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SAFETY CONSIDERATIONS FOR THE TRANSPORT OF HYDROGEN GAS IN LINE PIPES AND INDUCTION BENT PIPES

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ABSTRACT

With increasing impact of hydrogen-based economy it is necessary to consider relevant hydrogen embrittlement effects and risks resulting from it. As higher gas pressures are in discussion, the use of higher steel strength levels as compared to existing pipelines appears reasonable. Clarifying the product requirements is necessary for safe operation. Lately, the interaction of pressurized hydrogen gas with steel has been studied more detailed. This allows more precise safety considerations for the transport of hydrogen gas. The results of laboratory trials of different semi-finished products (medium and large diameter line pipes, induction bent pipes) exposed to hydrogen gas will be used to clarify this point. Several laboratory test methods were selected, allowing to focus on local effects of hydrogen enrichment in conjunction with mechanical loads. Challenges when testing in high pressure hydrogen environment result mainly from the safety aspects to be respected, but also from precautions required to achieve reproducible test conditions and to avoid unwanted system contaminations.

Substantiated test results indicate excellent behavior of the materials tested in terms of ductility and fracture toughness in high pressure hydrogen applications; all relevant ASME criteria for material selection are fully met.

To support the ongoing discussion regarding hydrogen testing protocols and relevant material properties (toughness parameters) in both, qualification of components and design of pipelines, Salzgitter and its Steel Processing business units are planning further extended R&D studies.

Keywords: hydrogen, H2ready, line pipe, induction bent, hydrogen uptake, ductility, SSRT, fracture toughness, K_{IH}

NOMENCLATURE

HFI	High frequency induction
SAWL	Longitudinal submerged arc welding
SAWH	Helical submerged arc welding
SSRT	Slow strain rate tensile
RA	Reduction in area
RAR	Reduction in area ratio
E_p	Plastic elongation to failure
E_pR	Plastic elongation to failure ratio
TTF	Time to failure
TTFR	Time to failure ratio
KIH	Threshold intensity factor
CMOD	Crack mouth opening displacement
BM	Base metal
WM	Weld metal
HAZ	Heat-affected zone
SEM	Scanning electron microscopy

1. INTRODUCTION

Steel materials exposed to hydrogen gas may suffer hydrogen embrittlement. The deterioration can be intensified by dynamic loads at high stress and when surface inhomogeneities are present (notch effect) [1]. The degrading effect of hydrogen

affects the materials ductility, fatigue and toughness properties and has been widely studied in the last decades [2 – 9].

Semi-finished products made of steel, such as medium and large diameter pipes, and induction bent pipes, have been in use in hydrogen transportation in single pipelines or small distribution systems for long time. However, they were designed to handle comparatively low gas pressures and flows. For a future upscaled hydrogen usage, operating parameters like those common in the existing natural gas grid must be applied for economic reasons. The properties of steel line pipes and induction bends under pressurized hydrogen must be therefore well known for a safe pipeline operation.

Extensive investigations regarding the influence of hydrogen on the hydrogen uptake, toughness and ductility properties have been performed on pipe and bend materials from different production routes. Inside surface aspects were considered by including coated (e.g. flow coat) materials in the testing program.

All experiment works were carried out at the laboratories of Salzgitter Mannesmann Forschung GmbH (SZMF). The tested materials were provided by:

- Mannesmann Line Pipe GmbH (MLP)
- EUROPIPE GmbH (EP)
- Salzgitter Mannesmann Grobblech GmbH (MGB)
- Mannesmann Grossrohr GmbH (MGR)

which are all part of the business unit Steel Processing of Salzgitter AG (SZAG).

The results are commonly discussed regarding qualification and design aspects of currently available and applied standards.

2. MATERIALS AND METHODS

All investigated materials and the applied testing methods are listed in **TABLE 1**. It is worth noting, that this work is a summary of results gained in several research projects with diversified emphasis.

For the coated samples, three common flow coats with different solids content have been selected (**TABLE 2**). In case of induction bent pipes, flow coat 2 was applied manually and tested.

Several test methods were used in this work to address the resistance of the materials to hydrogen embrittlement. The individual test methods and some special aspects concerning hydrogen to be considered are described below.

2.1 Hydrogen uptake by immersion test

Steel samples with dimensions 40 mm x 15 mm x 5 mm were used for hydrogen uptake measurements. The final step of machining was wet mechanical grinding resulting in a surface roughness (R_z) of 1 or better. Tests were carried out for 7 days and 30 days, respectively. The effect of the surface condition was evaluated by comparing as machined and freshly ground specimens. The as-machined specimens are expected to contain the native oxide layer of the steel, possibly preventing, or reducing the hydrogen uptake. On the other side freshly machined specimen surface (80K SiC emery paper, time gap between grinding and exposure to hydrogen less than 30 min) and storage in ethanol until exposure should effectuate an active metal surface, thus, increased hydrogen uptake.

