EPRG Draft best practice guidelines for small scale fracture toughness testing in hydrogen gas of carbon steel pipeline material

Rev. 4a – 20th June 2024

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Foreword

With the advent of gaseous hydrogen transmission in Europe and globally the material testing in hydrogen gas is becoming a paramount driver for safe and reliable design, repurposing and operation of future hydrogen gas transmission (and distribution) grids but is very much at a nascent stage. The common challenges in the hydrogen gas testing involves sample preparation, selection of the test method and test equipment, control of the environment namely the impurities and the post test interpretation of the results to define the representative fracture toughness. The intent of the EPRG best practice guidelines is to provide an informative way forward for the end user and the supply chain on the recommended practice alongside a supplementary narrative with the reasoning for the guidance. The guidance is aimed to bridge the gap between the current testing standards aimed at gaseous hydrogen fracture testing ANSI/CSA CHMC-1 and the future revisions of these or creation of new dedicated material testing standards for gaseous hydrogen material testing.

Scope of this document

The guidance document is primarily concerned with the small-scale laboratory fracture toughness testing of pipeline carbon steel materials using the ASTM E1820 rising displacement test approach. It refines and supplements the guidance of ANSI/CSA CHMC-1 in the areas of:

1. Test specimen
2. Test apparatus
3. Control of the environment and impurities
4. Test execution including crack growth monitoring
5. Post-processing of the results

Majority of the guidance described in this document can be directly transferred to other small-scale fracture toughness test methods (i.e. constant load, constant K) as well as inform other small scale material testing such as fatigue crack growth and slow strain rate (SSRT) testing. The later are subject to future revision of this document. The document also invokes use of an alternative constant load fracture toughness test method given in the Appendix to support the guidance on the test result interpretation of Section 5.

As this is a guideline the modal verb “shall” and similar verbs are to be considered highly recommended whilst “should” and similar modal verbs are advice.

This guideline does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this guideline to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

Introduction to rising displacement J-R testing in air

The fundamentals of the test method are commonly described in ASTM E1820 or ISO 12135 for testing in air. The rising displacement fracture toughness testing commonly uses high constraint single edge notched bending (SENB) or compact tension (CT) samples which are notch and fatigue pre-

cracked in air. Variations on the test geometry are possible such as use of a single edge notched tension specimens (SENT) in certain applications. The testing is done using a universal mechanical testing machine which can be servo-hydraulic or servo-mechanical screw driven machine capable to apply load at a controlled cross-head movement rate. During the test the force, sample deformation and crack growth are recorded.

The deformation of the sample can be measured in two ways;

- Directly using a clip-gauge or similar device (LVDT or non-contact camera) to record the deformation of the crack mouth during the test
- Indirectly by means of measurement of the frame cross-head movement or movement of the pull rods; Such indirect measurements need to correct for the compliance in the test fixtures by trials on the representative blanks.

The crack growth can be measured in two ways

- Directly using an electrical current based approach (potential difference method)
- Indirectly using the change in stiffness of the sample with a growing crack (the unloading compliance method)

The results are post-processed using the guidance of ASTM E1820 or ISO 12135 to construct a tearing resistance curve (termed J-R curve). For testing in air the fracture initiation toughness is commonly set at the 0.2mm offset blunting line.
For testing in environment additional considerations are required as discussed in this document. These are related to control of the environment purity inside the test chamber, mechanical completion of the test setup, test execution parameters and post-processing of the results.

Section 0. Organization of the document

The clauses presented in this document can be split in two grouping focused on the test execution or focused on the qualification of the test setup as suggested in Table 1. Some overlap can occur and the guidance in Table 1 is advisory only.

Table 1: Organization of the clauses

Grouping	Qualification of the test setup	Test execution
1. Test specimen		Section 1 clauses
2. Test apparatus	Section 2 clauses	
3. Control of the environment and impurities	Clause 3.2; Clause 3.3	Clause 3.1; Clause 3.3
4. Test execution including crack growth monitoring	Clause 4.2; Clause 4.4; Clause 4.5; Clause 4.6; Clause 4.8;	Clause 4.1; Clause 4.3; Clause 4.7;
5. Post-processing of the results	Clause 5.1; Clause 5.2; Clause 5.3;	Clause 5.2; Clause 5.3; Clause 5.4

Section 1: Test specimen

Clause 1.1:
The test geometry shall be of a high-constraint type. Common geometries are the compact tension (CT) and the single edge notched bending (SENB) geometries.

Commentary to clause 1.1:
The role of the crack tip constraint is not yet understood in hydrogen gas. The available research utilises predominantly the CT and SENB high constraint test geometries. Other high constraint geometries are permitted as per the reference in-air test standard such as ASTM E1820. Small differences in the (high) crack-tip constraint are known to exist between the SENB and CT geometries, however, these differences are acceptable for the in-air testing and are considered to have only a weak effect on the results from tests in hydrogen gas. Future research is required to understand and to define use of nominally low crack-tip constrained geometries such as single edge notched tension (SENT) samples.

