield strength of 483 MPa (API 5L X70 grade and above) rather consist of a microstructure of ferrite/bainite or ferrite/acicular ferrite [30]. Smaller volume fractions of micro-constituents such as micro-alloy-based precipitates and martensite/austenite islands can also be present [91,92]. Angus [93] found that grades with acicular ferrite microstructures exhibit higher diffusible hydrogen concentrations than grades with a ferrite/pearlite microstructure, making them more sensitive to hydrogen induced degradation. This result may be attributed to differences in dislocation density or low and high angle grain boundary surface area. Many researchers found that higher amounts of cold work (with accompanied increase in dislocation density) increase reversible hydrogen trapping [93–96].

Pipeline steels have evolved considerably in the past 60 years. Commercially available high-strength low alloy steels that combine strength and weldability along with required fracture toughness have been developed. The low carbon steels used for building the transmission pipeline grids have been developed with increasingly higher mechanical strength. The steel API 5L X42 was used in the 1960's, while today operators implement API 5L grades up to X80 (notwithstanding a limited number of projects with even higher grades). However, the higher the yield strength, the lower the resistance to crack growth is often considered to be. This risk is well managed for natural gas pipeline applications, but should also be evaluated for hydrogen. Moreover, over the years grades evolved within their category in microstructure and steel cleanliness. Hence, the chemical compositions, microstructures and mechanical properties vary between pipeline grades, but even within the same pipeline grade manufactured by different procedures (depending amongst others on the production year). For instance, an early type X60 can strongly differ in properties compared to a modern implemented X60 pipeline steel. This adds to the complexity of comparing different mechanical test results performed on different steel grades, as the microstructure and chemical composition should also be taken into account to assure a correct interpretation of the results. The existing pipeline system is constructed out of an enormous variety of materials as over the years different types of pipeline steel grades and grades produced following different production processes were implemented into the network. The complexity of making a general assessment of the hydrogen embrittlement resistance of the existing pipeline network becomes clear. Proper steel selection for hydrogen pipelines and evaluation of the hydrogen sensitivity of the current pipeline system requires an understanding of the relationship between microstructure and hydrogen assisted fatigue crack growth [97,98].

4.3 Pipeline welds

Hydrogen related cracking of welds is recognized to be a vital issue, as the reliability and lifetime of many structures is compromised by the presence of welds [99]. Pipeline welds may exhibit critical features compared to base material, such as the presence of stress concentrations due to geometrical imperfections, weld flaws, likelihood of poor coating conditions, critical microstructure and residual stresses. Welds in pipelines are typically the region in/near which most defects can be found, which render them sensitive to fatigue crack initiation [100]. Sharp cracks are considered the most dangerous [101]. It may be possible that flaws in the current pipeline network, which were accepted by workmanship rules, yield an unacceptable fatigue lifetime in the presence of hydrogen gas.

Many weld types and welding conditions are implemented in practice, generating welds with varying microstructures, residual stresses across welds and weld hardness levels. This makes the qualification of welds for hydrogen service challenging. The standard ASME B31.12 provides conservative acceptance criteria for welds based on the Vickers hardness [67]. Furthermore, many inconsistencies exist in literature due to the fact that the orientation and location of notches and pre-cracks strongly influence mechanical test results. Commonly, tensile and fracture toughness testing indicates that welds and heat affected zones (HAZs) of carbon steels show both higher and lower hydrogen embrittlement resistance compared to their base metals in high pressure hydrogen gas [2,100–102]. The limited research on fatigue crack growth rates of weldments in hydrogen is also contradictory and studies can be found both where the weld is more critical [44] or less critical [103–105] to hydrogen assisted fatigue crack growth than the base metal. These results motivate additional research concerning the relation between hydrogen assisted fatigue crack growth, the welding process and parameters, and the various microstructures in the weld and its HAZ.

Carbon equivalent (CE) formulae are used to predict the susceptibility of low strength carbon steels to hydrogen embrittlement based on their chemical composition. Although various expressions exist for CE, it is very commonly calculated as follows (numbers being expressed as weight percentages) [106]:

$$CE = C + Mn/6 + ((Cr + Mo + V)/5) + ((Ni + Cu)/15) \quad \text{(Eq. 2)}$$

In general, the higher the value of CE, the more susceptible the steel is considered to be to hydrogen embrittlement. Steel composition limits for acceptable hydrogen embrittlement include sulfur <0.01 wt%, phosphorus <0.015 wt% and a CE<0.35 [73]. The CE specification is designed to avoid the formation of untempered martensite, which is the most susceptible microstructure to hydrogen embrittlement [25], during welding.