TABLE 1: Line pipe materials used for testing.

Steel grade		Remarks	Pipe Dimensions OD x WT	Producer	Manufac-turing procedure	Testing method
API 5L	ISO 3183					
X52M	L360M		219 mm x 8.2 mm	MLP	HFI	Hydrogen uptake
X52N	L360N		610 mm x 12.7 mm	MLP	HFI	
X70M	L485ME		1016 mm x 16.8 mm	MGR	SAWH	
X70M	L485ME	Induction bend, mother pipe	1422 mm x 22.5 mm	MGB	SAWL	
X70M	L485ME	Induction bend, tangent with PBHT*	1422 mm x 22.5 mm	MGB	SAWL	
X70M	L485ME	Induction bend, extrados	1422 mm x 22.5 mm	MGB	SAWL	
X65	L450	Offshore	965 mm x 28.6 mm	EP	SAWL	
X70	L485	Offshore	1222 mm 34.6 mm	EP	SAWL	
X52N	L360NE		323.9 mm x 12.5 mm	MLP	HFI	SSRT
X52N	L360NE		406.4 mm x 10.0 mm	MLP	HFI	
X52N	L360NE		508.0 mm x 8.8 mm	MLP	HFI	
X52N	L360NE		406.4 mm x 17.5 mm	MLP	HFI	
X60M	L415ME		660 mm x 11.1 mm	MGR	SAWH	K_{IH}
X70	L485	Onshore	1422 mm x 21.5 mm	EP	SAWL	
X60M	L415M		508 mm x 12.7 mm	MLP	HFI	

*PBHT: post bent heat treatment

TABLE 2: Flow coat materials used for testing.

Flow coat materials*	Solids content
Flow coat 1	ca. 47 vol.-%
Flow coat 2	ca. 61 vol.-%
Anti-corrosion epoxy primer	ca. 97 vol.-%

*all flow coat materials are two component epoxy resins

For the exposure, the samples were placed in high pressure autoclaves (**FIGURE 1**) and charged with hydrogen gas at 100 bar and room temperature. The vessels were evacuated and pressurized with high purity hydrogen at least ten times before each test to achieve low oxygen partial pressure. To verify the latter, representative measurements of the test gas were conducted after a test exposure of 30 days. For this, a calibrated oxygen sensor (oxy.IQ, GE) was used. The oxygen content was found to be below the detection limit of the used sensor (0.2 ppm-v). Inhibiting effects resulting from residual oxygen or oxygen ingress during the test can therefore be neglected After the immersion test, specimens were dismounted and stored in liquid nitrogen until hydrogen measurement but not longer than

48 h to avoid unwanted hydrogen effusion. Carrier gas hot extraction technique was applied (400 °C, 20 min) to determine the hydrogen content. For this, Bruker G4 Phoenix equipped with thermal conductivity detector and IR oven was used.

FIGURE 1: Left: High pressure autoclave for immersion tests in hydrogen gas. Right: Fixture used for mounting multiple specimens in the autoclaves.

2.2 Slow strain rate tensile (SSRT) test

SSRT tests were performed on a testing machine equipped with a pressure vessel. Base metal round tensile specimens from material grades L360NE with four different pipe dimensions were manufactured in longitudinal direction and from mid-wall position. Prior to test, specimens were degreased, cleaned, and dry purged using nitrogen. Right before mounting the specimen into the test vessel, the gauge length of the specimen was mechanically ground using 320K SiC emery paper. This was to produce comparable surface condition for all tested specimens. However, by doing so the native oxide layer of the steel was destroyed, resulting in a more active surface condition, thus, making the test more conservative.

After specimen installation, the system was evacuated, followed by cyclic application of pressurized hydrogen gas (10 bar) and vacuum for at least ten times to achieve low oxygen partial pressure. Inhibiting effects resulting from oxygen impurities can therefore be neglected. Pure hydrogen (100%, quality 6.0) gas was applied at 80 bar. Reference mechanical properties were acquired using the same SSRT test while using inert gas (nitrogen). For each material, one specimen was tested in hydrogen and one in nitrogen (reference properties). All tests were performed at ambient temperature. SSRT tests were conducted at a strain rate of 1 x 10^{-5} s^{-1}. Mechanical testing started after pressurizing with H_2 or inert gas.

Two basic types of results were obtained: visual examination of the specimen gauge section for evidence of cracking, and measurements of specimen ductility with exposure to hydrogen and inert gas. Two ductility parameters were used for evaluation: plastic elongation and reduction in area. Additionally, time to failure was measured.