Clause 1.2:
The test sample should match the available pipe wall thickness.

Commentary to clause 1.2:
It is recommended that nominally full-thickness samples are tested in compliance with the standard in-air testing practice of ASTM E1820 or ISO 12135. The practical limitations of the available pipe thickness and the test machine capacity may impose limitations. From the research to date performed by DNV it is considered acceptable to test samples with the minimum thickness of no less than 5 mm with using CT test samples based on general observation of high degree of susceptibility to hydrogen effects. Thickness less than 5mm can result in global plasticity and the loss of through-thickness constraint in typical X52~X65 materials and give erroneous high toughness results. It may also lead to buckling of the sample in an SENB configuration and is not advised.
If sub-sized samples relative to the available pipe thickness are used then the sample should be extracted as close as possible to the inner pipe wall to sample microstructure in contact with gaseous hydrogen where the expected hydrogen concentration will be the highest.
If segregation in midwall is expected than the test sample should be taken from midwall to sample the relevant microstructure.

Clause 1.3:
The heat affected zone in a weld should be tested using full thickness test samples with notch positioning to sample grain coarsened HAZ over the largest extent possible.

Commentary to clause 1.3:
For testing HAZ microstructure in a double-V bevel configuration it is recommended to use full thickness test sample with the notch positioned such that the weld metal and mid-wall HAZ are sampled in approximately 50~50 ratio with preference given to the mid-wall HAZ sampling. For single V-bevel linepipe weld it is recommended to follow guidance of API RP 1104 Appendix A, Section A.3.4.3 for girth welds. Guidance of ISO 15653 Annex A can also be consulted. Further research is ongoing in EPRG and update to the guidance may emerge in the future.

Clause 1.4:
The test geometry can be pre-cracked to an a/W (crack depth / width) ratio in accordance with the permissive of the associated in-air testing test standard such as ASTM E1820 for CT test samples. For SENB the pre-crack depth should not exceed a/w=0.55.

Commentary to clause 1.4:
ASTM E1820 is commonly used to describe the requirements for a rising displacement fracture toughness test and is used as a reference test standard for a rising displacement testing in hydrogen gas. The standard permits crack depth pre-cracked to a/W of 0.7 for use with several test samples geometries including CT and SENB. At present time there is still limited understanding of the role of sample geometry, namely constraint loss effects arising from the sample size (thickness, crack depth, ligament length) relative to the plastic zone size that develops in hydrogen test. Until further research is conducted it is discouraged to purposely pre-crack beyond a/W of 0.55, however it is permissible to utilise a/W of 0.7 for testing CT samples which remain predominantly loaded in tension ahead of the crack tip even at deeper pre-cracks. In case of use of SENT test geometry further research is needed in understanding the effect of pre-crack depth on the SENT test sample constraint.
In all cases the validity requirements of the reference test standard on the final inferred toughness value need to be satisfied (check on the ligament, thickness and crack depth relative to the plastic zone size produced at the associated J value).

Clause 1.5:
The test geometry should have W/B (width / thickness) ratio no greater than 4.

Commentary to clause 1.5:
Limited in-air test data suggests that excessively thin samples relative to the width (ie. a large W/B ratio) may result in loss of out-of-plane crack tip constraint and result in an artificially inflated fracture toughness. ASTM E1820 recommends not exceeding W/B of 4 for in-air testing and these guidelines adopt the same recommendation for testing in hydrogen gas.

Clause 1.6:
The test geometry may have side grooves.

Commentary to clause 1.6:
It is common practice in-air testing of ductile material to introduce side grooves for two reasons – to control the straightness of the crack path during fracture testing (usually an issue in welds) and to reduce the thickness influenced loss of crack tip constraint in thinner samples. Side grooves provide a uniform strain distribution across the crack length. The side grooves can be introduced before or after fatigue pre-cracking.
The use of side grooves in fracture testing in hydrogen gas has not yet been systematically studied. While crack fronts are often not published, a few examples generally show that crack fronts remain uniform (i.e. no tunnelling) even in the presence of side grooves. Introducing side grows before fatigue pre-cracking may hinder monitoring the crack growth. Evidence from cathodically charged duplex material testing shows strong surface effect with failures initiating in the corners (pre-crack – side groove), however in gaseous hydrogen to date no abnormal crack growth patterns have been observed to discourage use of side grooving. In the absence of further evidence and considering the relatively thin wall /

relatively more ductile linepipe material applications it is recommended to apply side grooves to the test sample prior to fracture test. The side grooves may be applied before or after fatigue pre-cracking depending on the test lab preferences.

