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# MANUFACTURERS' VIEW ON SPECIFYING LINEPIPE REQUIREMENTS FOR HYDROGEN APPLICATIONS

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## ABSTRACT

Hydrogen is considered one of the most important energy carriers when it comes to the efficient use of renewable energy. Operators for natural gas pipelines are faced with the need to consider pure hydrogen or hydrogen blends in their existing grid and in pipelines to be constructed. The design code ASME B31.12 (2019) provides the possibility for a lifecycle assessment of high-pressure hydrogen pipelines with higher strength grades.

For qualification to ASME B31.12 (2019), pipeline material shall demonstrate its fracture mechanic behavior to verify the code's assumption for the pipeline operation period and the end of its lifecycle. The testing protocols especially for the end-of-life criterion are discussed in this paper. Currently, testing according to ASTM E1681 is valid, which does not describe very well the pipeline steel's toughness behavior.

The non-mandatory Appendix G of ASME B31.12 (2019) gives guidance for chemical requirements combined with microstructure aspects of a special material group for hydrogen pipelines. Material designs for pipes according to grade L415 and above may violate those rules with respect to Pcm.

Considering the frame conditions for testing in hydrogen with respect to worldwide test-capacities, the pipeline industry shall set priority with respect to material qualification in hydrogen.

## DISCLAIMER

# 1. INTRODUCTION

To reduce and avoid greenhouse gas emissions, it is foreseen to change the energy carrier from fossil sources to renewable energy. The sites of energy usage in industry, for mobility, for building heating and for power generation, and as feedstock will be located to a significant degree far from the production sites. Pipelines are the safest and most economical means for gas transport, as experience from the pipeline operation for natural gas has shown.

Thus, the operators for natural gas pipelines are faced with the need to consider pure hydrogen or hydrogen blends in their existing grid and in pipelines to be constructed. For long distance pipelines it is necessary to coordinate the transport grid over wide regions. In Europe, 31 gas infrastructure companies work together to plan a dedicated hydrogen transport infrastructure, the European Hydrogen Backbone [1]. Besides the conversion of natural gas pipelines, the construction of new ones is planned which needs to be completed by distribution pipelines to the points of hydrogen usage.

The pipeline industry is used to cope with hydrogen in steel and has a lot of experience from sour service pipelines. Compressed hydrogen transport in pipelines was covered by technical gas suppliers and the chemical industry. These applications are safely operated since decades.

For an upcoming hydrogen economy with significantly increased quantity of hydrogen in use, the current standards and specifications needs to be re-considered to reach both, a safe and efficient pipeline operation. The worldwide and consumer acceptance of a hydrogen economy will be reached only if safe operation is proved. But, beside the technical safety, an economic application is also needed for acceptance. Therefore, technical efficiency is needed, too.

In this paper, the view of pipe manufacturers on the current standards and rules for qualification of line pipes is given. It contains different aspects of hydrogen embrittlement of steels, relevant testing methods and its worldwide capacities, and materials properties.

# 2. IMPACT OF HYDROGEN GAS ON LINE PIPES

The pipe manufacturer and the pipeline industry are used to handle aspects of hydrogen embrittlement of line pipe steel grades due to sour service conditions of natural gas pipelines since decades. For the transport of compressed hydrogen in pipelines, it is of high importance to clarify, how this compares to the sour service experience. As the focus at sour service is more put on threats of hydrogen induced cracking and sulfide stress cracking, the aspects of hydrogen embrittlement are decisive for this application. To clarify the relevant embrittlement effects for the hydrogen transport in pipelines, the atomic hydrogen content in steel being absorbed from compressed hydrogen needs to be known.

The hydrogen uptake in steel samples was determined by immersion tests. Different line pipe materials and material grades were tested. The samples were placed in high pressure autoclaves and charged with hydrogen gas at 100 bar and room temperature. It is worth noting that the test procedure of evacuation and gas loading is of high importance to avoid any oxygen contamination during testing which was verified by gas measurements with an oxygen sensor (detection limit 0.2 ppm-v). The hydrogen uptake of the samples was measured by carrier gas hot extraction technique latest 48 h after finishing of immersion and interim sample storage in liquid nitrogen.

Figure 1 gives an overview about the measurement results for a variation of immersion times (30 days and 6 month) and surface conditions. The samples were tested as-machined with natural oxide layer after air storage for simulating the delivery condition of a pipe, with freshly ground sample surface for higher criticality in testing, and after aging (250 °C, 60’) with subsequent partial injury (Figure 2) for simulating an in-service condition. The hydrogen content within the samples is given in ppm-w. Please consider the low measurement values for hydrogen uptake of samples immersed to hydrogen gas. An interpretation of result differences for different materials is therefore not meaningful.

The gray bars represent the result for reference samples which were not stored in hydrogen gas with values well below 0.05 ppm-w. If the samples are not ground before immersion, no hydrogen uptake could be measured compared to the reference samples (light blue compared to gray bars). After 30 days of storage in hydrogen using ground samples a significant hydrogen uptake was detected (dark blue bars) which stays constant until an immersion time of 6 month (green bars). The level of hydrogen content does not significantly exceed 0.2 ppm-w at the maximum. Samples which were aged before testing and subsequently injured by scratching show nearly no overall hydrogen uptake (orange bars). Local effects may be hidden due to measurement of the whole sample volume.

