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Large Diameter Carbon Steel Pipe from EUROPIPE for Hydrogen Gas Transport

Dr. Ing. Christoph Kalwa

Europipe GmbH, Muelheim, Germany

ABSTRACT:
Reducing greenhouse gas emission, results in setting up concepts with converting surplus renewable energy to hydrogen for storing parts of the energy for later use. For transporting this gas, pipeline operators intend to blend natural gas with hydrogen or transport pure hydrogen through their existing or to-be-built pipeline grid. Pipelines are considered the safest way to transport any gases, as this relies on a long track record over the last decades. Near future scenarios set the industry's focus more on smaller diameters for pure hydrogen pipelines, whilst large diameter pipe is more dedicated to the application for hydrogen blends. As hydrogen quantities are expected to increase, also large diameter pipes might be used later for transporting pure hydrogen and the industry is going to be prepared for this situation.

For this reason, EUROPIPE has set up a program to evaluate the material behavior in pure hydrogen atmospheres. The program comprises investigations in different steels with different grades, chemistries, und microstructures. The program is still running and many results are pending. The paper helps to classify hydrogen levels in the material in comparison to other hydrogen pick-up scenarios, as e.g. from sour service testing environments.

As ASME B31.12 sets standards for worldwide hydrogen pipeline design and required material properties, the program also focuses on fracture mechanical investigation with both static and cyclic tests to evaluate the toughness level and the crack propagation behavior of CMn-steels. This paper discusses the available results of this program and gives an overview about the influence of pressurized hydrogen on critical stress intensity and crack propagation under cyclic loading.

1. INTRODUCTION

The reduction of greenhouse gas results in the search for carbon neutral energy sources. A fundamental role is expected to be taken over by hydrogen produced by renewable energy or by processing natural gas where the carbon dioxide is captured or the process product is solid carbon; both to be stored. Hydrogen consumers from the chemical or steel industry, or from private households can use this gas for their processes or for mobility and heating, accordingly.

To transport hydrogen to the consumer is an important issue to put a focus on. Depending on the required quantity to be transported, different means are available from tanker transport to pipelines. For natural gas, pipelines have proven to be the safest and most economical means for transporting large quantities of gas in the recent decades und it is self-evident to transfer this also to transporting hydrogen.

Within the next ten years, the current infrastructure is planned to be used for blending natural gas with hydrogen. Due to initially small quantities, some pipelines mainly with sizes below 400 mm shall transport pure hydrogen. As hydrogen quantities are expected to increase in the next decades, pipeline operators plan to blend hydrogen in natural gas to considerable shares or transport pure hydrogen in pipelines, which are in EUROPIPE's production range. All of this makes the question about hydrogen-readiness relevant to EUROPIPE's pipes and leads to the conclusion for EUROPIPE to start investigations in hydrogen performance of its material to be prepared for future requirements.

Many papers published that hydrogen can have a detrimental effect on carbon steel [1]. The operation of such pipelines was considered safe when using a steel grade at the lower end as e.g. L360 with low usage factors [2]. In the past decades, hydrogen pipelines were designed in accordance with these principles and operator's experience is based on such pipelines. The use of the existing pipeline grid for transporting hydrogen needs a detailed knowledge about the material's performance in hydrogen. Especially the toughness properties are of interest.

As of now, diameters up to 300 mm are selected to transport pure hydrogen in pilot pipeline projects to demonstrate the suitability of vintage and recently installed pipes. Larger diameter pipelines, in the diameter range of EUROPIPE's production, are more in the focus of blending hydrogen to natural gas. The study NATURALHY from 2009 stated that blends up to 20 % hydrogen does not have a significant de-rating effect on carbon steel [3]. However, material behavior in hydrogen depends more on the resulting hydrogen partial pressure than on the hydrogen percentage in the gas. This is different to the gas delivered to the consumers, where combustion properties are more linked to hydrogen percentage in the gas than to hydrogen partial pressure. Nevertheless, all of these considerations need the information about material's performance in hydrogen, which makes testing necessary, as no data for hydrogen performance for the current pipeline material in use are available.

2. TESTING IN HYDROGEN

All mechanical testing methods simulate a certain mechanical situation for which the material gives a response. Many methods are conducted until the mechanical load leads to failure of the material, which gives information of material's reserves and safety margins. Some simulate a certain state and the result of such testing is whether the material is able to cope with this state or not. Many testing methods produce a quantitative output that is integrated in design or safety calculations, some testing outputs allow ranking material behavior qualitatively, and some testing methods compare materials performance in a desired test situation with a standard situation.