Clause 1.7:
The test samples may be fatigue pre-cracked in-air or in hydrogen gas atmosphere.

Commentary to clause 1.7:
Hydrogen gas rapidly disassociates in the presence of fresh metal surface which is created at the onset of fracture test in a pre-cracked samples. The extensive small scale test data reported in literature clearly shows the hydrogen effect suggesting that pre-cracking in hydrogen atmosphere is not required for carbon steel materials. A comparison is available in the reference PVP2010-25825. These Guidelines leave the choice of the pre-cracking medium to the user.

Clause 1.8:
After fatigue pre-cracking the samples shall be stored in a reduced humidity atmosphere (in a desiccator). It is not required to apply nitrogen blanket atmosphere during storage.

Commentary to clause 1.8:
After fatigue pre-cracking in air the crack tip will immediately naturally re-oxidise. Upon start of the fracture toughness test the local oxide layer is rapidly broken due to local high stress concentration, which enables hydrogen gas to catalyse on the fresh metal surface and adsorb onto steel. It is not required that special consideration for keeping contaminants such as oxygen away from the pre-crack tip above and beyond the normal laboratory practice for in-air testing be applied in the transition between the fatigue pre-cracking and fracture toughness testing.

Clause 1.9:
The orientation of the crack relative to the pipe geometry shall be defined according to Table 1.

Table 1: Orientation of the crack relative to the pipe geometry

	CT geometry	**SENB geometry**	
		Pipe WT > 13mm	**Pipe WT < 13 mm**
Pipe body	ASTM E399 C-L orientation ISO 12135 Figure B.1 d) Y-X orientation	ISO 15653 Figure 2 or ISO 12135 Figure B.1 d) transverse sample with two notch orientation options: • surface notched with the X-Z crack orientation from the pipe ID • through-thickness notched with the X-Y crack orientation	ISO 15653 Figure 2 or ISO 12135 Figure B.1 d) transverse sample with a through-thickness notch and the X-Y crack orientation
Long seam weld	ASTM E399 C-L orientation	ISO 15653 Figure 2 transverse sample surface	ISO 15653 Figure 2 transverse through-

		notched with the NQ crack orientation from the pipe ID Or sub-size sample with an NP notch orientation, subject of ongoing research.	thickness notched with the NP crack orientation
Girth weld	ASTM E399 L-C orientation	ISO 15653 Figure 2 longitudinal sample surface notched with the NQ crack orientation from the pipe ID Or sub-sized sample with an NP notch orientation, subject of ongoing research.	ISO 15653 Figure 2 longitudinal through-thickness notched with the NP crack orientation

Commentary to clause 1.9:

The CT samples generally position the notch such that the crack front samples the (entire) pipe thickness. The SENB samples generally offer the advantage of growing the crack from an ID pipe surface towards an OD pipe surface and are more consistent to the actual pipeline containing a surface flaw situation.

For the pipe body and long seam weld, the governing loading mechanism is the internal pressure. The governing failure mode is burst from longitudinally oriented imperfections. The C-L sample orientation with the crack extending along the length of the pipe in the CT sample or the transverse SENB sample notched either from ID surface or through thickness (crack extending along the length of the pipe) is recommended to capture material resistance to fracture under internal pressure.

The SENB (or SENT) test samples provide opportunity for using surface notched samples which may better capture the real flaws extending from pipe ID surface. In these samples the recommended notching orientation will depend on the ligament size which will depend on the wall thickness. As a guide the distinction is made at WT of 13 mm where above this thickness either, a surface notched or a through-thickness notched samples can be used, while below this thickness the surface notch sample may not be valid and a through-thickness notched configuration is a recommended starting point.

For the girth weld the governing loading mechanism are the system loads composed of the longitudinal component of pressure (end cap or Poisson's effects), thermal expansion and other longitudinal loads (such as free spans or geotechnical loads (ground movement)). The representative material testing orientation is L-C in the CT sample with the crack extending in the circumferential direction of the girth weld in order to capture the resistance for a postulated crack growing along the fusion line in a girth weld. A surface notched or a through-thickness notched SENB test geometry can be used for this purpose.

Use of the SENB geometry requires care that the sample structural dimensions remain sufficiently large to avoid global structural constrain loss (remote bending affecting local crack tip) in relatively ductile linepipe materials. Validity requirements of the reference test standard (such as ASTM E1820 or ISO 12135 / 15653) must be respected for testing in hydrogen and not relaxed.

Section 2: Test apparatus

The test **Clause 2.1:**

apparatus shall use a suitable pressure chamber (autoclave) for conducting test inside a gaseous hydrogen atmosphere.

> **Commentary to clause 2.1:**
> Although various comparisons have been made between the gaseous hydrogen charging versus alternative means such as electrochemical charging or wet sour charging it is best practice to conduct fracture toughness testing in the gaseous hydrogen atmosphere.