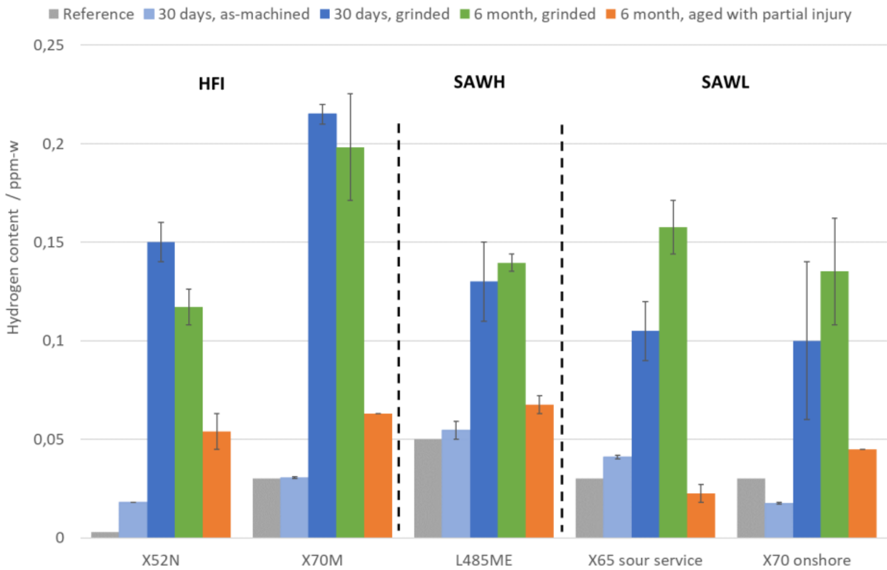


Figure 1: Hydrogen uptake in steel samples of different line pipe steel grades

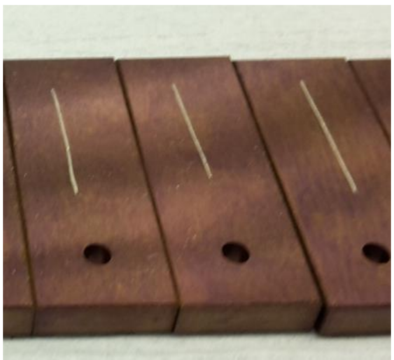

Figure 2: Samples prepared for immersion testing of hydrogen uptake: aged with partial injury of the oxide scale

For sour service conditions, testing of the materials resistance to hydrogen induced cracking (NACE standards) is state-of-the-art. For this testing, samples are immersed in $H_2S$ containing liquid testing media. Using the standard test conditions of pH 3 with 1 bar $H_2S$, a hydrogen uptake in the test samples of about 3 ppm-w is measured. Thus, the hydrogen uptake in standard sour service testing is more than 100 times larger than that during immersion in compressed hydrogen gas and still 10 times larger than for freshly ground samples free of oxygen access.

The reason for this different behavior originates from different surface reactions: In sour service testing, an acid corrosion reaction at the whole sample surface in liquid medium with low pH and a given $H_2S$ concentration takes place under formation of hydrogen and iron sulfide. Part of the hydrogen is absorbed by the steel sample, at which the uptake is increased by the formed iron sulfide on the sample surface.

Hydrogen absorption from compressed gas is in contrast a physical process of molecular adsorption at the surface. Splitting of the molecules to atomic hydrogen is possible at new formed metallic surface (without oxide layer) only. Low amounts of oxygen gas (100 ppm) already allow for a re-passivation of the surface. As a result, hydrogen uptake takes place only locally and depending on the material surface.

In case of this very low amount of hydrogen within the material, hydrogen induced cracks are not presumed to occur. Other integrity threats need to be considered: Although there is an overall low amount of hydrogen present within the material, hydrogen enrichment at positions with locally injured oxide layers at the surface, higher stress levels, or plastic deformations (higher dislocation densities) is possible. This enhancement might be caused by diffusion to the aforementioned features or by hydrogen absorption through freshly formed cracks at the surface. Thus, compressed hydrogen is expected to affect material properties related to high stress concentrations and to the formation and propagation of cracks i.e. ductility and toughness properties. Consequently, the mechanical-technological properties of steel regarding elongation at fracture, reduction of area, fatigue properties, fracture toughness or fatigue crack growth rate are in the focus of investigation.

## 3. STANDARDS

Current standards for high pressure natural gas pipelines (e.g. ISO 3183 [2], EN 1594 [3], API 5L [4]) do not contain specific requirements for hydrogen transport whereas hydrogen specific standards are limited to lower strength levels (e.g. EIGA IGC Doc 121/14 [5] to API 5L X52) and low strength utilization (e.g. EIGA IGC Doc 121/14 [5] 0.4 x SMYS). The design code ASME B31.12 (2019) [6] provides the possibility for a lifecycle assessment of high-pressure hydrogen pipelines with higher strength grades based on a fracture mechanical approach. The German Technical and Scientific Association for Gas and Water (DVGW) is currently revising its technical standards (e.g., G463 [7]) to hydrogen usage considering these ASME-rules. In both cases, fracture mechanics is used to design pipelines for hydrogen transport. The reduced material toughness properties due to hydrogen exposure of the steels is considered. For following editions of the state-of-the-art standards for high pressure natural gas pipelines, the introduction of rules for hydrogen transport are under discussion.