2.3 Threshold stress intensity factor (K_{IH}) test

The threshold stress intensity value K_{IH} were determined in accordance with ASME B31.12-2019, ASME BPVC Section VIII Division 3-2013, article KD-10 and ASTM E1681-2013.

For the determination of stress intensity factor K_{IH}, bolt-loaded specimens in accordance with ASTM 1681 have been used (**FIGURE 2** top). The thickness of the specimens was in any case at least 85 % of the original thickness of the material as required by the ASME BPVC Section VIII Division 3 KD-1043; no flattering of the pipes was conducted. The final preparation of the specimens, including the notch, was carried out using electrical discharge machining technique. The specimens were prepared in TL direction in accordance with ASTM E399, **FIGURE 2** bottom. For weld metal (WM) specimens, the notch was machined in the centre of the width of the weld and was normal to the surface of the material. For heat-affected zone (HAZ) specimens, the notch was machined at best normal to the surface of the material and in such manner that pre-crack includes as much HAZ as possible. After machining, specimens were cleaned by ultrasonic degreasing in alkaline solution.

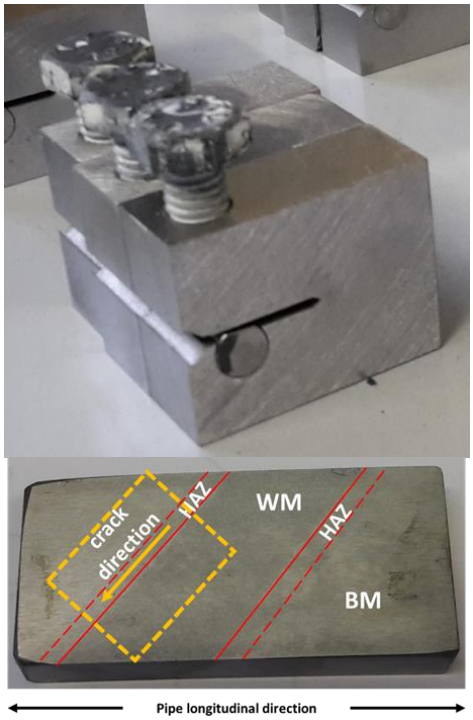

FIGURE 2: Top: Bolt-loaded specimens according ASME 1681 used for KIH testing. Bottom: Postion of specimen showing the crack direction.

For X70 line pipe material provided by EP additional side grooves were introduced into the specimens on each side along the crack direction. The side groove depth with a total thickness reduction of 20% was used, and the side groove radii were less than 0.4 mm.

Fatigue pre-cracking in ambient air environment was then performed. The depth was controlled to be not less than 1.0 mm beyond the tip of the machined notch. A final 1 mm increment at maximum stress intensity factor (K_{max}) of not more than 60 % of expected K_{IH} value was applied. It was found that the harmonization of the specimen orientation, the notch direction and the fatigue pre-cracking procedure is of great importance as it could significantly impact the outcome of the test. Also, the notch was not always straight, aggravating its monitoring from the outside.

In this work, K_{IH} tests results are presented for spiral welded (SAW) pipe from MGR (L415ME, 660 mm x 11 mm), longitudinally welded (SAW) pipe from EP (X70, 1422 mm x 21.5 mm), and longitudinally welded (HFI) pipe from MLP (X60M, 508 mm x 12.7 mm).

Before exposure to hydrogen gas, the specimens were loaded using constant displacement method. The displacement was applied using a bolt tightened against a flattened pin. The fatigue pre-cracked specimens were loaded to a stress intensity (K_{IAPP}) of at least 110 MPa√m. A crack-mouth opening displacement gauge was used to measure the displacement. The required CMOD value was calculated based on K_{IAPP} by equations given in ASME B31.12-2019 / ASME BPVC Section VIII Division 3-2013, article KD-10. In addition, some tests were performed to evaluate the effect of higher applied stress intensity factor on the fracture resistance in hydrogen gas.

The specimen loading was carried out under inert conditions (< 5 ppm O_2 and < 50 ppm H_2O). For this, all specimens, loading equipment and autoclave were introduced in a glove box.

After loading, specimens were exposed to pure hydrogen gas (99.9999% H_2, quality 6.0) at a pressure of 100 bar and room temperature for the total duration of six weeks. After test end, the pressure in the test vessel is released and specimens taken out. Using the very same measuring equipment, CMOD values were recorded while unloading the specimens.

Post-test analysis included 3D images and SEM measurements (perpendicular to pre-crack at 25% B, 50% B, 75% B) of the fractured surfaces and calculation of K_{IH} value according above-mentioned test standards.