Clause 2.2:
The test apparatus shall have a means of applying active mechanical force on the test sample (i.e. will provide for passage of pull rods through the autoclave).

> **Commentary to clause 2.2:**
> A rising displacement fracture toughness test or similar dynamic material testing requires a test frame with the ability for a controlled application of load and displacement inside the autoclave vessel.

Clause 2.3:
The test apparatus shall be designed to conduct a fracture toughness test on one test sample at a time.

> **Commentary to clause 2.3:**
> It is not recommended to test multiple samples concurrently in series or in parallel configurations due to uncertainties on the measured force values (unless a separate internal load cell is used for each sample), as well as other practical considerations of using a larger autoclave to accommodate more samples.

Clause 2.4:
The test apparatus shall provide means of measuring the crack growth and the sample deformation in-situ during the test.

> **Commentary to clause 2.4:**
> Monitoring of the crack growth and measuring the sample deformation (preferably the crack mouth opening) are required for post-processing of a rising displacement fracture test. The preferred methodology is to install measuring devices on the test sample. Alternative means such as use of a non-contact measuring equipment can be used where proven.

Clause 2.5:
The test apparatus shall be designed to enable purging the entire internal volume and avoid dead zones.

> **Commentary to clause 2.5:**
> For the control of impurities – principally Oxygen and moisture - it is important to have effective purging capability. The positioning of the inlet and outlet high pressure ports needs to

be considered alongside the positioning of the test internals to avoid remnant pockets of contaminated atmosphere. It is recommended that a flow-through autoclave setup is devised. Alternatively a single sided entry can also be used with a purging procedure to demonstrate suitable atmosphere inside the autoclave. See subsequent sections on the recommended purging and control of impurities.

Clause 2.6
The test setup shall include provision for the Oxygen and moisture analysis of the gas contained in the test chamber before and after the test.

Commentary to clause 2.6:
The control of the impurities notably the Oxygen and humidity is important to achieve the objective of the test. It is recommended that the Oxygen and humidity analysing equipment is implemented in-line on the vent lines running form the test chamber. Alternatively an offline batch gas analysis can also be done. Such analyses can take longer time and are prone to introducing further contaminants in the sampling process.

Clause 2.7:
The test apparatus shall use appropriate proven dynamic seals to avoid ingress of external air and impurities at the seal arrangement during the preparation (purging) and during the execution of testing.

Commentary to clause 2.7:
The sealing arrangement between the moving parts – typically the pull rod and the autoclave chamber - is critical for successful execution of a test. Several sealing arrangements between the push/pull rods and the autoclave chamber are possible. The seal needs to prevent ingress of impurities into the test chamber and retain the hydrogen gas inside the test chamber. The sealing arrangement will affect the possibilities for purging the chamber (ie. use of vacuum may be inhibited) and introduce frictional forces at the seal. The frictional forces are also important when the force is measured with an external load cell as discussed further below. During dynamic testing the seal face will wear and a seal replacement on a regular basis is advisable best practice. The frequency of such replacement to be agreed with the test lab.

Clause 2.8:
The test frame shall be able to achieve and control the specified crosshead displacement rate with high precision at any time during the test.

Commentary to clause 2.8:
The control of the crosshead displacement rate is of high importance for a rising displacement fracture toughness test. As discussed further below the expected loading rates are slow. The test frame needs to be able to achieve and control the selected loading rate applied in the form of a displacement rate. From experience it is known that use of servo-mechanical driven frames is better for control at slow rate testing. A dedicated and suitably calibrated servo-hydraulic machines can also be used successfully. The laboratory needs to demonstrate the target loading rate values are achievable and controlled for the proposed test equipment. The constant displacement rate corresponding to a certain K-rate in the linear elastic part of the test is normally defined. For the rest of the test the

loading rate is not controlled, but is a consequence of material response and imposed crosshead displacement.

Clause 2.9:
The force measurement should be made by use of an internal load cell when possible. The external loading cells are allowed, provided the reliability of readings is demonstrated by proper verifications on the apparatus.

Commentary to clause 2.9:
The measurement of force is one of the most important measurements during the test. Use of internal or external load cells have been successful at various laboratories. Both options have advantages and disadvantages and both options can be used.
With the internal load cell, the uncertainty of frictional forces at the seals are removed; however the load cell can be affected by hydrogen-induced drift. In some cases, the signal will stabilize with time (24-48 hours), but stabilization will depend on the sensing technology. A custom-made load cell may be required for successful implementation. Currently, several laboratories use a load ring with a linear-variable displacement transformer (LVDT) calibrated to load. A properly designed load ring displays a linear force-displacement response, no hysteresis and stable frequency response (up to at least 10 Hz).