ASME B31.12 (2019) fatigue life evaluation assumes a flaw (initial crack) at the lower detection limit of nondestructive testing or which was identified and sized. The smaller the assumed flaw, i.e. the assured detection limit (N5 instead of N10), the larger the resulting fatigue life. Pressure cycles characterize the pipeline operation period, for which a da/dN-ΔK-master curve reflects the crack propagation behavior. The structure finally collapses, when the crack reaches a critical stress intensity, which exceeds the material's toughness limit. For the design, a safety factor is applied to determine the design lifetime.

To apply the ASME B31.12 option B design principles, the intended material must prove that its crack propagation rate stays below the assumptions from the master curve and the toughness exceeds the minimum from the ASME-code, which is defined as 55 MPa√m. Appropriate testing might result in higher values, which might be used for more realistic life cycle assessments.

For testing, ASME B31.12 refers to ASME BPVC Sec. VIII Div. 3 article KD-10 [8], which indicates methods according to ASTM E1681 [9] for static testing of toughness and according to ASTM E647 [10] for cyclic tests of fatigue crack growth rate. The testing shall be carried out in an environment, which simulates the intended operation with respect to gas composition and pressure. ASME B31.12 (2019) mentions the determination of the threshold stress intensity values $K_{IH}$ (PL-3.7.1 2-a-1) to qualify the construction material. Tests for three different heats and test positions (BM, HAZ, WM) with sets of three samples each are required (sum of 27 samples). For high-toughness material, as thermomechanically rolled pipeline steels usually are, ASTM E1820 [11] or ISO 12135 [12] might describe material's toughness behavior under quasi-static loads better. This will be discussed later.

The standards mention further general aspects on welding of girth welds, hardness limitations, cold forming to chemical compositions and microstructures. They are partly based on experience from sour service material aiming again at a preferably safe service.

Non-mandatory Appendix G of ASME B31.12 (2019) provides precise guidance on chemical requirements combined with microstructure aspects of a specific material group for hydrogen pipelines. For the time being, some pipeline operators use these values as mandatory requirements in their specification without questioning, whether these parameters are decisive for hydrogen service. The necessity of these parameters to fulfil the design and qualification requirements needs to be reassessed and will be discussed later.

## 4. MATERIALS PROPERTIES IN HYDROGEN

Different material properties are investigated in hydrogen atmosphere considering the influence of compressed hydrogen gas on ductility and toughness of the material. The selection of the test methods considers both, availability, and significance for the pipeline application. The results are discussed concerning the existing standards and reasonability of application for materials´ qualification.

### 4.1. Slow Strain Rate Tensile Testing

Tensile testing using slow strain rates (slow strain rate tensile – SSRT testing) under corrosive media is a commonly used test method to evaluate the impact of corrosion reactions. There are standard practices for conduction of SSRT tests in general, e.g. NACE TM0198 [13] and ASTM G129 [14]. For testing in hydrogen atmosphere, this technique is relevant as it provides time for the interaction of hydrogen with the steel surface, plastic deformation of the samples influencing a possible hydrogen uptake and resulting test values reflecting the ductility properties of a material.

SSRT tests were performed in a testing machine equipped with a pressure vessel. Round bar tensile test samples were machined, and the gauge length of the specimen was mechanically ground right before mounting. By this procedure, the specimen's surface was free of an oxide layer and formation of a new one was avoided, which makes the test more conservative (see also Chapter 2). The test procedure for loading of the pressure vessel with pure hydrogen to 80 bar was carefully adopted as for the measurement of hydrogen uptake. Evacuation and gas loading were performed after sample and vessel mounting to reduce not only the free oxygen gas in the vessel but the adsorbed oxygen at the steel surfaces, too. The impact of residual oxygen within the system should be excluded. The tests were conducted at room temperature with a strain rate of $1 \times 10^{-5}\ s^{-1}$. The reference test was performed in 80 bar pure nitrogen with the same strain rate and temperature.

Figure 3 shows exemplary test results performed on tubes of steel grade L360NE. An influence of the hydrogen medium in the pressure vessel is obvious in these test results using activated sample surfaces and a defined testing procedure. The ductility values reduction of area and plastic elongation at failure are reduced to about 60 % and 80 % of the reference values, respectively. The stress-strain-curves of the tests show an equal behavior until the region of sample necking without any changes in yield or tensile strength. The tested samples display the reduced reduction of area for testing in hydrogen atmosphere as well as certain secondary cracking in the necked region.

Thus, the influence of compressed hydrogen gas on the ductility values reduction of area and plastic elongation at failure can be measured in slow strain rate tensile tests of smooth tensile test samples. Using notched samples, additionally an influence of hydrogen atmosphere on tensile strength is expected. Possible qualification values (50 % of tensile strength in hydrogen compared to that in inert atmosphere) are proposed in [15]. Further systematic investigations are planned to clarify the influence of test parameters as well as different line pipe materials on the results of slow strain rate tensile tests. Even though these values are not anticipated to be useful for pipeline design, these tests are expected to allow comparison between materials and can therefore be considered for material's optimization. If in addition the results suggest reasonable usage for qualification testing, will turn out.