Testing in hydrogen uses all of such methods. Conducting tests in hydrogen needs to consider that the diffusion process of atomic hydrogen controls material property alteration. This results in slow testing for giving hydrogen atoms time to saturate in the material and to diffuse to stress concentrators or to preferred microstructures.

A basic method is Slow Strain Rate testing where tensile specimens are slowly loaded until failure in pressurized hydrogen atmosphere and compared with the same testing in air or nitrogen [4]. In most cases, CMn-steels do not show any difference between behavior in hydrogen and air/nitrogen in the range where specimens strain uniformly (i.e. strains below uniform elongation). The materials differentiate in the range when specimens strain locally and start necking where the deformation process is more toughness controlled. Results of this testing is more qualitative as the behavior in the area of high strain is usually not considered in pipeline design.

EUROPIPE performed such tests in 2012. Grade X70 was tested in 80 bars pressurized hydrogen with a strain rate of 2.0×10^{-5} 1/s. For comparison, the material was tested in pressurized pure nitrogen. The test samples were machined from base metal and weld metal. Figure 1 shows the result of the tests.

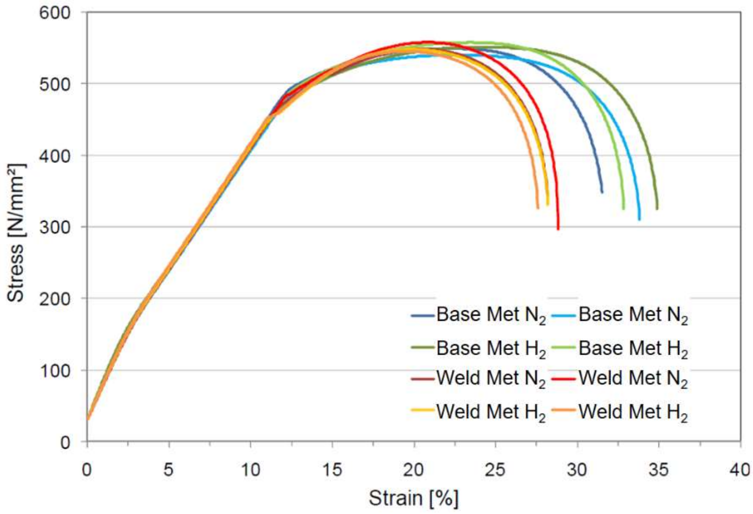


Figure 1: Slow strain rate testing of Grade X70 at 2.0×10^{-5} 1/s in 80 bars hydrogen or 80 bars nitrogen

The figure proves for the selected material no affection by pressurized hydrogen in comparison to nitrogen. The blue and the green lines of the base metal and the yellow and the red lines of the weld metal represent a limited scatter of material property's variation.

Fracture mechanical testing delivers quantitative material values, which can be used for safety or life cycle calculations. For safety analysis, static (ASTM E 1681 [5]) or quasi-static (ASTM E 1820 [6]) testing methods are performed in hydrogen environment. For life cycle calculations, cyclic fatigue testing methods (ASTM E 647 [7]) are used in hydrogen.

Usual grades for pipelines are developed to a high toughness behavior, which is beneficial for safety margins in multi-axial loading situations, as present at notches or at cracks. The above-mentioned codes use in most cases the stress intensity K_I, which is a linear-elastic parameter of fracture mechanics. Linear elastic fracture mechanics was developed to quantitatively describe behavior of brittle material. A plastic zone in front of the crack leads to blunting of a crack tip and with this to de-escalation of the loading situation. High toughness materials, as pipeline steels are, react on multi-axial loads with deformation instead of crack propagation. Linear-elastic fracture mechanics allow plastic zones as long as they are about two magnitudes smaller than the dimension of the ligament. The size of the plastic zone depends on material grade and increases with lower yield strength. For the plane strain case, which reflects the highest loading case at a crack tip, equation 1 estimates the plastic zone radius, which results in plastic zone sizes displayed in Table 1 for different grades for three levels of K_I. The table also shows minimum dimensions for the ligament according to equation 2.