5. INFLUENCE OF ENVIRONMENTAL CONDITIONS ON HYDROGEN EMBRITTLEMENT SUSCEPTIBILITY

5.1 Hydrogen gas pressure

Sievert's law states that the hydrogen concentration in a metal lattice is proportional to the square root of the hydrogen fugacity. For gaseous environments at low hydrogen pressures, the hydrogen gas can be considered as an ideal gas and the fugacity may be replaced by the pressure p. The sensitivity to hydrogen embrittlement of pipeline steels is, as such, dependent on the hydrogen gas pressure. As the amount of atomic hydrogen available per unit volume increases, the localized hydrogen embrittlement effects at the tip of a growing crack are increased [73,107]. Moreover, operating a pipeline at a higher gas pressure does also create a higher stress level in the pipe, which also influences the hydrogen embrittlement sensitivity. Regrettably, rapid decay of a steel's ductility and fracture toughness with increasing hydrogen pressure was reported. Nevertheless, saturation occurs for high gas pressures (Fig. 8 and 9) [2,33,34,75,76,108].

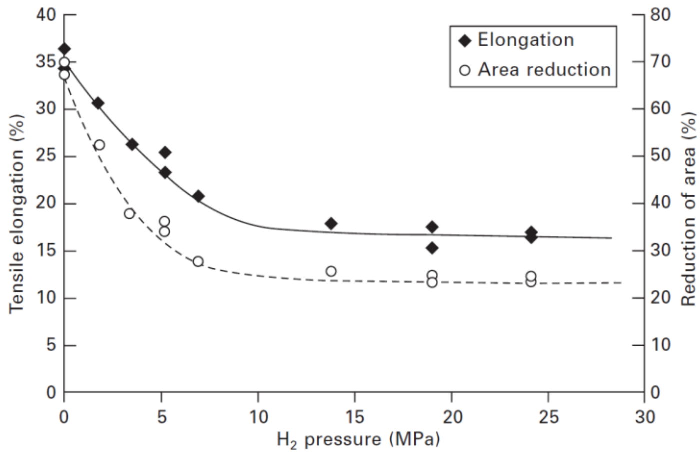


Figure 8: Dependence of ductility on hydrogen gas pressure for an ASTM A-106 Grade B steel. Reprinted with permission from Ref. [34].

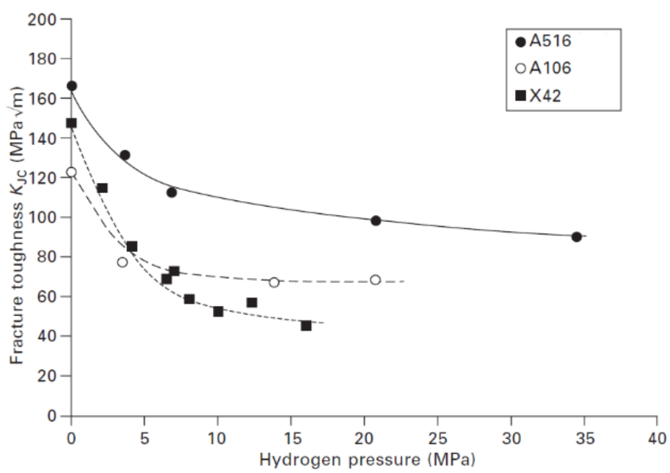


Figure 9: Effect of hydrogen gas pressure on the fracture toughness K_{JC} of three steels: A516, API 5L Grade B and X42 steel. Reprinted with permission from Ref. [34].

Fig. 10 shows that the trap occupancy is a strong function of pressure over a limited pressure range and exhibits a plateau behavior as the traps saturate. This trap saturation could possibly explain the mechanical-property plateau at higher pressure. The pressure at the onset of the plateau may be different depending on the material characteristics and specific trap site that governs fracture [36]. The pressure dependence is of relevance as pipelines in the existing natural gas infrastructure are operated at varying pressures and mixtures of hydrogen/natural gas can, moreover, be implemented with varying hydrogen partial pressures. Moreover, future pipeline system transporting pure hydrogen gas should transport a volume of hydrogen three times that of natural gas to satisfy a similar energy demand, as the heat of combustion of hydrogen is only 0.32 times that of methane [97].