An external load cell is more readily available (commercial solutions), however external options suffer with uncertainty of the frictional forces at the seal arrangement. Pressure, load path alignment, displacement, seal degradation, multiple seals and other factors can strongly influence friction loads. The interpretation of an external load measurement needs to account for the contribution from the internal gas pressure that can be challenging to account for in a multi-seal arrangement. Any small misalignment in the load train setup can exacerbate the frictional forces in a non-linear manner during the test. The seals can introduce both tension and compression loads – for example a very stiff and tight seal may flex rather than slide thus creating the potential for more complex transmission of load. A proving test should be conducted when using the external load cell to measure the frictional forces using a representative dummy sample setup. Experience across several labs (SNL, DNV, University of Wollongong) suggests that external load cells are suitable up to moderate pressure of 3000 psi (210 bar). Above the pressure (and depending on the seal configuration even at lower pressure), the friction at the seals becomes excessive and internal load cells are recommended. Nevertheless, these outcomes are strongly related to the presence or not of induced load compensating chamber and the type of seals adopted for the test. If an apparatus shows a reliable and accurate reading of load from the external load cell, this is acceptable to be adopted.

Clause 2.10:
For measuring the crack growth during the test the potential difference-based methods (DCPD or ACPD) should be used.

Commentary to clause 2.10:
The measurement of crack growth during the rising displacement test is essential information for postprocessing the result and constructing a J-R curve. Two commonly used methods are available:

- Direct measurement using a potential difference (DCPD or ACPD) method
- Indirect measurement using the unloading compliance method

The direct measurement methods can be the direct current potential difference (DCPD) or the alternating current potential difference (ACPD). The potential difference (also colloquially referred to as the "potential drop") based methods have two advantages:

- Provide information about the onset of crack propagation (transition from blunting to growth) which is useful to establish the fracture toughness threshold.
- Provides continuous monitoring of crack growth with no need for mechanical unloading or adjustments during the test (load compliance method)

DCPD systems generally use between 1 ~6 Amps of current; The required precision of potential drop reading will dictate the required current amperage. Higher currents should be used with caution due to risk of local heating at the connections. On the other hand, larger amperage can improve the signal to noise ratio.
The drift in the potential difference signal due to thermal junction effects and other electronic system effects that may result in drift can be compensated by the current switching (reverse polarity) or by use of a reference test samples.
Commercial solutions are available for DCPD systems. Care must be taken to ensure the potential difference postprocessing software consistently reports the crack at the same point in the current switching cycle to avoid excessive scatter in reported crack depth.
The measurement system needs to provide sufficient data readings to support the post-processing and definition of the threshold toughness. The sampling rate of the potential difference signal should be set to meet this objective.
The potential difference based methods are capable to track evolution of a fatigue pre-crack into a blunted crack and a growing propagating crack during the initial stage of the test. These transitions can be particularly significant to define the resistance to fracture.
The potential difference methods have a further advantage of avoiding the introduction of partial fatigue unloading cycles which can disturb the local crack tip conditions, provide real time information on the crack depth and shorten the duration of the test.
More research may be needed on the implication of using the unloading compliance method due to reverse cyclic plasticity this method introduces. At present the recommended approach is to use the potential difference method.

Clause 2.11:
The crack mouth opening (notch opening) shall be measured on the front face of the sample during the test via a clip gauge or a LVDT setup or via a non-contact optical means.

Commentary to clause 2.11:
Direct measurement of the crack mouth opening by use of a clip gauge attached to the test sample or a non-contact device able to achieve the same aim are the recommended methods. Measurements performed directly on the specimen remove the uncertainty associated with the elastic stiffness from the test frame and the test fixtures. As described in PVP2010-25825 an LVDT can be attached to the front face of the specimen; the displacement measurement location must be considered for proper determination of crack opening displacement at the load line. Alternatively a clip gauge attached to the specimen on the load line (or

specified position relative to the load line, such as the front face) provide a good combination of flexibility and resolution. Whichever method is used it must have sufficient resolution and accuracy for the post-processing needs.
A potential drawback of using a direct measurement inside the test chamber is drift of the measurement equipment signals when immersed in gaseous hydrogen and a more complex test setup, however technical solutions are available and successfully used.
The optical based monitoring systems with the same intent is a possible alternative.

Clause 2.12:
When the crack mouth opening is measured using cross-head displacement the test fixtures (clevises and pins) shall be of a consistent design (tolerances of through holes) and a material grade to reduce variability in the stiffness between tests. Any change in the load train setup will require re-measurement of the stiffness.

Commentary to clause 2.12:
Experience shows that small changes in the material used for the clevises and pins or the fabrication tolerances of the pin holes can alter the stiffness of the test setup and influence the inferred stiffness values. The same set of clevises and pins used in the stiffness calibration of the test setup should be used consistently throughout the test on a given test frame. Any change in the load train setup will require re-measurement of the stiffness.