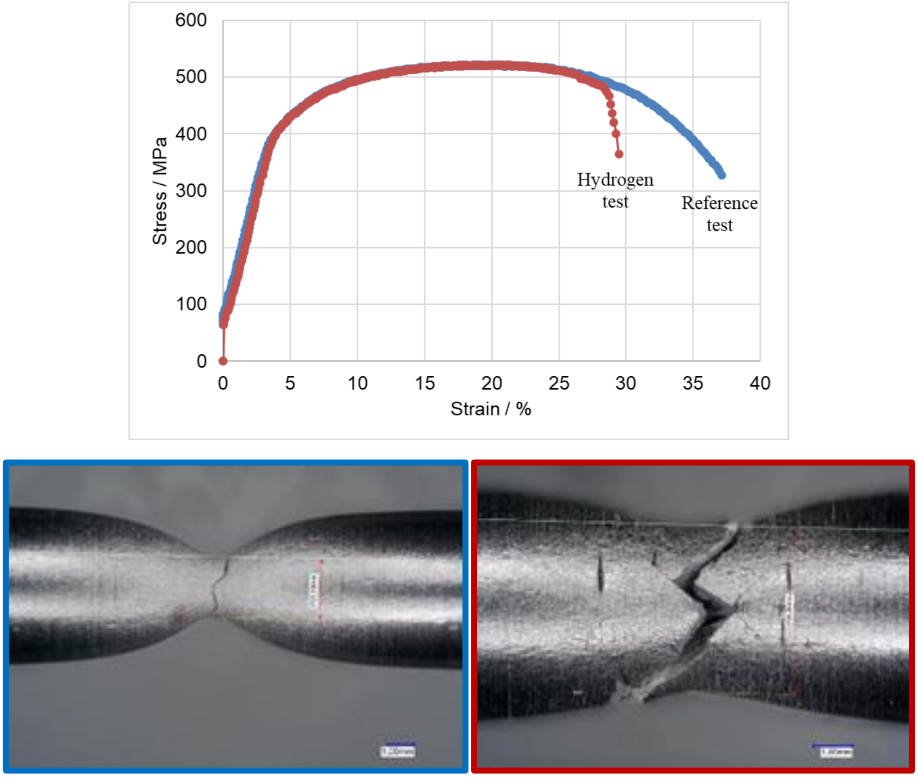


Figure 3 top: Stress strain curves of SSRT tests on L360NE tube material, 508 x 8.8 mm, comparison of test curve in compressed hydrogen gas and in inert atmosphere, bottom: corresponding samples

## 4.2. Charpy Impact Testing of Charged Specimen

Charpy impact testing is a common testing method to qualify pipeline materials for the temperature dependent toughness. A typical approval value is 27 J at a design temperature of e.g. -20 °C [4]. The investigation shall clarify whether this test can characterize loss of toughness by hydrogen as well. As the deformation rate during the test is very high, the diffusion-controlled enrichment of hydrogen at the notch tip cannot set in and no effect is expected. As the test set up does not allow a test procedure in pressurized hydrogen atmosphere, hydrogen charging is getting a challenge. Thus, impact testing was performed on samples which are pre-charged with hydrogen.

For charging, two different techniques were used: chemical charging using an acidified brine solution consisting of sodium chloride (NaCl) and acetic acid ($CH_3COOH$) dissolved in deionized water combined with $H_2S$ introduction at ambient temperature and pressure and physical charging by immersion in 100 bar compressed hydrogen. For both charging methods, the same hydrogen uptake was adjusted to approx. 0.2 ppm-w. For the chemical charging, this was achieved by variation of the $H_2S$ partial pressure (1 bar and 0.01 bar). For comparison, samples without any charging and samples charged to 3 ppm-w hydrogen were tested, too. The test procedure and especially the sample handling after pre-charging in liquid nitrogen with the transfer to the impact testing machine was carefully chosen and kept constant.

Exemplary results of Charpy impact tests on hydrogen pre-charged samples are shown in Figure 4. In all cases the results scatter very much, which makes a clear statement about hydrogen influence difficult. Specimens charged by immersion in compressed hydrogen show insignificant differences, whereas a significant drop in impact energy seems to be found on chemically pre-charged samples compared to uncharged specimens. Nevertheless, the result level of the pre-charged samples is in all cases high enough to even satisfy existing limits for uncharged samples.

The different pre-charging techniques of the samples might have an influence on the distribution of hydrogen within the sample and microstructure. Thus, it might also have an influence on the test result. Especially for the local hydrogen uptake in compressed gas, the influence might be underestimated. However, the large influence of only low overall hydrogen concentrations in the samples chemically charged with 0.01 bar $H_2S$ is another open topic.

The unrealistic procedure for hydrogen charging and the inconsistent results leads to the conclusion that this testing method is not suitable to characterize the material's hydrogen performance. Therefore, a comparison or determination of a weighing factor for Charpy impact values measured in air is not recommended, too.