$$r_{pl} = \frac{1}{2\pi} \cdot \left(\frac{K_I}{Y}\right)^2 \cdot (1-2v)^2 \qquad [1]$$

r_{pl}	plastic zone radius
K_I	stress intensity
Y	yield strength
v	Poisson's ratio

$$(W-a) \geq 2.5 \cdot \left(\frac{K_I}{Y}\right)^2 \qquad [2]$$

W-a	ligament size

Table 1: Plastic zone size and minimum ligament size for different stress intensity levels

Grade	L360			L415			L450			L480			L550		
K_I [MPa√m]	20	55	120	20	55	120	20	55	120	20	55	120	20	55	120
r_{pl} [mm]	0.08	0.6	2.84	0.06	0.45	2.13	0.05	0.38	1.81	0.04	0.33	1.59	0.03	0.26	1.21
min (W-a) [mm]	8	58	278	6	44	209	5	37	178	4	33	156	3	25	119

For testing, specimen dimensions shall consider the limits in Table 1. Especially for the higher loads, the specimen sizes would get unrealistically high to be machined from a pipe or to fit in testing equipment for hydrogen testing. Side grooves on the specimens achieve the plane strain condition also at the specimen's surface for which Table 1 is valid. Missing side grooves resulting in plane stress conditions, would require even bigger dimensions, as plastic zones are approximately six times bigger as those from plain strain conditions. KD-10 from ASME Sect. VIII, Div. 3, considers for the displacement controlled test protocol only half of the applied stress intensity (equation 3) in case of subcritical crack extension, which is expected for the high toughness steel pipes in current pipelines. To prove a level of 55 MPa√m, means to test at min 110 MPa√m, which leads to high plasticity at the crack tip according to Table 1. In this case, the steel is tested under unrealistic material conditions with considerably plastic deformed microstructure, which is known to be more susceptible to hydrogen absorption. For the displacement controlled test protocol, the applied stress is derived from the displacement with the rules from linear-elasticity. As soon as plastic zones get impermissibly big, this stress-displacement correlation loses validity and the applied stresses are dependent on plasticity properties of the material. In this case, the violation of validity rules of linear-elastic fracture mechanics results in an undefined applied stress intensity K_I, as the plastic zones relax loads at the crack tip at any amount.

$$K = \sigma \cdot \sqrt{\pi \cdot a} \cdot f\left(\frac{a}{W}\right) \qquad [3]$$

K	stress intensity

σ stress
a crack length
f geometry function

Therefore, it seems to be the better approach to use parameters from the elasto-plastic fracture mechanics, as CTOD or J-Integral, and convert them into stress intensity, as the stress intensity describes the multi-axial loading situation well at notches and cracks. The material testing shall use reasonable loads to show that material can cope with the realistic mechanical situation and to avoid unrealistic plasticity conditions.

For investigating the different toughness behavior of materials, it makes sense to exaggerate the loads and to challenge the toughness properties. From our perspective, the quasi-static testing procedure is able to cover a bigger range of toughness requirements going into the regime of elasto-plastic fracture mechanics.

3. EUROPIPE'S HYDROGEN TESTING PROGRAM

EUROPIPE has setup a testing program to investigate the performance of usual pipeline grades up to X70, as many of the recently installed pipelines are made of such steels. The outcome of this program shall be, whether such steels can be used for hydrogen transport in pipelines designed to the principles of ASME B31.12 [8].

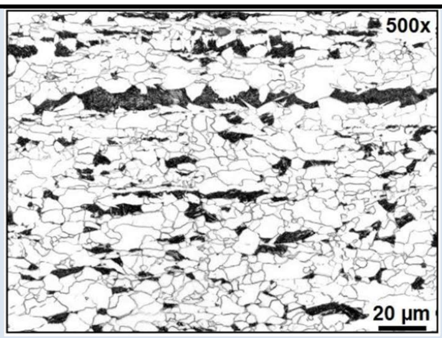
3.1 Pipe Materials

For its program, EUROPIPE selected three different steels from previous orders. The steels represent three typical cases of the pipeline industry, an X70 for onshore use, an X70 for offshore use, and a sour service X65 material. All testing is performed in pure hydrogen, as this is expected to be the worst-case scenario and serves the preparedness for future pure hydrogen transport pipelines.

The onshore X70 is typical for pipes recently installed in Europe. The wall thickness of such pipes ranges up to 23 mm and these pipelines operate at pressures between 80 and 100 bars. They are optimized for meeting the requirements for gas pipelines of Annex A of ISO 3183 [9] with respect to Y/T-ratio in the tensile testing and temperature-toughness behavior in CVN-tests and DWT testing. The microstructure is ferritic-pearlitic. The chemical composition is pure CMn type, Nb-microalloyed, and with carbon contents up to 0.12 %. The basic data are shown in Table 2.