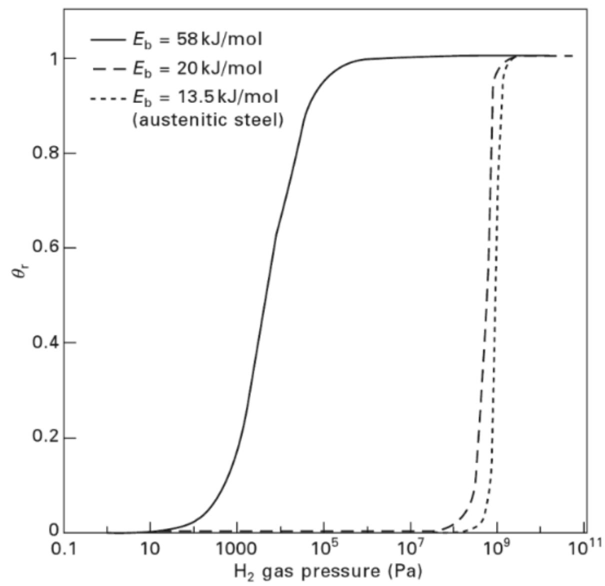


Figure 10: Fractional trap site occupancy (θ_r) plotted as a function of hydrogen gas pressure for two binding energies assuming properties of ferritic steel (58 and 20 kJ/mol) and 13.5 kJ/mol assuming properties of austenitic stainless steel. Reprinted with permission from Ref. [36].

An increase of FCGR with rising hydrogen (partial) gas pressure is observed (Fig. 11) [48,52]. Nonetheless, the considered ΔK ranges are not applicable to pipeline operations or if they are, it is very near the end of life. More research is required at lower ΔK to confirm if these trends persist. Moreover, other studies indicated that the actual extent of the pressure dependence of FCG varies with the microstructure of the material [44,51,53,77] and ΔK [57,109,110]. These dependencies make it challenging to determine a general limiting hydrogen gas pressure value for the overall pipeline system. Rather, it is advised to perform a case by case evaluation of the typical steel grades found in the system under investigation and test them adequately. As fatigue testing is very time-consuming and labor-intensive, the importance of development of an adequate screening methodology based on relevant hydrogen characteristics can be of interest.

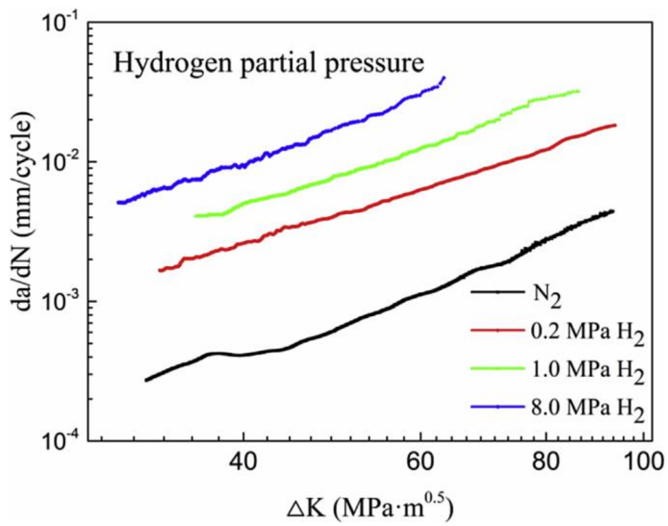


Figure 11: FCGR of X80 under different hydrogen partial pressures in hydrogen/natural gas mixtures at R = 0.1 and f = 1 Hz. The total pressure of the test environment was 12.0 MPa. Reprinted with permission from Ref. [48].

5.2 Temperature

Temperature affects many aspects of hydrogen interaction with metals including surface reactions, solubility, diffusivity, trapping, etc. Xing et al. [111] stated that temperature is a determining factor in bulk hydrogen concentration and diffusivity and, therefore, also determines the level of hydrogen induced degradation. For ferritic steels, hydrogen embrittlement is reported to be most severe between 200 and 300 K [76,112]. Carbon and low-alloy steels exhibit less severe hydrogen embrittlement as temperature increases (Fig. 12) [73]. Frandsen and Marcus [112] found that FCGR were maximum at 273 K. The influence of temperature can be explained based on the hydrogen trapping model, in which hydrogen is considered to diffuse through the material or be trapped with a certain binding energy at microstructural constituents and defects in the material. At temperatures lower than room temperature, the diffusivity of hydrogen is too sluggish to substantially accumulate at traps and critical regions. At high temperatures, hydrogen mobility is strongly enhanced and trapping is diminished and detrapping facilitated [107,113,114]. Xing et al. [111] also stated that elevating the temperature facilitates hydrogen movement and increases the surface hydrogen concentration, but restricts hydrogen accumulation near defects.