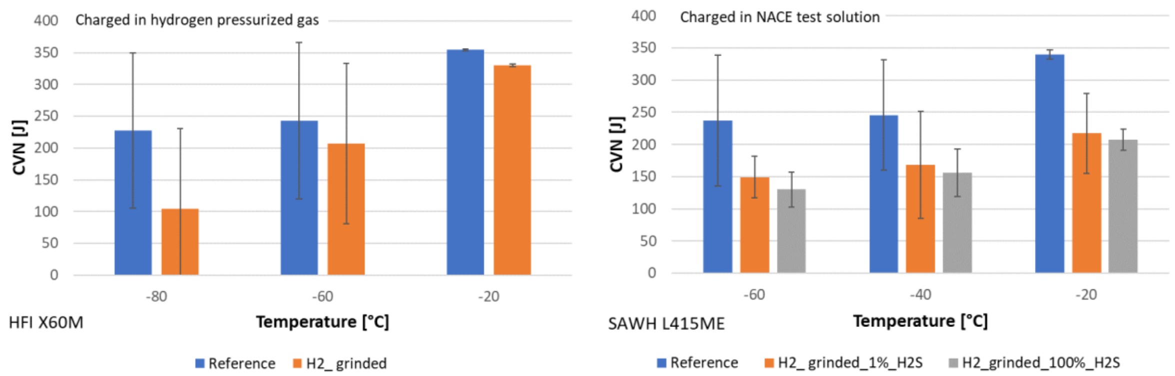


Figure 4*:* Charpy impact test results of hydrogen pre-charged samples in comparison to uncharged reference samples (blue bars), left: charged by immersion in compressed hydrogen, right: chemically charged, different levels of $H_2S$ partial pressure

## 4.3. Determination of Toughness Behavior

For qualification, ASME B31.12 (2019) [6] currently proposes the determination of the threshold stress intensity factor $K_{IH}$ referencing ASME BPVC Sec VIII Div. 3 KD-10 [8], which in turn references ASTM E1681 [9] test standard (see Chapter 3). This testing method and the determined values are based on linear-elastic fracture mechanics. The test standard ASTM E1681 is related to the determination of a $K_{IEAC}$ value under environmental conditions. Together with the criteria of the Boiler Pressure Vessel Code (BPVC) several criteria are intertwined. For instance, ASME B31.12 and ASME BPVC mention the number, position, and orientation of samples in the pipe wall, which ASTM E1681 does not. The BPVC standard specifies a thickness criterion according to which the specimen thickness must be greater than 85 % of the pipe wall. The thickness criterion in ASTM E1681 ensures the reasonable application of linear-elastic fracture mechanics. This cannot be used, as for the applied stress intensity demanded in the BPVC standard and the given material strength, specimen thicknesses had to be used which are even above the pipe wall thickness. For vessel material qualification, BPVC (KD-1048) states suitability of the material up to the design thickness (wall thickness) if the 85 % sample size criterion is fulfilled. Another difference in the standards is the hydrogen exposure time. In accordance with ASTM E1681, this is 10,000 hours (approx. 60 weeks). According to the decisive BPVC standard, it is 1,000 hours (six weeks) instead. Other criteria, such as the load for fatigue pre-cracking and the analysis of the fracture surfaces, are the same in both standards.

The determination of the threshold stress intensity factor $K_{IH}$ according to this procedure was performed for several line pipe materials using the constant displacement method. For one qualification testing, CT-type samples of three heats in the position of base material, heat affected zone and weld metal were extracted in the T-L orientation (Figure 5) followed by final sample machining. Fatigue pre- cracking to the right position was conducted in air. Constant displacement was applied under inert atmosphere in a glove box by a bolt tightened against a flattened pin to a stress intensity ($K_{IAPP}$) of at least 110 MPa√m. That is double the value of $K_{IH}$ (55 MPa√m) which needs to be proven as limit owing to the adapted loading mode assuming no crack propagation. A crack mouth opening displacement (CMOD) gauge was used to measure the displacement during loading. The required CMOD value was calculated based on $K_{IAPP}$ by equations given in ASTM E1681. After exposure of the loaded samples in pure hydrogen gas at a pressure of 100 bar at room temperature for 1000 h (six weeks), the samples were unloaded using the same measuring equipment to record the CMOD values during unloading. The investigation is finalized by heat tinting, break-up of the samples, and investigation of fracture surface using scanning electron microscopy (SEM) followed by the evaluation of $K_{IH}$ value.

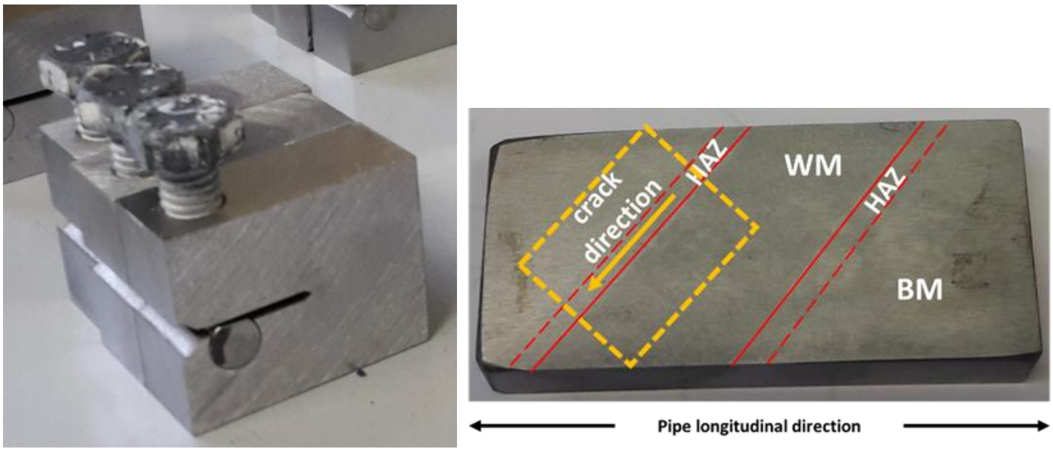


Figure 5: Samples for $K_{IH}$ testing with constant displacement and example of sample orientation in heat affected zone for helical welded line pipes

