Qualification of High-Strength Linepipes for Hydrogen Transportation based on ASME B31.12 Code



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Abstract

A number of CPW manufactured HFW and SAWL pipes were tested for fracture toughness properties in high pressure 100% hydrogen environment. All tests were performed in RINA laboratory, following a developed test procedure based on code ASME B31.12 Option B (qualification of the material threshold stress intensity factor KIH). Testing involved API 5L grades of quality X60M to X70M, with a hydrogen test pressure of 80bar and varying applied stress intensity factors 110-145 MPa·√m.

Following a test exposure of 1000h, all parent material, weld and HAZ specimens presented an excellent resistance to hydrogen embrittlement showing no measurable crack propagation from the fatigue pre-crack front. Based on the results, a KIH value of 55 MPa·√m and above was established in all cases, fulfilling the minimum qualification criteria of ASME B31.12 Option B

1. INTRODUCTION

Hydrogen is the most environmentally friendly carrier of energy: when consumed it solely emits water. Energy carrier means that its potential role has similarities with that of electricity. Both hydrogen and electricity can be produced by means of various energy sources and technologies. Both are versatile and can be used in many different applications. No greenhouse gases, particulates, sulfur oxides or ground level ozone are produced from the use of either hydrogen or electricity [1].

Conversely, hydrogen can be produced in an environmentally sustainable way by using only water and energy. This excellent energy solution requires however currently costly electrolysis equipment and is accompanied by a substantial energy loss during the extraction process. Nevertheless, also under this aspect, R&D efforts are producing important results with more efficient and cost effective electrolyzers available in the near future [2].

Consequently, hydrogen is currently enjoying unprecedented political and business momentum, with the number of policies and projects around the world expanding rapidly; in July 2020, EU Commission adopted a new dedicated strategy on hydrogen in Europe: the strategy explores actions to support the production and use of clean hydrogen, focusing in particular on the mainstreaming of renewable hydrogen. The strategy highlights a condition for a widespread use of hydrogen as an energy carrier in the EU is the availability of energy infrastructure for connecting supply and demand, and this can be done in a cost effective way via pipeline. Hydrogen offers ways to decarbonize a range of sectors, as well as helps improve air quality and strengthen energy security. Although hydrogen can be produced from a wide variety of fuels, its greatest potential lies in assisting with variable output from renewables, like solar photovoltaics and wind, whose availability is not always well matched with demand. As a result, hydrogen is one of the leading options for long term storing of converted electricity.

The production of hydrogen from renewables can be achieved at lower cost in regions with abundant solar and wind resources. For large volumes and long distances, transportation via pipelines to large energy consumers is the most financially attractive alternative [3] [4]. Additionally, blending hydrogen into natural gas has been proposed as a mean of delivering pure hydrogen to markets, by using separation and purification technologies downstream in order to extract hydrogen from the natural gas-H2 blend close to the point of end use [5]. Blending hydrogen would provide a boost to hydrogen supply technologies without incurring the investment costs and risks of developing new hydrogen transmission and distribution infrastructure. [1]. The recent interest in developing a hydrogen-based energy economy resulted to the need for hydrogen compatible materials to the forefront, especially dealing with the effect of Hydrogen Embrittlement (HE). HE is the degradation of the mechanical properties of a metal, most frequently manifested by the emergence of low energy fracture mechanism when exposed to hydrogen.

The phenomenon of HE has been recognized since 1875 [6] and has been extensively studied. While the fundamental mechanism behind HE is a matter of continuous investigation from the scientific community, the amount of data on the effect of hydrogen on mechanical properties of different metals and alloys made the standardization of appropriate materials possible, for a number of applications involving gaseous and liquid hydrogen systems. [7].

2. STATUS OF CARBON STEEL HYDROGEN LINEPIPES

The transport of gaseous hydrogen through pipelines has been realized by use of mild carbon steel for almost a century and it is estimated that there are over 4,500 km of hydrogen linepipes in operation worldwide [8]. Typically, hydrogen linepipes are designed to transport gas over only short distances, from the production facility to the end user. Many such applications operate with a very good safety record but at maximum pressures which are considerably less than the ones that would be required for long-distance pipeline transmission of hydrogen [9]. In addition, typical pipeline size is 300mm or less, manufactured with X52 or lower strength steels [10] and in comparison to natural gas, H2 pipelines normally operate at relatively conservative conditions.

However, owing to the low volumetric energy density of hydrogen (0.0108 MJ/L) in comparison to natural gas (0.0364 MJ/L) and the forecasted expansive utilization of renewable energy sources mentioned in section 1, it will be necessary to transmit hydrogen at high pressures using large size pipelines in order to be financially competitive. The combination of high pressure and large size pipe demands the use of higher strength steels.