Clause 2.13:
For an indirect crack mouth opening estimation using load line displacement approach the stiffness of the load frame and the test fixture shall be determined at the start of each test campaign for the combination of clevises, load frame and the project test sample size and test material.

Commentary to clause 2.13:
Experience shows that each test frame ~ clevis fixture is unique and should not be transferred between test frames in order to not invalidate the stiffness calibration measurements. The test sample size and test material will also influence the overall frame stiffness response. The correction for the stiffness effect therefore must be performed at the start of each test campaign. It is important to repeat several (at least 3) load-unload cycles to account for any slack introduced through repeat exposure of the fixtures to the test conditions. Slack may occur due to local compression of dielectric coating on the pins for example and will shake down with repeat cycles.

A procedure for assessing the load frame stiffness can comprise of the three steps:

1. Stiffness calibration using an unnotched blank with the same material, orientation and size as the project test sample ; on each frame (even on nominally identical frames the check should be performed for each frame) ; with at last 3 load unload cycles at a slow loading rate (~10^{-5} mm/s).
2. Baseline in-air toughness performed directly on the same material at the same loading rate as the unnotched blank , sample pre-cracked to a/w=0.5 and using the clip gauge mounted on the crack face to measure the CMOD. Aim to achieve at least 1 mm of crack growth
3. Correlation of the two datasets to correct for the frame stiffness.

Section 3: Control of impurities

Clause 3.1:
The Oxygen and moisture (water) impurities shall be controlled during the test according to the project specified target values.

Commentary to clause 3.1:
The main natural impurities with reported effects on the fracture toughness in gaseous hydrogen are Oxygen and moisture as they temporarily inhibit the hydrogen analytical reaction. Other gasses may also have beneficial effect for example carbon monoxide however these are not natural impurities to be safeguarded from during the test. Moisture is generally understood to inhibit the effect of hydrogen (and should thus be controlled), however insufficient studies specific to the effect of moisture have been performed to date and some suggest moisture may accelerate the effect of hydrogen. This guideline is recommending that both impurities are considered detrimental to the intent of measuring fracture toughness in pure hydrogen atmosphere and should be controlled.
Particular attention on impurities in the test volume is prudent when conducting fracture toughness testing (or other similar testing) at low partial pressure Hydrogen gas such as below 10 barg partial pressure. Experience shows that relatively small concentrations of remnant impurities (from air) in the test chamber have an amplified effect when testing in low pressure environments compared to testing in high partial pressure Hydrogen gas. A workaround solution in such cases is use of a mixed-gas to increase the total pressure or use a flow-through setup with care to not let O_2 back in;
In the absence of project specific requirements on control of Oxygen and humidity the guidance in ANSI/CSA CHMC-1 should be followed.

Clause 3.2:
The control of impurities shall be achieved with a purging procedure prior to the start of the test on a fully assembled autoclave setup.

Commentary to clause 3.2:
The purity of the hydrogen gas in which mechanical testing occurs (test gas) shall not be assumed equal to that of the source gas. The most effective way to control the impurities is with a robust purging procedure. Purging can be achieved in several ways. Purging is dependent on the specifics of the test setup, manifold, sealing configuration, etc. The gas dilution concept is utilized for purging and depends on the pressure difference between the purge pressure and the vent pressure. A positive pressure is ideally maintained to prevent pulling air into the system (as seals are designed for high pressure, not necessarily vacuum).
It is recommended to flow purging gasses through the test chamber at sufficient rate (velocity) to induce turbulent flow inside the test chamber. A pressure wave approach can also be adopted where several cycles of slow pressurisation followed by a faster discharge are applied but care is needed in this case to prevent damage to the soft seals if the depressurisation is too rapid.

Nitrogen is commonly used gas for purging. The final purging cycles are applied with hydrogen gas flow.

There is not yet commonly agreed purging test procedure. Guidance on purging is available in ANSI/CSA CHMC-1. There are lab to lab variabilities in the purging process which will depend on the test setup. Several variations of purging practice can be done considering the test setup limitations. An example purging procedure is a pressure proof test of the setup with nitrogen, then purge out and 3 cycles of Nitrogen and 3 cycles of Hydrogen. Then, flow gas from the test chamber through both oxygen and moisture gas analysers to ensure Oxygen and moisture are within the set specifications. The main assurance step for a successful purging is demonstration with measurement that the test gas in the autoclave chamber has achieved the target impurity level values at the start of the test. The measurement should also be performed at the end of the test to confirm the target values were maintained during the test.
Note that care is required if vacuum purging is also implemented to prevent inadvertently re-introducing air into the test chamber, i.e., pulling air back into the system via the dynamic seals or via the external gas-analysis equipment.
The combination of effective purging and the online gas analysis are the most important steps in assuring control of the impurities before the start of the test. The user should consider both in concert and not prioritise one over the other.