2.4 Flow coat test

The investigations on the flow coat materials were carried out according to DIN EN 10301 annex C.

For the evaluation of the resistance to pressure variations samples of steel substrates coated with the three flow coat materials were subjected to 10 pressure cycles in a high pressure autoclave: Cycles 1 to 4 and cycles 6 to 9 comprised 20 hours immersion at 100 bar hydrogen, pressure relief and 3 hours atmosphere pressure; cycles 5 and 10 comprised 68 hours immersion at 100 bar hydrogen, pressure relief and 3 hours atmosphere pressure. The test provides useful information on the bonding quality of the applied coating, as hydrogen gas may reach the metal/coating interface and cause, e.g., the formation of blisters.

For evaluation of the resistance to blistering in liquid transport media the coated samples representing internal coatings for line pipe and induction bends were completely submerged in saturated $CaCO_3$ solution and pressurized with 100 bar hydrogen with a pressure relief after 24 hours immersion.

3. RESULTS AND DISCUSSION

3.1 Hydrogen uptake

Immersion tests with subsequent hydrogen measurements are a suitable method for investigating the hydrogen uptake of the steel material when exposed to high-pressure dry hydrogen gas. Immersion tests are performed to determine the hydrogen uptake with respect to different immersion times and surface conditions. The results enable the comparison with other kinds of H_2 charging, i.e. electrolytical charging.

The results of the hydrogen measurements are shown in **FIGURE 3**. Uncharged specimens were used for comparison purpose. The measured hydrogen levels give clear indication for a hydrogen uptake under the selected conditions. For the uncharged specimens the hydrogen content was below the detection limit of the measuring equipment. Increased hydrogen levels were observed for some of the charged specimens in dependance of the exposure time and/or surface condition; however, the hydrogen levels never significantly exceeded 0.2 wppm. For the line pipe materials, longer exposure times (168 h vs 720 h) generally led to higher hydrogen levels in the material. For the induction bends and SAWH pipes of same material grade, longer exposure times resulted in slightly lower hydrogen uptake, which is not meeting the expectations.

For induction bends (tangent and extrados) the hydrogen uptake is significantly lower compared to the as-welded mother pipe (no heat treatment) due to the process related heat treatment and PBHT.

Surface preparation was found to be even more deciding for the hydrogen uptake. Grinding just before exposure to hydrogen gas significantly increased the hydrogen uptake. An effect, which is being attributed to the removal of native oxide layer of the steels surface, thus, leading to a more active surface condition.

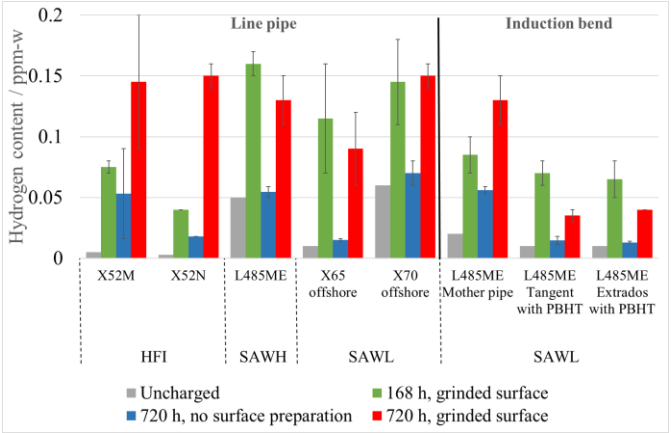

FIGURE 3: Measured hydrogen content after immersion tests in hydrogen gas for different steel grades/products.

No significant difference in the hydrogen uptake behavior was observed for line pipe materials with different microstructure (X52M vs. X52N).

In any case results from hydrogen measurements need to be taken with a pinch of salt, as the measured values are relatively low. Furthermore, the hydrogen uptake behavior significantly depends on the activation of the specimen surface. This effect may cover other effects like microstructure influence or influence on the exposure time due to a somehow limited reproducibility of the specimen surface condition. Further measurements need to be performed to statistically support the existing results.

3.2 SSRT tests

The SSRT test is a tensile test with a low strain rate. The test is performed comparatively under in-situ loading in hydrogen gas and in nitrogen as inert reference gas. The aim is to determine the influence of hydrogen on the mechanical properties of maximum tensile stress, elongation at break and constriction at break, as well as on the fracture behaviour. There are standard practices for conducting SSRT tests in general, e.g. NACE TM0198 and ASTM G129. The testing conditions correspond in most cases the intended practical application.