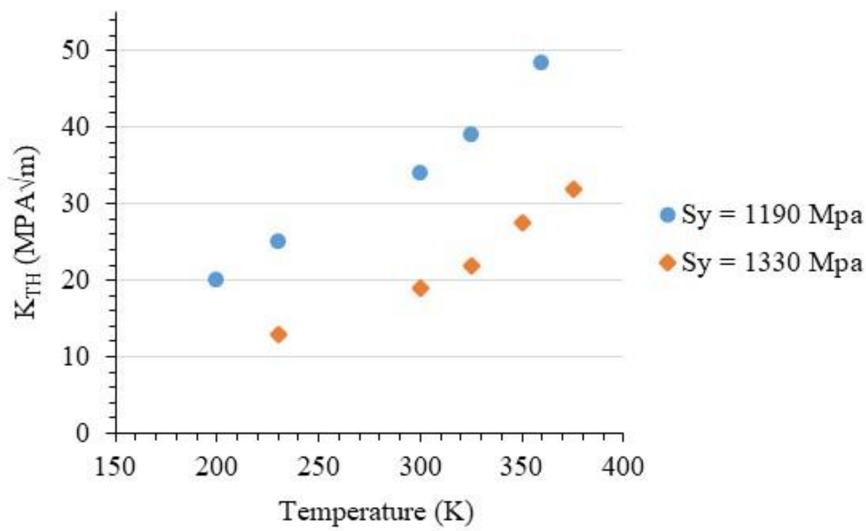


Figure 12: Effect of temperature on the threshold stress intensity factor (K_{TH}) for hydrogen-assisted fracture of 4130 low-alloy steel in 0.08 MPa H_2 gas. Adapted from [73].

5.3 Gas impurties

A very important question arises: “Can the negative effects on the structural integrity of pipeline systems caused by gaseous hydrogen be limited or avoided, both in new and existing installations?”. Addition of specific chemical gas components to a mixture of natural and hydrogen gas shows a lot of potential. Hydrogen gas purity strongly influences the uptake of gaseous hydrogen in a material. As such, depending on the specific gas species, impurities may increase [115], may have no effect on or may reduce the severity of hydrogen induced degradation as measured from tensile fracture properties, fracture threshold and fatigue crack growth rates [116]. Fig. 13 shows the effect of impurities on the FCG behaviour of a 2.25Cr-1Mo low alloy steel, relative to its behavior in pure hydrogen. Studies have been carried out to search for impurities which could potentially act as inhibitors for the deleterious effects of hydrogen gas on the mechanical properties of metals by impeding the surface reactions associated with hydrogen uptake into the metal [117]. Gases such as oxygen, acetylene, carbon monoxide, and nitrous oxide showed a dominant inhibiting effect when in combination with hydrogen, while methane and carbon dioxide had only a small effect on crack propagation when added to hydrogen [40,69,112,118]. A promising inhibitor is considered to be oxygen [40,119]. Fig.

14 illustrates the effect of oxygen addition on the hydrogen embrittlement through fracture toughness tests [40] in gaseous hydrogen environments. Increasing the oxygen concentration leads to measured properties in hydrogen gas that systematically approach those obtained in air or an inert environment. Generally, the absolute partial pressure of the impurity species is thought to be the variable responsible for the degree of inhibition. Therefore, impurity concentration becomes increasingly important as the hydrogen gas test pressure increases [118]. However, even though beneficial short-term inhibitory effects were obtained through gas impurity addition, the longer term benefits of impurities have so far not been studied. Further investigation of impurities and their long-term working could be of great value.