The advantages of specifying a higher grade line pipe for transportation of hydrogen or hydrogen-gas mixtures can be substantial: According to independent analysis [11], for a baseline scenario using a 24" HFW longitudinal pipe operating at 1,500psi (10.34MPa), the use of X70 material can result into cost savings up to 31% relative to the use of X52.

The amount of published results on the effect of hydrogen to the mechanical properties of higher grade API line pipe steels under high pressure is rapidly increasing and

results of systematic work have been presented by NIST and Sandia National Laboratories. According to published work [10] [12], a number of toughness tests on API carbon steels have shown that the absolute fracture toughness remained high under high pressure hydrogen conditions, even though it was lower than respective measurements in air or inert gas. In addition, a comprehensive testing program to determine fatigue crack growth rate of pipeline steels in pressurized hydrogen gas verified no change in FCGR (Fatigue Crack Growth Rate) with increasing yield strength up to X100 [13].

K. Xu [10], reviewed a number of published results for carbon steels up to X70 and 10.3MPa test pressure when tested under static loading condition and no subcritical crack extension was exhibited under various loading conditions. The same report presents also a number of rising load method fracture toughness KJC tests for micro alloyed steels up to X80 in 6.9MPa H2 where the measured fracture toughness was found above to be 95 MPa·m1/2 in all cases. San Marchi et al [12] [14] reported also fracture toughness values in the range of 80 to 100 MPa·m1/2 using a rising load test method in high pressure gaseous hydrogen (5.5 and 21MPa) for two X60 and X80 pipeline steels.

In comparison to plain carbon ferritic steels, API 5L steels of higher grade typically contain additional alloying elements, such as small quantities of niobium and titanium. These "microalloying" additions as well as processing by thermomechanical rolling provide a combination of elevated strength with excellent low temperature fracture toughness. In metallurgical terms, many modern higher grade API 5L steels utilise a ferrite/bainite or ferrite/acicular ferrite microstructure to attain these properties. The lower pearlite volume fraction of these steels is considered to provide enhanced hydrogen resistance, an effect obtained by reducing the amount of H2-trapping sites i.e. the interfaces between microstructural constituents [15] [16].

3. APPLICABLE STANDARDS AND PRACTICES

There is a limited number of standards that can be used for material qualification for pipeline gaseous hydrogen transportation:

- International European standardization bodies are working in revising EN 1594, EN 16348 and EN 12732 in order to consider H2 and H2NG mixtures also.
- EIGA (European Industrial Gases Association) published a document (IGC Doc 121/14) which recommends maximum steel grade to be used and suggests testing to be carried out, but with not specific instructions on how to qualify the material.
- ASME B31.12 is a US standard for material qualification for use with H2 and H2NG mixtures. Two basic approaches are adopted: Design Option A and B, that are

briefly described hereinafter.

It is worth highlighting EIGA report makes specific suggestions to limit the effects of hydrogen embrittlement on materials, such as appropriate material classes, compositional and strength limits, and suggests appropriate testing methods, but is a recommended practice and not a standard. At the same time, new ISO standards under revision are expected to follow the ASME B31.12 approach for the material qualification of pipelines for high pressure gaseous hydrogen transportation; ASME B31.12 is now the most used standard for material qualification and can be expected to be the reference one also in the next future.

4. ASME B31.12 CODE

The ASME B31.12 Hydrogen Piping and Pipeline Code [17], has been initially published in 2008, in order to deal with design, construction, operation, and maintenance requirements for piping, pipelines, and distribution systems in hydrogen service. The B31.12 committee has developed two design methods that can be considered in conjunction with steel/piping specifications (i.e. API 5L PSL2) and acceptable manufacturing routes for welded pipes (HFW, SAWL or SAWH) [15].

The first (Option A) is prescriptive and similar to design processes contained in ASME B31.8 Natural Gas Pipeline Code. It considers the use of lower basic design factors, F, and a material performance derating factor, Hf, derived from pressure and tensile strength relationships.

The second (Option B) is performance based, using a fracture mechanics approach (on the basis of ASME Section VIII, Div. 3 - Alternative Rules for Construction of High Pressure Vessels). The qualification of the pipeline materials is performed by use of fracture mechanics and crack propagation testing that empowers the use of enhanced design factors and withdraws the limitations on pressure due to the use of the Hf derating factor.