Table 2: Characteristics of Onshore X70

C	Mn	Si	P	S
≤0.12	1.6-1.8	≤0.45	≤0.020	≤0.002
Ni	Cu	Cr	CE	PCM
-	-	-	≤0.42	≤0.22
$R_{t0.5}$	R_m	Y/T	A_f	
485-605	570-760	≤0.90	≥18%	

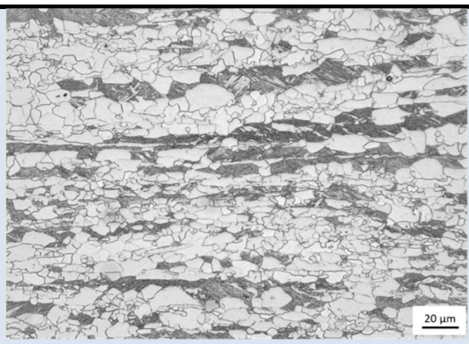


The offshore X70 concept is more dedicated to higher wall thicknesses up to 40 mm, due to high external pressures for deep-sea applications and high pressures above 200 bars. The basic technical rules are fixed in the DNV standard

ST F 101 [10] and the material is optimized to meet the toughness requirements in the heat affected zone of the seam weld, which is welded with very high heat input due to the wall thickness. The microstructure is ferritic-pearlitic with a significant bainite content. The chemical composition is lower carbon CMn type, Nb-microalloyed, with Ni, Cu, and Cr additions. The basic data are shown in Table 3.

Table 3: Characteristics of Offshore X70

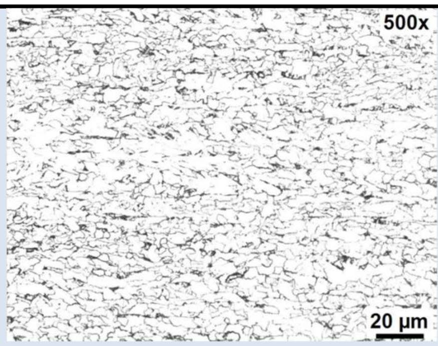
C	Mn	Si	P	S
≤0.08	1.7-1.9	≤0.30	≤0.015	≤0.002
Ni	Cu	Cr	CE	PCM
≤0.35	≤0.30	≤0.30	≤0.42	≤0.20
$R_{t0.5}$	R_m	Y/T	A_f	
485-605	570-760	≤0.93	≥18%	



The investigation on sour service X65 is conducted to find out whether high purity steels produced for transporting natural gas with a significant share of H_2S has a significantly better mechanical performance in hydrogen than non-sour steel. Sour service is also dealing with hydrogen being absorbed in the steel, but in this case by a chemical process not by dissociation of pressurized hydrogen. The necessary high purity is achieved by measures in the steel mill like desulphurization and dephosphorization, degassing and cleanness stirring, and segregation controlled, continuous casting. All efforts are made to reduce microstructure inhomogeneities as hydrogen induced cracking sites. The microstructure is ferritic-bainitic. The chemical composition is a very low carbon CMn-type, Nb-microalloyed, with additions of Ni, Cu, and Cr. Table 4 shows the basic characteristics of this steel.

Table 4: Characteristics of Sour Service X65

C	Mn	Si	P	S
≤0.05	1.3-1.5	≤0.40	≤0.010	≤0.001
Ni	Cu	Cr	CE	PCM
≤0.35	≤0.30	≤0.30	≤0.31	≤0.15
$R_{t0.5}$	R_m	Y/T	A_f	
485-605	570-760	≤0.93	≥18%	



3.2 Levels of Hydrogen Content in the Steel

The pipe industry copes since years with hydrogen, which is absorbed in steels. The main effects are known from sour service pipelines with H_2S accompanying the natural gas to be transported. In many cases, CO_2 is present which tightens severity. The steel absorbs hydrogen by a chemical process under formation of FeS. Once the hydrogen has entered the steel, the atoms diffuse to areas in the microstructure, where local stresses distort the lattice, or to interfaces with non-metallic inclusions. If the hydrogen atoms recombine to molecular hydrogen, the gas might split the material and cause hydrogen induced cracking (HIC). A further effect can occur, where hydrogen promotes crack formation and crack propagation in the steel exposed to external stresses (SSC).