All investigated materials in all sample positions fulfilled at least the limit value of 55 MPa√m. Some samples were loaded to higher values to evaluate any effects on the test results. Exemplary results for different pipe materials and production processes are shown in Table 1. In only one case (not shown here) of all investigations for higher sample loading, small crack propagation was detected showing that the testing steps and evaluation methods are properly working.

Table 1*:* Exemplary results of $K_{IH}$ values tested for different materials and test positions

| | **Position** | **$K_{IH,min}$ MPa√m** | **$K_{IH,max}$ MPa√m** |
|---|---|---|---|
| L415ME | BM | 56 | 61 |
| | WM (SAWH) | 55 | 62 |
| | HAZ | 57 | 61 |
| X52N | BM | 61 | 63 |
| X60M | BM | 62 | 86 |
| | WM (HFI) | 63 | 85 |

ASTM 1681, chapter 8.6.5 [9] gives a ratio of 90 % CMOD during loading and unloading of the samples as compliance criterion. The evaluation exhibits values between 53 % and 78 %, average 63 % in a variety of grades and applied loads. Origin of this behavior is attributed to plastic deformation at the crack tip. Systematic investigations of one material have shown that applying greater stress intensity factors generally results in a drop in CMOD ratio (Figure 6). This can be taken as an indication that larger stress intensity factors lead to a larger plastic zone, which affects the final CMOD value after testing.

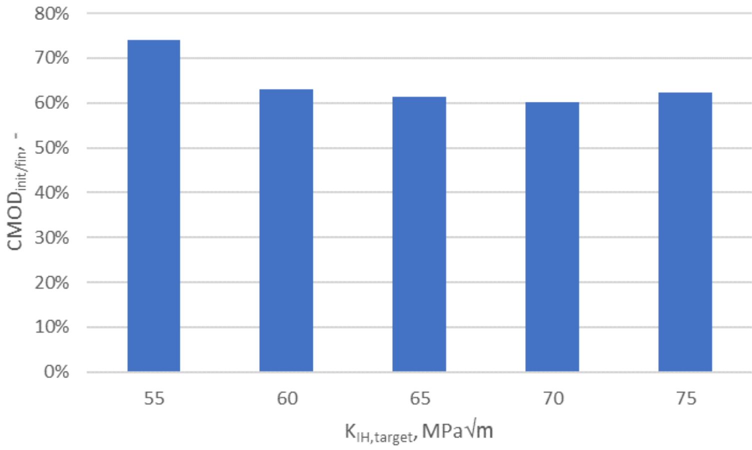


Figure 6: CMOD ratio at various $K_{IH,target}$ values using the constant displacement method

To tighten the overall stress state, side grooves were introduced into the specimens. Consequently, the specimens were closer to exhibit a plane strain state hence reducing the plastic zone in front of a crack tip. CMOD ratios of 70 % to 94 %, average 78 % were determined. This validates the consideration regarding plastic zone. Further investigations und FEM simulations are planned to confirm these results. Application of linear-elastic fracture mechanics is therefore not valid.

In the currently given requirements of $K_{IH}$ testing, the constant load method is another option. The boundary conditions for the test are analogous to the constant displacement method. This applies for the thickness criterion, too. However, the changed loading conditions are advantageous for applying a constant force on the crack tip during testing. In accordance with ASME BPVC Sec VIII Div. 3 article KD-10, the applied stress intensity factor $K_{IAPP}$ can be reduced to $K_{IH}$ (the limit value 55 MPa√m) reducing the estimated size of the plastic zone at the crack tip. However, for the applied sample size in combination with strength level and load state, the conditions for linear-elastic fracture mechanics are again not considered to be met. It is planned to set-up a suitable test equipment to systematically investigate the influence of the changed load method on the test results.

For the characterization of fracture toughness of ductile materials, as valid for novel line pipe steels, the application of elastic-plastic fracture mechanics is appropriate. The material characteristic value $K_{JIC}$. is determined based on a fracture mechanics resistance curve (J-R) in accordance with ASTM E1820 [11]. In contrast to the $K_{IH}$ value, $K_{JIC}$ is an actual material characteristic value. Hence, the test is not used to just exceed a lower limit value.

To determine $K_{JIC}$, a specimen is strain-controlled tested. It is loaded, partially unloaded, and then further loaded. With each cycle, the crack mouth opening displacement (CMOD) increases, leading to consecutive plastic deformation and crack growth. During the test, crack opening and corresponding force are recorded. From these data, both the J-integral and the crack propagation Δa can be determined for each partial unloading. The result is a pair of values J - Δa. The value pairs are then plotted using a power-law. The intersection of the power-law curve with a 0.2 mm offset line then gives the $J_{IC}$ value, which in turn can be converted into a $K_{JIC}$ value. An example of this measurement and evaluation is given in Figure 7 for an X70 onshore material. The result of the measurement in the assumed most critical position of the pipe (heat affected zone of longitudinally submerged arc welded line pipe) gives a $K_{JIC}$ value of 114.5 MPa√m, i.e. more than double the limit value of 55 MPa√m. This result even exceeds determined values for various pipe grades shown as intermediate results of a large investigation program of vintage and novel pipe materials from existing natural gas pipelines in Germany. Results of around 80 MPa√m are shown there [16].