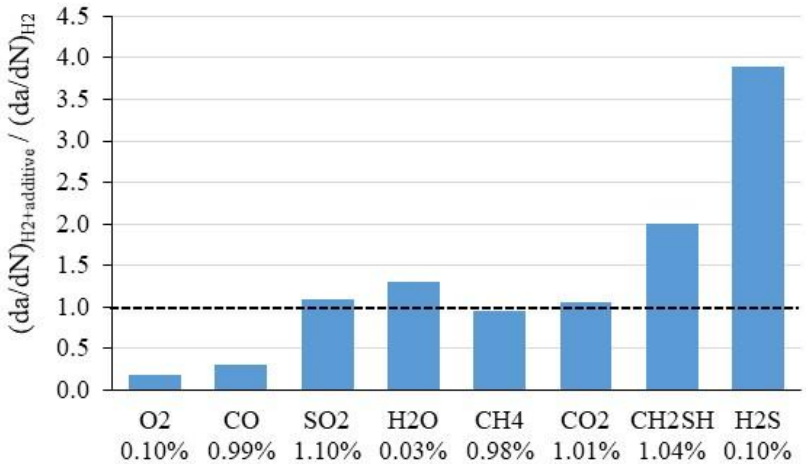


Figure 13: Ratio of fatigue crack growth rate in hydrogen gas with additives to fatigue crack growth rate in pure hydrogen gas at fixed stress-intensity factor range for 2.25Cr-1Mo low alloy steel. (S_y = 430 MPa, 1.1 MPa H_2 gas, ΔK = 24 MPa√m, frequency= 5 Hz, R= 0.1, T = 293 K) Adapted from [73].

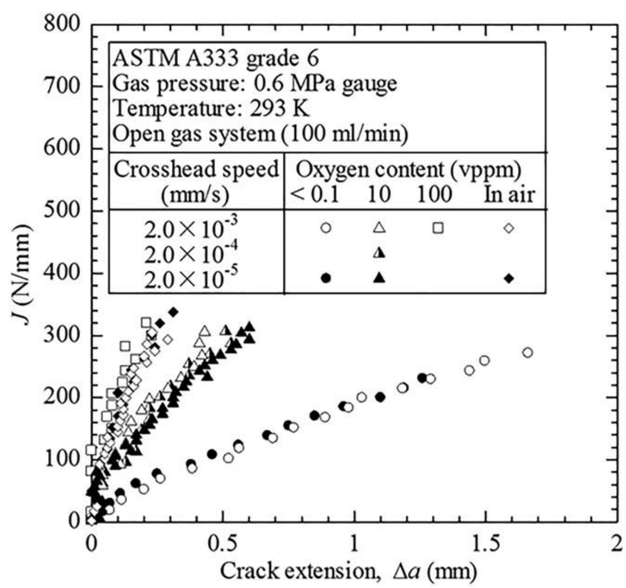


Figure 14: Crack resistance curves representing the potential inhibitory effect of oxygen on hydrogen-induced fracture. Rising displacement conditions are employed, together with 0.6 MPa hydrogen gas and different oxygen impurities, ranging from 0.1 vppm to 100 vppm. Reprinted with permission from Ref. [40].

6. CONCLUSIONS

The ductility and fracture toughness of pipeline steels is significantly degraded in hydrogen gas. Even more pronounced effects are observed on the crack growth resistance for quasi-static loads, as values measured in hydrogen can be up to 90% lower than the values measured in an inert environment. Various studies show that an increased fatigue crack growth rate of one or two orders of magnitude higher than in air can occur. The exact influence is affected by many parameters such as the material chemical composition and microstructure, the load frequency, the hydrogen gas pressure and gas purity. A method to conservatively estimate the lifetime of a pipeline system is to use an upper bound of FCGR data gathered from experiments, using different pipeline steel grades and loading conditions. This method is proposed in a new modification of the ASME B31.12 code concerning the fatigue lifetime of pipelines transporting gaseous hydrogen. Impact tests such as the Charpy impact test are not adequate for evaluating hydrogen embrittlement in pipeline steel.

Numerous studies state that the most technologically important trend for structural metals is that susceptibility to hydrogen embrittlement increases as the material strength increases. However, other authors conclude that the FCG response in a hydrogen environment is determined by the pipeline steel and its specific microstructural characteristics, rather than its strength grade. Different microstructural features all interact with hydrogen in a different way, adding to the complexity of hydrogen related studies of mechanical performance. Moreover, over the years different types of pipeline steel grades and grades produced following different production processes were implemented into the network, making it hard or impossible to have a clear overview of the risks of hydrogen assisted mechanical degradation in various existing pipeline systems. Understanding the hydrogen degradation resistance in thermo-mechanically processed microstructures is essential to help guide future microstructural design and alloy application for pipelines in hydrogen gas environments. Particular attention is to be given to welded connections, given their variety of microstructures, the potential presence of weld flaws and the presence of stress contributors additional to the nominally applied stress (due to geometry or residual stress). Literature concerning the hydrogen degradation resistance of welded connections reveals inconsistencies that impede the formulation of general conclusions. As a final important note, the addition of certain gaseous impurities, such as oxygen and carbon monoxide, to hydrogen/natural gas mixtures shows a lot of potential in inhibiting hydrogen assisted degradation of pipeline steels and welds.

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