To prove the resistance against HIC and SSC, industry has established testing methods where specimens are exposed to a corrosive solution, purged by H_2S, with and without stresses for SSC [11, 12] and HIC [13], respectively. The standard solutions with the H_2S serve as hydrogen sources for simulating real hydrogen attack conservatively. The level of severity in terms of hydrogen concentration is controlled by the partial pressure of H_2S and the pH of the solution. Milder conditions can be adjusted with e.g. the solution of EFC 16 [14]. For more severe test conditions, hydrogen absorption is promoted by cathodic charging methods.

The hydrogen content in the steel from pressurized hydrogen shall be discussed within this paper. Absorption of hydrogen in the steel depends significantly from active surfaces – surfaces, which are not protected by microscopic oxide layers. If this oxide layer is damaged by e.g. machining or gauging, passivation sets in spontaneously. For testing purposes, the specimen's surfaces were grinded to remove the passivation layers, before they were exposed to pressurized hydrogen. The specimens were stored for 30 days under a hydrogen pressure at 100 bars. The results of these tests are compared with results from investigations on grade X70 in 2012, where the steel specimens pick up hydrogen by chemical or by cathodic charging. Table 5 shows this comparison.

Table 5: Hydrogen contents in steel absorbed by different methods

Charging Conditions					H concentration [ppm]
Method	Duration [h]	Solution	Pressure	pH (start)	
cathodic charging	4	$H_2SO_4 + As_2O_3$	Voltage: -1000mV		11.2 – 15.0
chemical	48	NACE A	H_2S 1 bar	2.7	1.7 – 3.2
chemical	144	NACE A	H_2S 1 bar	2.7	1.9 – 3.3
chemical	144	EFC 16	H_2S .02 bar	3.5	0.8 – 1.6
pressurized hydrogen	720		H_2 100 bar		0.08 – 0.15

None of selected grades differentiates significantly in its tendency to pick-up hydrogen from the pressurized hydrogen atmosphere. Therefore, the results are presented as ranges. The standard NACE procedure, with 1 bar H_2S and approximately pH 3, delivers a more than one magnitude higher hydrogen content than the material exposure at 100 bars hydrogen. Even the milder conditions in EFC 16 solution, with .02 bar H_2S and pH 3.5, generates a magnitude higher H_2 content than the pressurized hydrogen. Cathodic charging extends the hydrogen content by a further magnitude, which leads to very high conservatism when selected for performance testing. With respect to the selection of steel for hydrogen pipelines under 100 bars pressure, it seems that a sour service approach seems to be overconservative, as the hydrogen content in 1 bar H_2S environments is significantly higher than in 100 bars H_2 environments.

3.3 Fracture Mechanical Testing

In the program, fracture mechanical testing plays an important role for evaluating the steel's performance in hydrogen. The tests are performed with the quasi-static and the cyclic method at the National Institute for Standardization and Technology (NIST) in Boulder, Colorado.

The quasi-static method is selected because it gives more information about material performance in hydrogen than the static method, which only results in passing / not passing a certain loading level. In a real structure, flaws might form in the heat affected zone developing e.g. from undercuts. Therefore, the fatigue crack is located in the heat affected zone of the seam weld. As this region is known to be weakest with respect to toughness from testing in air due to the high heat input from the seam weld, the results can be considered as conservative. Three specimens are prepared and tested to eliminate the expected scatter by a small statistical approach. The testing environment consists of 100 % hydrogen pressurized to 100 bar. Figure 2 displays the results of this testing and the evaluation of the critical J-value for crack growth. The value 57.8 kJ/m² shown as J_Q represents the onset of stable crack growth under multiaxial load can be recalculated into a K_{IJ} with the equation 4. The calculation results in a K_I-value of 114.5 MPa√m for the onset of stable crack growth which is well above the 55 MPa√m that is fixed in the ASME rules. Stable crack growth has still reserves for coping with increasing loads, which leads to the conclusion that a design based on this value is still conservative.