Clause 3.3:
The achieved level of impurities in the test chamber shall be demonstrated with measurement performed on the gas sampled from the autoclave prior to the start of the fracture test and again at completion of the test.

Commentary to clause 3.3:
It is essential that Oxygen is controlled in the test. A combination of effective purging and use of Oxygen analyser at the start of the test is the recommended approach. It is also recommended to analyse the Oxygen content at the end of the test because Oxygen impurity can be retained in the dead volumes, could sip-in at the seal and could de-gas from the soft seals during the test. The targets for the remnant Oxygen are set per Clause 3.1.
The role of moisture at low levels (~10 ppmv) is less influential on the fracture toughness result based on the available literature, although there is a lack of systematic studies. Moisture is difficult to control in the setup as moisture may seep out from soft seals and non-metallic components (e.g., wire insulation, o-rings, etc.) inside the autoclave during the test. A good purging procedure that achieves the desired low level of oxygen is expected to equally reduce the moisture levels into single digit values (<10 ppmv). It is recommended that moisture analyser is used to confirm the levels of moisture in the autoclave before the start and again at the end of the test. This becomes a requirement if higher moisture levels than ~10 ppmv are specified on purpose for the test.
In general all impurities that have a temporary inhibitive effect needs to be controlled and minimised, in particular the CO and H_2S impurities in addition to the Oxygen and humidity.
It should be noted that in-line measuring equipment is generally only available for measuring the Oxygen and moisture values. Analysis of the concentrations of other purposely added impurities would need to be performed offline with gas chromatography techniques.

Section 4: Test execution

Clause 4.1:
The test pressure shall not be less than the maximum allowable pressure for the specific project hydrogen pipeline.

Commentary to clause 4.1:
It is common practice that material qualification is conducted under project specific conditions. It is recommended to apply such approach to testing in the gaseous hydrogen too.

Clause 4.2:
The test pressure should be controlled within 10% of the target value but shall not be lower than the target pressure.

Commentary to clause 4.2:
The target test pressure needs to be maintained as constant as possible during the test. The test apparatus needs to compensate for any pressure losses such as due to leaks past the dynamic seals, especially in longer duration tests. It is recommended to use the accumulator vessels to reduce fluctuations of the source gas. Commonly for testing below 150 barg the pressure from the gas bottles can be used directly. For testing at higher pressures gas booster pumps are used and in such configuration the accumulator vessel is required to smooth out the pressure fluctuations. A well setup test facility should be able to control the pressure to be within +10% -0% of the target pressure.

Clause 4.3:
The test temperature inside the autoclave can be the laboratory ambient temperature (commonly 20~24 degree C).

Commentary to clause 4.3:
Temperature strongly drives the kinetic rate of hydrogen transport in the steel. No systematic study is presently available studying the effect of temperature in the hydrogen gas assisted fracture of carbon steel. Studies on the role of temperature on hydrogen assisted sour cracking in mild wet sour solutions have shown temperature between 4 degree C to 20 degree C provide the optimum synergy between the rate of disassociation of hydrogen and transport to the crack tip versus outgassing. It should be cautioned though that the mechanism of charging in wet sour solution differs from that in hydrogen gas (bulk charging versus surface charging).
For corrosive sour service, material qualification the temperature of 24 degree C is commonly used as the laboratory ambient temperature. For the application to transmission pipelines in Europe (principal scope of this guideline) the expected temperature range is between ground ambient ~0 degree C and up to 60 degree C near the compressor discharge points. For this range of temperature, the standard lab ambient temperature (20~24 degree C) is considered adequate. At warmer temperature research in corrosive sour conditions has shown that the effect of hydrogen is lesser due to enhanced kinetics of diffusion and outgassing. It is postulated that the same will hold for testing in hydrogen gas although strictly this remains a low priority gap.

Clause 4.4:
The test temperature inside the autoclave shall be kept stable during the test.

Commentary to clause 4.4:
This is usually achieved by controlling the laboratory test temperature to be stable. A suggested permissible laboratory ambient air temperature variation is ±3 degree C which can be achieved by common building climate control devices. It should be noted that gases are compressible thermofluids, thus the gas temperature can vary based on pressurization and depressurization rates. It is essential to ensure stabilization of the complete system prior to test execution.

Clause 4.5:
Pre-soaking the sample in hydrogen gas prior to the start of the fracture toughness test is not required.