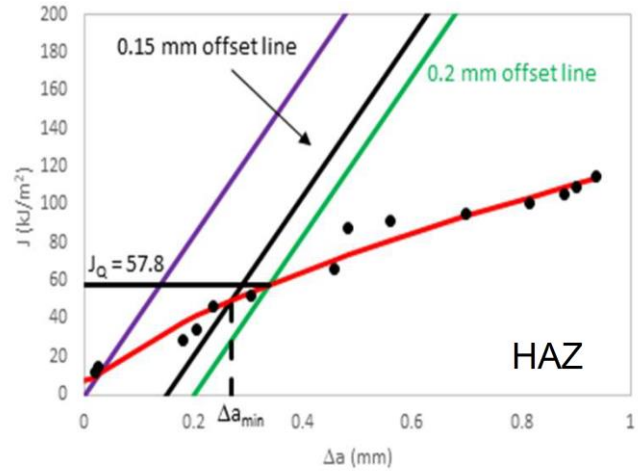


Figure 7: Determination of $K_{JIC}$ for X70 onshore pipeline material, position heat affected zone (HAZ) of longitudinal welded line pipe

Currently, determination of the threshold stress intensity factor $K_{IH}$ is the only procedure of ASME B31.12 (2019) to investigate the material's toughness and for materials qualification. In the given approach adopting ASME BPVC and ASTM E 1681 linear-elastic fracture mechanics are applied. It was shown that these conditions are not met for high-toughness line pipe materials due to a large plastic zone at the crack tip. However, ASME BPVC (KD-1048(b) in conjunction with KD-1043(a)) sets frame conditions, which overrule requirements from the test standard ASTM E1681 and makes the qualification procedure valid for the material up to the design thickness.

The approach allows two different loading methods: constant displacement or constant load. More laboratories preferentially offer the constant displacement testing method, since not overly sophisticated equipment is needed, and multi specimen testing can be easily provided. Nevertheless, the testing effort is quite high, as 27 tests with a duration of six weeks (1000 h) have to be carried out. As no fracture propagation is expected, sample loading is required to be set to double the value to be proven. This is a significant safety factor for the material's qualification but results in an even larger plastic zone. A reduction of the size of plastic zone is possible by introduction of side grooves at the sample geometry, which tightens the overall stress state. Another option to reduce the plastic zone using constant displacement test method might be a load reduction: loading in such a way that the CMOD value upon unloading just exceeds the limit value of $K_{IH}$. (currently larger than factor 1.5). Unloading is an elastic material's reaction. In case of the constant load method, the required $K_{Iapp}$ is already set to the limit value $K_{IH}$. Thus, stress state and expected plastic zone before the crack tip are advantageous compared to the constant displacement method. In contrast, availability of test lab facilities is reduced and time frame for qualification testing is increased.

However, in all cases application of this test procedure violates the linear-elastic fracture mechanics. It does not provide valid results of materials fracture toughness. Nevertheless, for material qualification, such a test method may be a reasonable procedure. In case, any test standard's frame conditions are violated, the pipeline operator should agree on the applied test procedure.

For determination of fracture toughness of a material, elastic-plastic fracture mechanics should be applied. The material characteristic value $K_{JIC}$ is the most reasonable parameter to evaluate materials toughness properties of high-toughness line pipe grades in hydrogen atmosphere. It gives valid results for material's qualification useful for pipeline design. However, the testing effort for each individual specimen is even higher than for constant load testing and worldwide testing capacities are very limited. ASME B31.12 (2019) option B (PL-3.7.1 2-a-3) already mentions the possible usage of the determined value for different pipes if material properties and production parameters are constant. Pipe manufacturer can typically fulfil these requirements, allowing qualification programs for certain general pipe grades. Currently, this approach is still hindered by the low accessibility of appropriate test labs. However, this is again a topic to be discussed between pipeline operator and pipe manufacturer.

## 4.4. Composition and Microstructure

In different hydrogen related standards some aspects concerning composition and microstructure of line pipe materials are given. Most aspects are related to limitation of Phosphorus, Sulfur, and Carbon contents together with limited strength level of materials [5, 6]. It is reasonable that rules working for sour service are adapted to the transport of compressed hydrogen.

As described in chapter 3, ASME B31.12 (2019) [6] provides recommendations in the non-mandatory appendix G. The appendix is understood as an example that the selected material production concept fulfills properties in hydrogen sufficiently. However, also other concepts did show this.

The given constraints are already introduced in material specifications without respecting the given background. The limits of carbon content in conjunction with very low Pcm can be achieved but they also affect mechanical properties, in detail the ratio of yield to tensile strength (Y/T) and pushes this to values of sour service steels as fixed in API 5L Annex H. To fulfil Y/T according to ISO 3183 annex A, Pcm values in the range of 0.19 are needed, for which properties in hydrogen are proven.