SSRT test results (**TABLE *3***) in terms of ductility ratios were calculated using following equations:

$$RAR = \frac{RA\ (in\ hydrogen\ gas)}{RA\ (in\ nitrogen\ gas)} x\ 100\% \quad (1)$$

$$E_pR = \frac{E_p\ (in\ hydrogen\ gas)}{E_p\ (in\ nitrogen\ gas)} x\ 100\% \quad (2)$$

$$TTFR = \frac{TTF\ (in\ hydrogen\ gas)}{TTF\ (in\ nitrogen\ gas)} x\ 100\% \quad (3)$$

Exemplary stress-strain curve is shown in **FIGURE 4** for material L360NE (508 mm x 8.8 mm).

TABLE 3: SSRT test results for grade L360NE with different pipe dimensions. SSRT tests performed at 80 bar total pressure and room temperature. RAR: reduction in area ratio, s. Eq. (1); E_pR: plastic elongation to failure ratio, s. Eq. (2); TTFR: time to failure ratio, s. Eq. (3).

Steel grade	Dimensions		RAR / %	E_pR / %	TTFR / %
	OD / mm	WT / mm			
L360NE	508	8.8	58	79	79
	406.4	10.0	64	75	80
	323.9	12.5	65	81	82
	406.4	17.5	66	66	72

Based on the SSRT test data, a distinct impact of the hydrogen gas on the ductility parameters of the tested steel grade can be found. For all specimens tested in hydrogen, a decrease of the reduction in area and elongation to failure is visible as shown by the calculated ratios. No correlation between the dimensions of the tested tubes and SSRT test results could be observed.

The visual examination of the specimens tested in hydrogen showed the formation of secondary cracking (radial cracks) near the primary fracture surface. No such aspects were found for specimens tested in an inert environment. However, the formation of secondary cracks is a typical feature for constant extension rate test in hydrogen. An exemplary appearance of the specimens surface after test is depicted in **FIGURE 5**.

The test results do not suggest any dependance of the ductility properties as a function of pipe wall thickness, assuming homogeneous microstructure of the produced products.

Although SSRT method is a good approach for ranking materials, it has its own weak points. During the pull process, the native oxide layer of the metal is being destroyed because of its low extensibility. The so formed “active” metallic surface provides enough energy for the dissociation of hydrogen molecule to hydrogen atoms. The latter can easily penetrate the metal and diffuse to critical locations of the microstructure, for example crack tips. The cold work or plastically deformation which occurs during SSRT test significantly changes the materials mechanical properties. It is important to consider this aspect during qualification by this method because a failure mechanism may be induced which is not expected in real life service of the steel pipes and bends.

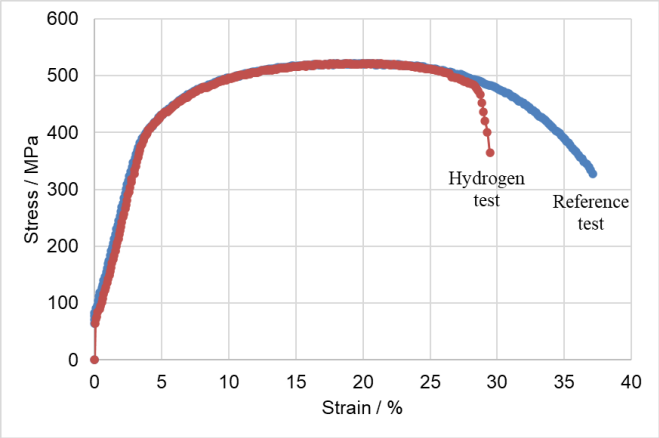

FIGURE 4: Stress-strain curves in SSRT tests for the base metal of grade L360NE with dimensions 508 mm x 8.8 mm (curves for other dimensions similar). Blue curve: reference specimen in nitrogen; Red curve: specimen tested in hydrogen.

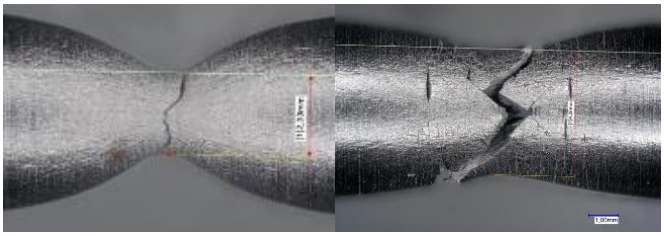

FIGURE 5: 3D images showing the visual appearance of SSRT specimens after testing. Left: specimen tested in an inert environment; Rigth: specimen tested in hydrogen gas.