In regards to the second design method, the code introduces additional requirements for pipe material, related to lower Phosphorus content (<=0.015%) and consideration of API 5L Annex G for CVN testing (Enhanced Ductile Fracture Propagation Properties). More specifically, the ASME B31.12 code requires that the threshold stress intensity factor for hydrogen-assisted cracking (denoted as KIH) should be measured according to ASME VIII [18] and ASTM E-1681 [19].

When designing a pipeline for hydrogen transportation, the benefits of compliance with ASME B31.12 Option B can be substantial. This is illustrated in Figure 1 for an API X6OM grade: the design factor for Option B can be 72% of the specified yield strength for all applicable pressures up to

20.7 MPa (3,000 psi). On the contrary, the same design factor for Option A is limited to a maximum yield strength percentage of 43,7% or even lower, due to additional limitations of the material performance (Hf[¬]) factor when the design pressure approaches 3,000 psi (20.7MPa).

The latest version of ASME B31.12, specifies for Option B that fracture toughness qualification testing is required to validate the minimum threshold stress intensity factor (KIH) at the design pressure and 100% H2 concentration. The test on the pipes should be performed at the base metal, weld metal and heat affected zone positions, on three heats of the pipe material. It is highlighted that the tests qualify also other materials with similar chemical composition and tensile properties (Yield and Tensile Strength) up to 5% higher than the qualified ones. Therefore, samples should be selected from the upper end of the tensile properties distribution. The KIH value that gualifies the material in accordance with ASME B31.12 Option B is 50ksi·in1/2 (or 55 MPa·m1/2) unless otherwise specified by design analysis. It should be noted that the latest version of the ASME B31.12 code has removed the requirement to perform specific FCGR testing for the qualification of a hydrogen line pipe and generic curves are provided, applicable for all carbon steels in gaseous hydrogen up to 20.7 MPa (3,000 psi) service pressure.

5. FRACTURE TOUGHNESS QUALIFICATION TESTING

Aimed at validating the performance characteristics of high grade API 5L pipes in pressurized hydrogen, CPW organized a number of fracture toughness qualification (KIH) tests under the ASME B31.12 code Option B scheme, including both High Frequency Welded (HFW) and Longitudinal Submerged Arc Welded (SAWL) pipes. All tested pipe material is presented in Table 1. As presented in Figure



Figure 1: Design pressure factors for X60M for Option B vs Option A in areas characterized as Location Class 1, Division 2

2, the selected pipe dimensions for the HFW pipe, belong to the upper diameter and thickness segment of the 26" mill's product range. All the tests were carried out at room temperature (around +15°C).

6. PROCEDURE FOR KIH TESTING

ASME-based hydrogen material tests were performed in RINA Consulting – Centro Sviluppo Materiali SpA, an acknowledged European Company specialized in the development of new materials and in the performance assessment of materials and equipment in new operating windows; with regard to the subject, RINA has specific skills and laboratories specialized to evaluate materials and components performance in presence of gaseous hydrogen up to 1,000bar external pressure.

Fracture toughness testing protocol in pure hydrogen gaseous environment was determined in terms of KIH for all notch positions in compliance with ASTM E1681 [19]



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Tensile properties Test Test Pipe OD WT YS Steel TS CVN Service Steel Grade Pressure Gas type Rto,5 at -10°C supplier Rm MPa MPa J MPa in. mm HFW Onshore 26' 15.9 L415 (X60M) 492 612 299 ArcelorMittal HFW Onshore 20" 19.1 L485 (X70M) 539 655 275 Bremen HFW Onshore 11.1 L415 (X60M) 498 627 219 26' 100% Offshore, DNV 450PD ArcelorMittal 8.0 Hz HFW 16" 19.1 524 605 265 Reeling (eq. X65MO) Fos-sur-Mer Voestalpine SAWL Onshore 32" 20.0 L485 (X70M) 496 614 284 Grobblech

Table 1: Overview of CPW pipes tested for fracture toughness (KIH) in pressurized hydrogen

according to the constant displacement configuration, with the additional prescriptions of ASME B31.12 [17] and ASME BPVC Section VIII, Division 3 [18] [20].

The procedure for KIH fracture toughness testing is presented schematically in Figure 3.