Pipe manufacturers control their processes and materials characteristics to adapt the properties to the requirements for specific applications. Production of base material and the welding processes, inclusive combination of welding wire if applicable, are controlled to guarantee high toughness and ductility. Furthermore, the properties of weld metal and heat affected zone for seam welds and girth welds are decisive for the overall properties and are additionally controlled.

In further tasks, the influence of composition and microstructures on all different properties should be systematically investigated to clarify the correlations. A common effort for extensive testing programs to evaluate further reasonable requirements is recommended. Nevertheless, for defining frame conditions in specifications, the decision has to be taken, which properties shall govern for the evidence of hydrogen: The proven toughness properties in hydrogen or the chemical composition and microstructure. The combination of both leads from manufacturer’s perspective to conflicts of contradictory requirements.

# 5. CONCLUSIONS

Considering frame conditions for testing in hydrogen - relevant material properties and adequate testing techniques - the pipeline industry shall set priority with respect to material qualification in hydrogen. From the pipe manufacturers point of view the following aspects are important:

- Hydrogen uptake in compressed hydrogen is generally much lower than by chemical reaction during sour service testing. Hydrogen induced cracks are not presumed to occur.
- Local enrichment of hydrogen can affect properties of line pipe materials: ductility and toughness.
- Slow strain rate tensile (SSRT) testing in compressed hydrogen gas provides possibility to compare material’s performance by ductility properties at large plastic deformation rates.
- Charpy impact testing of hydrogen pre-charged samples does not show clear results and should therefore not be considered for material’s qualification.
- Requirements for material qualification according to ASME B31.12 (2019) is fulfilled for appropriate tested materials.
- From a scientific point of view, current procedure of threshold stress intensity factor $K_{IH}$ testing according ASME B31.12 (2019) applying linear-elastic fracture mechanics is not suitable for high-

toughness line pipe materials. However, due to lab accessibility, test effort, and the aim of material qualification the test procedure might be applied.

- Details of the qualification test procedure must be discussed and agreed on with the pipeline operator. Some proposals of test adaption are given in this paper.
- Fracture toughness behavior of novel line pipe materials is best characterized by the material characteristic value $K_{JIC}$. Current lab accessibility hinders this approach.
- Qualification program of materials should be considered valid for same grades and conditions for different pipeline operators.
- Secondary properties like chemical analysis and microstructure should not be limited at the expense of compliance with primary mechanical-technological properties relevant for the hydrogen application.
- Cooperation between pipeline operator and pipe manufacturer is necessary to balance necessities of design, producibility, and qualification.

## 6. REFERENCES


1. Gas to climate: *European Hydrogen backbone*. https://gasforclimate2050.eu/ehb/, April 2022.
2. EN 1594 (2013), *Gas infrastructure — Pipelines for maximum operating pressure over 16 bar — Functional requirement*s. European Committee for Standardization, CEN, 2013-12.
3. ISO 3183 (2019): *Petroleum and natural gas industries - Steel pipe for pipeline transportation systems*. International Organization for Standardization, 2019-10.
4. API 5L 46th Edition: Line Pipe. American Petroleum Institute, 2018-05.
5. EIGA IGC Doc 121/14: Hydrogen Pipeline Systems. European Industrial Gases Association, 2014.
6. ASME B31.12 (2019): Code for Pressure Piping, Hydrogen Piping and Pipelines. American Society of Mechanical Engineers, 2019.
7. DVGW G 463 (2021): High Pressure Gas Steel Pipelines for a design Pressure of More Than 16 bar; Design and Construction. Deutscher Verein des Gas- und Wasserfaches e.V., 2021-10.
8. ASME Boiler Pressure Vessel Code (BPVC), Section VIII, Div 3: Alternative Rules for Construction of High Pressure Vessels. American Society of Mechanical Engineers, 2019-03
9. ASTM E1681 (2013): Standard Test Method for Determining Threshold Stress Intensity Factor for Environment-Assisted Cracking of Metallic Materials. American Society for Testing and Materials, 2013.
10. ASTM E647 (2015): Standard Test Method for Measurement of Fatigue Crack Growth Rates. American Society for Testing and Materials, 2015.
11. ASTM 1820 (2018): Standard Test Method for Measurement of Fracture Toughness. American Society for Testing and Materials, 2018.
12. ISO12135 (2021): *Metallic Materials – Unified method of test for the determination of quasistatic fracture toughness*. International Organization for Standardization, 2021-07.
13. NACE TM0198 (2020): Slow Strain Rate Test Method for Screening Corrosion-Resistant Alloys for Stress Corrosion Cracking in Sour Oilfield Service. NACE standards, Association for Materials Protection and Performance (AMPP), 2020-09.
14. ASTM G129 (2021): Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking. American Society for Testing and Materials, 2021.
15. ANSI/CSA CHMC 1-2014: Test methods for evaluating material compatibility in compressed hydrogen applications – Metals. Canadian Standards Association and CSA, America, Inc. (CSA Group), 2014.
16. Steiner M, Marewski U, and Engel C (2022): Qualifizierung von Gashochdruckleitungen für den Transport von Wasserstoff. 3-R Fachzeitschrift für sichere und effiziente Rohrleitungssysteme, 1-2/2022, pp 32-38, Vulkan-Verlag.