3.3 KIH

The design code ASME B31.12 – 2019 proposes a fracture mechanics service life analysis of a pipeline for higher strength materials to prevent the pipeline from failing prematurely due to pressure fluctuations. (option B). Three parameters are decisive in this respect: The possible starting crack – in pipes and bends the detection limit of non-destructive testing in absence of real cracks – for the fracture mechanics, the crack propagation according to the intended operation, and the toughness as a material property relevant for the last cycles of instable crack growth.

The threshold stress intensity factor K_{IH} as specified by ASME B31.12 – 2019 PL-3.7.1 (2) Option B as a qualification criterion with a value of 55 MPa√m. The K_{IH} value determines a threshold value below which no crack propagation occurs under general in-service conditions. For L415ME provided by MGR, K_{IH} test was performed on three different heats including base metal (BM), weld metal (WM) and heat-affected zone (HAZ). The position for the specimen machining out of the pipe section is shown in **FIGURE 2** bottom.

For specimen preparation a machined notch was introduced into the specimens with final fatigue pre-cracking to acquire a conservative stress state for testing. Fatigue pre-cracking was conducted in an ambient air environment. For specimen loading a K_{IAPP} was applied with the constant displacement method using bolt tightened against a flattened pin according to ASTM E1681 and ASME BPVC Section VIII Division 3. The loading process took place in a glove box with inert gas to prevent the formation of oxides on the pre-crack surface which could inhibit hydrogen uptake during testing.

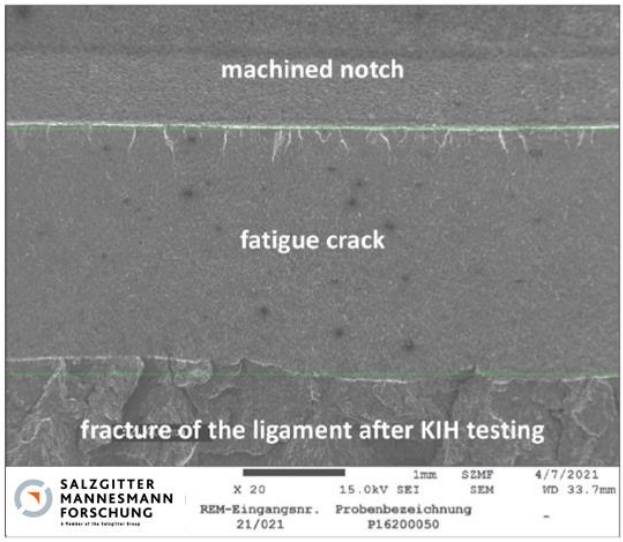

FIGURE 6: Typical fracture surface of K_{IH} specimen without hydrogen crack growth.

After the test, fatigue crack length was measured and scanning electron microscopy (SEM) was applied to measure any potential crack growth due to hydrogen uptake at 25%, 50% and 75% specimen thickness. **FIGURE 6** shows a typical fracture surface of an K_{IH} specimen. K_{IH} was calculated based on crack mouth opening displacement (CMOD) as applied during the stressing procedure. Test results were calculated using Eq. 4 in ASTM E1681. In accordance with ASME BPVC Section VIII Division 3 an additional criterion applies. In case the SEM examination does not reveal crack propagation exceeding 0.25 mm, the calculated K_{IH} value equals to 50% of the applied stress intensity K_{IAPP}. The results for both values are summarized in **TABLE 4**.

The calculated K_{IH} values are in the range of 55 to 62 MPa√m depending on slight variations of the fatigue crack length. SEM examinations showed no crack propagation on any of the specimens due to hydrogen. For all cases (BM, WM, HAZ) a $K_{IH,min}$ value ≥ 55 MPa√m was calculated. The requirements of ASME B31.12 PL-3.7.1 (2) Option B, are accordingly met.

It is reasonable that larger K_{IH} values would have been possible, since even at 62 MPa√m no sign of crack propagation due to hydrogen was detected and no further range of K_{IAPP} was tested. Consequently, at which K_{IH} value crack propagation under hydrogen influence occurs is not evident in this study due to the approach required by ASME BPVC Section VIII Division 3. A limiting K_{IH} may be acquired based on several pre-tests with stepwise increased constant displacement.

TABLE 4: Test results (K_{IAPP} and K_{IH}) for material L415ME (660 mm x 11 mm) tested in 100 bar hydrogen gas.

	K_{IAPP}		**K_{IH}**	
	MPa√m		**MPa√m**	
	min	**max**	**min**	**max**
Base metal	113	123	56	61
Weld metal	110	124	55	62
Heat-affected zone	115	122	57	61

For all positions the required number of valid results was obtained as required by ASME BPVC Section VIII Division 3, article KD-10. No crack propagation was observed hence K_{IH} is equal to 50% of K_{IAPP}. Test results are summarized in **TABLE *5***.