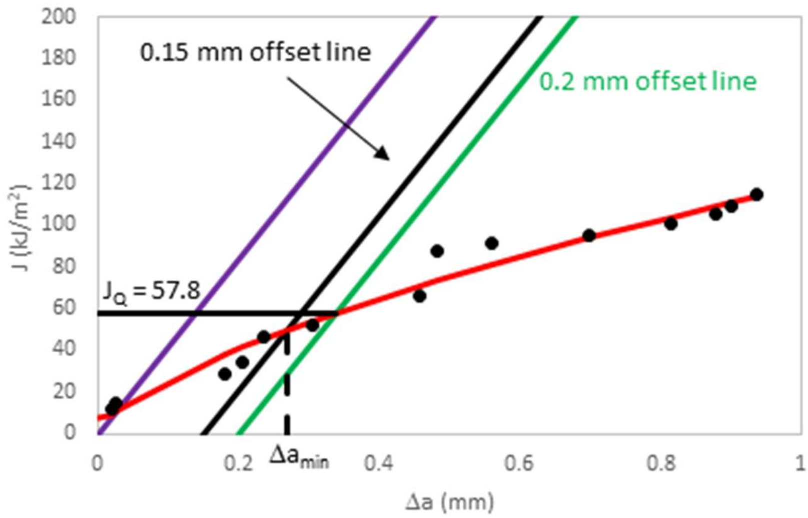


Figure 2: Crack resistance curves of heat affected zone in 100 bar pure hydrogen for Onshore X70

$$K_{IJ} = \sqrt{\frac{E\,J_I}{1-\nu^2}} \qquad [4]$$

K_{IJ}	stress intensity factor calculated from J-Integral
E	Young's modulus
J_I	J-Integral value
ν	Poisson's ratio

The cyclic crack propagation measurements cover a loading range for ΔK from 8 MPa√m to 20 MPa√m with a lower / upper load ratio of 0.5. For a 1mm flaw, this relates to stress levels from 160 MPa to 400 MPa in usual operation with a pressure fluctuation of 50 %. The testing is performed in the base metal, as here the structure is more homogeneous and is not so sensitive to little differences in fatigue crack tip placement. Eight specimens are prepared and tested in an autoclave filled with pure hydrogen at a pressure of 100 bars. A test frequency of 1 Hz was selected, which is a compromise between testing practicality and worst-case scenario. Testing at lower frequencies (0.1 or 0.01 Hz) would extend testing duration significantly. Earlier investigations have shown that 1 Hz exhibits a significant difference in fatigue behavior between hydrogen atmosphere and inert environment, and the effect of lower frequencies can be re-calculated [15]. The results of the onshore grade X70 are shown in Figure 3.

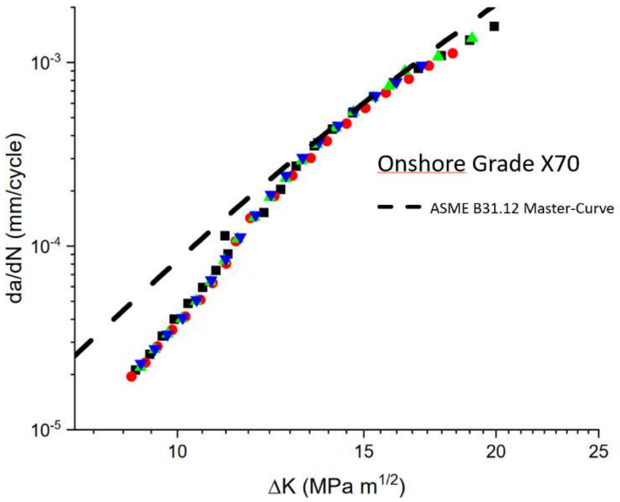


Figure 3: Crack-propagation rates of base metal in 100 bar pure hydrogen for Onshore X70

The differently colored symbols represent single tests. All values line up in a very homogeneous curve. ASME B31.12 defines a master-curve for the crack-propagation ratio dependent from cyclic stress intensity, which can be used for predicting the pipeline life cycle. The black interrupted line displays the development of the ASME B 31.12 master-curve. The testing delivered values, which are close to this curve or fall below the prediction values. This means that pipelines made of such steel grade can be designed according to ASME B31.12 and this design estimates the calculated life cycle well and conservatively.

ASME B31.12 requires for its life cycle calculation an initial crack length, which results from non-destructive testing. For quality control, the entire seam weld length of all pipes are ultrasonically tested. The testing is performed by setting a threshold, which distinguishes indications to examine further. The sensitivity of this threshold results from several NDT-equipment runs on a reference pipe with artificial reflector in the weld and heat affected zones. As a relevant flaw for a fracture mechanical approach might be an undercut, the notches machined in the heat affected zone are the most interesting reflectors of the reference pipe. Specifications usually specify the depth of this reflector to 5% of the wall thickness. This can be considered as an initial flaw size, which might not be captured by the mill's NDT.