Samples are machined in bolt-load compact configuration in compliance with the prescriptions of ASME E1681 [19] for the Modified bolt-Load, Compact Specimen; H/W=0.486, where W/B is 2:1 (Figure 4). No pipe flattening was applied prior to sample machining and the largest possible thickness was obtained depending on pipe curvature. In any case, the request of having at least 85% of the pipe nominal thickness was always satisfied. The determination of the threshold stress intensity factor involves a specimen containing a machined notch, which is placed in base material and, for HFW pipes, in bond line or, for SAWL pipes in weld metal and Heat Affected Zone crossing the fusion line (Coarse Grain HAZ) at the maximum extent. This notch is extended by fatigue cracking under controlled conditions for maximum loading, especially for the final part of the crack growth. The fatigue precracked specimen is then placed in a glovebox filled with a nitrogen atmosphere, under very low oxygen and moisture levels as required per ASME code.

The specimen is then loaded by means of a bolt to the attainment of the target Crack Mouth Opening Displacement, established on the basis of the target stress intensity



Figure 3: Outline of KIH testing procedure



Figure 4: Compact tension specimens in RINA laboratory

KIAPP for plain strain conditions. According to the code, the applied KIAPP should be at least 1.6 times greater than the estimated KIH but not more than 180 ksi. \sqrt{in} (198 MPa· \sqrt{m}). After loading, the samples are put inside the test chamber which is sealed while still inside the glove box, preventing any contact of the loaded samples with atmosphere oxygen and moisture.

The test chamber is then charged with pure hydrogen gas at the target test pressure and maintained at this pressure for 1,000h. In this way, any fresh crack surface that is possibly generated by ductile tearing during bolt loading has never been exposed to oxygen or moisture and is hence prone to hydrogen permeation from the gaseous hydrogen environment. After the specified test period, the specimen is examined to assess whether the initial fatigue crack did or did not grow. The specimens are heat tinted and broken open in liquid nitrogen. The fracture surface is then examined by optical observation and scanning electron microscope. Measurements of the crack front extent are taken in five positions and the average crack growth in hydrogen is calculated.

7. FRACTURE TOUGHNESS KIH TEST RESULTS AND EVALUATION

The results of all validated fracture toughness KIH tests are summarized in Table 2. Four samples per material/notch were prepared in order to obtain at least three valid results per position. According to KD-1047 clause of ASME code [18] for the constant displacement method, if the average measured crack growth does not exceed 0.01 in. (0.25mm) KIH is equal to 50% of KIAPP. Taking this clause into consideration, the KIAPP initial stress was selected to be at least double of the minimum threshold stress intensity value required by the code of 55 MPa·√m.

No hydrogen crack growth was noticed at any specimen after visual and SEM examination at high resolution. In all cases also the SEM micrographs highlighted a dimpled fracture surface in front of the fatigue pre-crack, extending a few microns (Figure 5). Presence of this surface represents an evidence of a newly generated surface, formed as a consequence of the load application by the bolt and serving as a site for hydrogen permeation during the hydrogen 1,000h exposure.

Pipe	Test item code	Diameter (inch)	Thickness (mm)	Grade	Specimen type	Test gas	Test pressure	Test duration	Crack plane orientation	Notch position	Applied initial stress intensity (Kı₄₽₱) in MPa∙m ^{1/2}		Crack propagation after exposure	Min fracture toughness Kin (=½:Kiapp)	
							bar	hr			Min	Max		MPa· \sqrt{m}	
HFW	А	20	15.9	L415M (X60M)	Bolt- Ioad CT	100% H2	80	1000	T-L	BL	109.4	143.1	No	55-72	
										BM	109.9	144.6	No	55-72	
	в	20	19.05	L485M (X70M)	Bolt- Ioad CT	100% H ₂	80	1000	T-L	BL	120.7	121.4	No	60-61	
										BM	115.7	118.2	No	58-59	
	с	26	11.1	L415M (X60M)	Bolt- Ioad CT	100% H2	80	1000	T-L	BL	110.0	137.9	No	55-69	
										BM	110.2	138.5	No	55-69	
	D	16	19.1	450PD (X65MO)	Bolt- Ioad CT	100% H2	80	1000	T-L	BL	110.1	139.1	No	55-70	
										BM	110.1	124.7	No	55-62	
SAWL			20	L485M (X70M)	Bolt- Ioad CT	100% H2	80	1000	T-L	HAZ	110.3	138.3	No	55-69	
	Е	32								BM	110.2	138.8	No	55-69	
										WM	110.4	139.3	No	55-70	

Table 2: Results of fracture toughness ASME KIH testing



Figure 5: Visual and SEM examination of representative post-exposure examination results from the 26" x 15.9mm HFW test item