TABLE 5: Test results (K_{IAPP} and K_{IH}) for material X70 (1422 mm x 21.5 mm) tested in 100 bar hydrogen gas.

	K_{IAPP}		**K_{IH}**	
	MPa√m		**MPa√m**	
	min	**max**	**min**	**max**
Base metal	116	119	58	63
Weld metal	118	122	57	61
Heat affected zone	110	117	55	59

It is noticeable, that none of the tested specimens fulfill the 90% crack mouth opening criterion mentioned in ASTM E1681 chapter 8.6.5. Origin of this behavior may be found in plastic deformation at the crack tip due to reduced specimen thickness and the corresponding insufficient stress state. Side grooves were introduced into the specimens, which changes the stress state more to plain strain conditions.

The SEM examination did not reveal any indication for hydrogen crack growth for all the tested specimens. An example for the respective surface appearance of machined notch, fatigue pre-crack growth and brittle fracture of the specimen ligament after breaking the specimen is given in **FIGURE 7**.

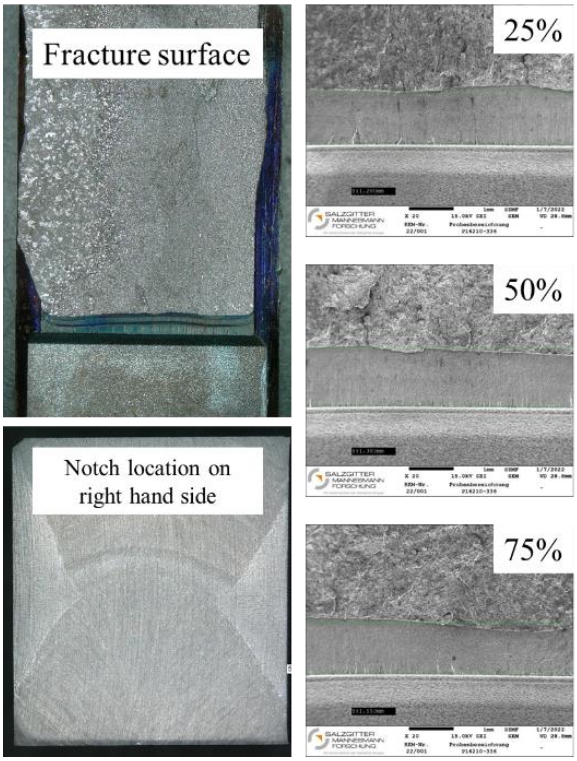

FIGURE 7: An example of surface appeaarnce after K_{IH} test (here HAZ specimen).

TABLE 6: Test results (K_{IAPP} and K_{IH}) for material X60M (508 mm x 12.7 mm) tested in 100 bar hydrogen gas.

	$K_{IHTarget}$	K_{IAPP}		K_{IH}	
	MPa√m	MPa√m		MPa√m	
		min	max	min	max
Base metal	55	124	126	62	63
	60	134	139	67	69
	65	143	144	71	72
	70	142	171	71	85
	75	170	172	85	86
Weld metal	55	127	145	63	72

K_{IH} test was also carried out for material X60M provided by MLP. Here, only base metal and weld metal specimen from one heat were tested. The main aim of the investigation was to evaluate the effect of higher applied stress intensity factor on the fracture resistance in hydrogen gas. For this, base metal specimens were stressed to achieve the default K_{IH} value of 55 MPa√m, but also 60, 65, 70 and 75 MPa√m; weld metal specimens were stressed to 55 MPa√m. For every stress level, a set of three specimens was used. The calculated K_{IH} values after test end were in any case higher than the intended ones. Furthermore, all results were above the limit of 55 MPa√m, which is the qualification requirement of ASME B31.12-2019. All test results are summarized in **TABLE 6**. No crack extension was observed for any of the tested specimens by means of SEM analysis.

3.4 Flow Coats

Beside the investigation on the steel materials also the applicability of commonly used flow coats in hydrogen atmosphere is proofed. Since hydrogen can easily diffuse into the coating, blistering, or peeling from the steel substrate due to an impulsive pressure relief must be avoided. The requirements on internal coatings for line pipes are given in DIN EN 10301/ISO 15741 and API RP 5L2. These specifications apply for the transport of non-corrosive gases and for the most parts the given requirements do not depend on the transport medium itself except for the resistance to pressure variations and resistance to blistering in liquid transport media.

The investigations show that all three flow coat materials are applicable for the use in hydrogen transport pipelines including induction bends.

3.5 CONCLUSION AND PROSPECT

In this paper, the performance in high pressure hydrogen gas exposure conditions of different materials (pre-material, hot rolled strip, welded pipes, and induction bent pipes) is presented.