The following approach might help to rank the results from the above testing. A 1420 mm (56") pipeline with 100 bars operation pressure has a wall thickness at more or less 23 mm with a safety factor of 1.6. The assumed initial flaw length would result in 1.2 mm, considering the 5 % reflector relevant. According to equation 5, the cyclic loading level is at about 10 MPa√m, assuming 50 % stress relaxation cycles. The results from testing would give a propagation rate of approximately 4.1 10^{-5} mm/cycle, the master curve would estimate the propagation rate with 8.3 10^{-5} mm/cycle. A ΔK being 20 MPa√m would calculate from a flaw depth of approximately 4.7 mm, which is a crack penetrating 20% of the assumed wall thickness. The appropriate crack propagation rates would be 1.6 10^{-3} mm/cycle for the test results and 2.1 10^{-3} mm/cycle for the master curve. The values from testing are determined from specimens with a crack tip from specimens with pre-fatigue cracking, whilst the assumed undercut has a less severe shape. This gives a further conservatism from the fracture mechanical approach.

$$\Delta K = \sigma \cdot \sqrt{\pi \cdot a} \cdot R \cdot f\left(\frac{a}{W}\right) \quad [5]$$

ΔK	cyclic stress intensity
σ	stress
a	crack length
W	specimen width or wall thickness
R	stress ratio (ΔK_{min} / ΔK_{max})
f	geometry function

4. CONCLUSIONS

To reduce greenhouse gases, hydrogen generation is an instrument to transport energy from renewable electricity generation. Pipelines are known to transport any gases safely to the consumers and it is expected to do so with hydrogen. Therefore, it is important to know about the interactions of pressurized hydrogen with the steel pipe and the consequence a potential loss of toughness by hydrogen would have on pipeline design. ASME B31.12 defines design rules, which consider static and cyclic toughness properties of the steel grades being used in pipelines.

As hydrogen quantities are expected to increase in the next decade, pipeline operators plan to blend hydrogen in natural gas to considerable shares or transport high pressure pure hydrogen in pipelines, which are in EUROPIPE's production range. Therefore, EUROPIPE starts investigations in hydrogen performance of its material to be prepared for future requirements. Three different material grades are selected from the EUROPIPE portfolio, which differentiate in microstructure and chemical composition.

To cope with hydrogen pipelines is not new. Hydrogen pipelines are in operation since years, but the conservative design with pipeline grades at the lower end and utilization below 50 % gives limited experience to the plan applying an approximate pipeline design, which is current state-of-the-art for natural gas. Hydrogen pick-up in pipeline steels is also known from sour service pipelines, pipelines transporting natural gas with essential portions of H_2S, but it was not clear whether the rules should be transferred to pressurized hydrogen. The outcome of EUROPIPE's program is that the dissolved hydrogen content in the steel is by more than one magnitude lower in the pressurized hydrogen case than in the corrosion testing case according to NACE or EFC 16. This leads to the conclusion that sour service approaches might be not necessary.

Fracture mechanics allows a design, which considers flaws on the one hand and material toughness properties coping with such discontinuities on the other. It uses stress intensity K_I as a multiaxial loading parameter and the critical stress intensity K_{Ic} of a certain material as material's resistance to such multiaxiality. Using these parameters means to act in the linear-elastic regime of the fracture mechanics, which allows small plastic zones at the crack tip compared to other dimensions of the structure. Testing with too high stress intensities violates the rules of linear-elastic fracture mechanics and leads to results from an undesired, because plastically deformed, material condition. As hydrogen is diffusing to deformed regions, because the dislocation density is high, the effect of falsifying material's performance in hydrogen might be enforced. When using usual specimen geometries for grades up to X70, stress intensity values at and above 100 MPa√m might cause such effects.

EUROPIPE's program comprises quasi-static and cyclic fracture mechanical testing of the three pipeline steel grades. Currently the work is not complete. First results from quasi-static testing of the heat affected zone and cyclic testing of the base metal shows that the design according to ASME B31.12 is still safe for pipelines made of X70 steel and operated at a hydrogen partial pressure of 100 bars. Results from steels with different chemistry and microstructure about improved performance in hydrogen will follow.

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