8. DISCUSSION

The excellent resistance of the tested pipes against hydrogen embrittlement was endorsed by the chemical analysis characteristics of the tested pipes (Table 3) as in all cases the steel quality was characterized by low carbon content and carbon equivalent (PCM) and high levels of cleanliness (very low P, S). In addition, the TMCP processed coils (or plates, for the case of the SAWL pipe) presented in all cases a fine polygonal or acicular ferrite microstructure with finely dispersed pearlite and no or minimal banding (Figure 6). Such characteristics in steel chemical composition and microstructure are in-line with the recommendations of the hydrogen linepipe code (Table 4). It has been documented that pipeline steels containing acicular ferrite microstructures present higher resistance to hydrogen damage compared to ferrite/pearlite microstructures due to reduced potential of hydrogen trapping sites at the interface between microstructural constituents [15] [21]. In addition,

a fine ferrite grain microstructure with minimal banding can reduce the mobility of hydrogen, lower the diffusion coefficient and eventually enhance resistance to hydrogen embrittlement [16]. Lower carbon microstructures reduce also the probability of having high strained martensitic phases in the pipeline steel which have also been evaluated to increase susceptibility to hydrogen damage [22]. The test results presented in the current report seem also to be consistent with existing other published work, where the measured results surpassed the minimum ASME B31.12 value of 55 MPa·√m.

9. CONCLUSIONS

The certification of pipes for the transportation of pure gaseous hydrogen or H2/NG gas mixtures without additional design pressure limitations can be achieved, on the basis of pipe material's fracture resistance properties qualification following design "Option B" requirements of



Figure 6: Representative micrographs of X70M HFW pipe on PM (left) and weld seam (right) presenting a fine polygonal ferrite microstructure. Etching: Nital 2%

Test item	c	Si	Mn	Р	S	Cr	Мо	Ni	AI	Cu	Nb	۷	Ti	N	Рсм	IIW
A (X60M)	0.05	0.28	1.57	0.007	≤ 0.001	0.02	0.01	0.03	0.04	0.01	0.04	-	0.01	0.004	0.15	0.33
B (X70M)	0.05	0.31	1.67	0.010	≤ 0.001	0.02	0.01	0.26	0.03	0.01	0.05	-	0.02	0.004	0.15	0.35
C (X60M)	0.05	0.29	1.58	0.012	≤ 0.001	0.02	0.01	0.03	0.04	0.01	0.05	-	0.02	0.003	0.15	0.33
D (X65MO)	0.07	0.21	1.35	0.013	≤ 0.001	0.04	0.02	0.03	0.03	0.02	0.03	-	0.01	0.004	0.15	0.31
E (X70M)	0.06	0.25	1.56	0.010	≤ 0.001	0.08	0.09	0.09	0.04	0.01	0.04	-	0.01	0.007	0.16	0.36
Limit*	≤ 0.12	≤ 0.45	**	≤ 0.025	≤ 0.01						***	**	**		≤0.25	≤0.43
* According on API 5L PSL2 ** Up to 1.70% depending on grade *** <u>► Nb</u> + V + TI ≤ 0.15%																

Table 3: Chemical analysis of tested pipes (% wt.)

Desired microstructure of polygonal and acicular ferrite TMCP made steel is recommended Phosphorus content ≤ 0.015% wt. Recommended Carbon content ≤ 0.07% wt. Recommended Carbon Fquivalent (Pcm) < 0.17% wt. Maximum UTS 110ksi (758MPa) Nb micro alloyed steel is recommended

Table 4: ASME B31.12 Option B ϖ Appendix G: Steel chemistry requirements and recommendations.

code ASME B31.12. The respective qualification procedure, among other requirements, require primarily the long-term exposure of artificially pre-cracked specimens under high pressure 100% H2 conditions. Following the above qualification scheme, Corinth Pipeworks is currently progressing with an extensive R&D program for fracture toughness testing of HFW, SAWL (longitudinal) and SAWH (helical) pipes in high pressure hydrogen. All tests are accomplished in RINA, an acknowledged external European Company, highly experienced in hydrogen testing and fracture mechanics.

According to the up-to-date test results for HFW and SAWL pipes in grades up to L485M/X70M, all tested specimens in base metal, weld and HAZ (where applicable) positions demonstrated high resistance against hydrogen-assisted crack growth and the measured values for the KIH fracture toughness property were always higher than the minimum required value of 55 MPa√m. Furthermore, the observed fracture mechanism does not pose any evidence of brittle or low-energy cracking phenomena. It has been therefore demonstrated that the requirements of the code for the pipe material are consistently feasible, thus certification of a higher grade line pipe for 100% hydrogen transportation using Option B can be provided. This certification can be the first step towards the efficient transportation of larger volumes of hydrogen through the steel pipeline network in the future.

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