The results show that hydrogen diffused into the tested materials strongly depending on surface conditions. However, even in case of activated surfaces, the amount of hydrogen uptake is low. Such surfaces are generally not expected during service of pipes or bends where they are typically covered by thin oxide layers. Only in case of larger plastic deformations, these layers could be destroyed due to limited ductility of oxides. This determines the material properties which needs to be considered regarding a possible embrittlement in hydrogen pressurized gas: mechanical properties like ductility, crack propagation, and toughness.

The test results on coated samples show the resistance of the tested flow coats and its application conditions against blistering in standard tests. Beside the proof of product performance these investigations are the basis to investigate and understand the role of coatings for the application of pipes and bends in hydrogen pipelines: influence of the interface coating - steel on hydrogen uptake, and subsequently on diffusion and the resulting product performance.

Slow strain rate tensile (SSRT) testing is a common method to investigate the influence of environmental effects, e.g. high-pressure hydrogen gas, on the technological-mechanical properties of materials and allows a sound comparison of the performance of different materials. The presented results on smooth round tensile test specimens show an influence of hydrogen on the reduction in area and the plastic elongation to failure. The observed effect is given only at very high plastic strains which are normally not expected during service time of pipes and bends. Also, yield strength and tensile strength, which are relevant for the initial pipeline design, are not affected by the

high-pressure hydrogen environment. Further SSRT tests are planned with the main aim of clarifying the influence of chemical analysis, microstructure, weld zones, etc. on the material's performance.

In case of pipelines for hydrogen gas or gas blends for higher pressure levels or higher material grades the design and qualification is reasonably performed based on rules of fracture mechanics. Among current standards and regulations, the most frequently cited document is the ASME B31.12 - 2019 design code. All obtained results for the threshold stress intensity factor K_{IH} are in line with the qualification criterion of ASME B31.12 - 2019 PL-3.7.1 (2) Option B. Thus, the individual material characteristics show the tested pipes and bends meet the current requirements set in the standards, and even conditions beyond.

The K_{IH} tests are to be performed according to ASME BPVC Section VIII Division 3-2013, article KD-10 and ASTM E1681-2013. The relevant fracture mechanics parameters are currently discussed in standardization bodies, which are to be used for both, material qualification and service life assessment of pipelines due to several reasons.

Firstly, standard ASTM E1681-2013 requires a minimum specimen thickness for the testing procedure. For typical strengths in pipelines, this means that specifically large specimen thicknesses must be given. Values determined with lower specimen thicknesses are therefore not providing a material characteristic value. In combination with (ASME BPVC, 2013), an engineering parameter can be determined that allows 85% of the pipe wall as specimen thickness criterion. Such an approach is reasonable for qualification tests considering the difficulty / impossibility to determine K_{IH} values above a certain pipe wall thickness. Nevertheless, this means that K_{IH} values may only be transferred to other wall thicknesses within very narrow limits. Exceeding said limits means performing new pipe material qualification. This is addressed in the combination of the three mentioned standards by the number of required test samples: set of three samples in BM, HAZ and WM for three heats and each wall thickness.

Secondly, the determined K_{IH} values only allow a statement of a certain limit value. The actual material property is not determined in the K_{IH} test, as it is required in ASME B31.12 - 2019. Other characteristic values like K_{IC}, J-integral, K_{JIC}, which are determined in standard fracture mechanics tests (e.g. ASTM 1820), however, allow the common usage for construction or design calculations. These values are also capable of taking material behaviour (brittle, plastic deformation, ductile crack propagation) into account.

It is to be discussed with the standardizing bodies, the pipeline designers, constructors and operators, and the component manufacturers how to address these aspects in both, material qualifications and –differently – for the design of pipelines. It is important to consider the scientific rules behind to make sure that the service of hydrogen pipelines is of high safety. Based on this knowledge reasonable qualification tests can be adapted addressing both, sufficient safety, and economic aspects of testing (capacity and effort).

SZMF is already capable of performing tests under high pressure conditions. Testing machines allowing the determination of fracture mechanics parameters, which are relevant for fatigue life assessment (K_{IC}, J-integral, K_{JIC}, fatigue crack growth rate da/dN) are also planned in the future. The performed investigations of the pipe and bend material presented in this work already indicate a good performance under hydrogen pressurized gas which will further be systematically investigated regarding the influence of different product properties as well as further test methods.

4. CONCLUSION

Substantiated test results indicate excellent behavior of the materials tested in terms of ductility and fracture toughness in high pressure hydrogen applications, and all relevant ASME criteria for material selection are fully met.

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