Commentary to clause 4.5:
Pre-soaking is conducted in wet sour fracture testing to introduce hydrogen in the test sample body prior to start of the mechanical test. The governing mechanism of hydrogen ingress in wet sour is corrosion with suppression of hydrogen recombination thus promoting ingress into steel and diffusion to the active fracture process zone. In hydrogen gas with low impurity content corrosion processes do not occur. In hydrogen gas the mechanism of hydrogen adsorption also changes to a local adsorption promoted by presence of the fresh metal surface rather than bulk absorption through the corrosion process. It is seen from fracture toughness testing starting from the in-air fatigue pre-cracked sharp crack that the local oxide layer at the crack tip is rapidly broken at the start of the hydrogen test and hydrogen can rapidly interact with steel in the initial stage of the test. Slow loading rates are required to allow the hydrogen adsorption kinetics to saturate the crack tip fracture process zone. Under these conditions no dedicated pre-soaking time is necessary to introduce hydrogen atoms into steel substrate prior to the start of the fracture toughness test in gaseous hydrogen testing.
In practical experience samples can be left immersed in hydrogen gas for reason of test apparatus setup (adjusting the drift of the load cell, achieving thermal stability of the gas, assuring stability of electric signal readings, proving sealability) for a duration of a few hours and up to a day. This is not considered to be soaking on purpose.

Clause 4.6:
The applied displacement rate commonly measured with the elastic K rate shall be controlled during the rising displacement fracture toughness test to a slow value.

Commentary to clause 4.6:
The displacement rate is an important parameter for tests that aim to establish the rising portion of the material fracture resistance curve (J-R curve). This applies in particular to the test methods that utilise the rising portion of the J-R curve. Experience from the work at DNV suggests that threshold defined near $\Delta a \sim 0$mm is less influenced by the displacement

rate (ref PVP2023-105972 and DNV H2Pipe JIP) however these still need to be maintained to a slow value.
Test interpretation shows that too rapid displacement rates can outrun the kinetics of hydrogen adsorption into steel and transport to the fracture process zone thus push the growing crack outside of the hydrogen assisted crack growth regime. See example in EPRG PJ 221 Figure 34 based on work of Somerday et al. This can lead to the measurement of erroneous high toughness values. The effect is particularly critical at a lower partial pressure hydrogen gas and in tests under less strict impurity control.
The displacement rate (K rate) in terms of stress intensity factor change over time in the initial portion of the test in the linear elastic regime can be established by performing a standard fracture toughness test on a standard sample in air at a chosen frame displacement rate, recording the force vs time history and afterwards calculating the K versus time to obtain the K rate. It is also possible to define the K rate by post-processing the actual test result performed in hydrogen gas. Currently no consistent agreed procedure exist in defining the elastic loading rate and this is a gap.
Some experience suggests the toughness curves can increase at lowering the test rate too excessively. This can be consequent to a couple of possible mechanisms:
1. material creep that reduces the severity of the stress field at the crack tip as the loading rate is decreased
2. reduced hydrogen uptake in presence of too slow strain increment
Further guidance on establishing the K rate that gives results independent of the test execution is given in Clause 4.7.

Clause 4.7:
The user shall define the target displacement rate or elastic K rate for a given test such that loading rate insensitive result is obtained. Guidance of ANSI/CSA CHMC-1 and published resources (ref PVP2023-105972) can be consulted for reference values.

Commentary to clause 4.7:
Currently there are still inconsistencies on how the elastic K rate is inferred from the test result performed at a given frame displacement rate. There are also inconsistent results obtained at the same elastic K loading rates among laboratories, although no systematic study has taken place fixing all other parameters equal. It is recommended that the available guidance in ANSI/CSA CHMC-1 for the displacement / elastic K loading rate value is used as a starting point alongside any published references showing the effect of the displacement rate on the fracture toughness. It is recommended that a sensitivity assessment on the displacement rates is performed at a start of a given test programme to demonstrate displacement rate insensitive results for the test condition and material (pressure, gas composition, microstructure).
This step is critical for tests performed in a lower partial pressure Hydrogen gas (in particular below 5 barg) such as gas blends or when impurities are chosen to purposely exceed the ANSI/CSA CHMC-1 guidance.
Initially this sensitivity study should be done on a project basis. As the lab (and industry) develop more experience this can be reduced to periodic checks or omitted altogether in the future.

Clause 4.8:
Surface oxide layer does not have to be removed from the sample prior to start of a rising displacement fracture toughness test in hydrogen gas.

Commentary to clause 4.8:
The surface oxide layer is a short term barrier to disassociation and ingress of Hydrogen into metallic substrate. For the rising displacement fracture toughness test the local stress concentration at the tip of a prior fatigue pre-crack rapidly breaks the oxide layer at the initial portion of the test and enables hydrogen catalysis on a fresh metal surface. It is not necessary to conduct additional steps to avoid or remove the oxide layer from the fatigue pre-crack trip prior to the start of the test in hydrogen gas.

Section 5: Post test interpretation

Clause 5.1:
Crack depth measured with the potential difference method shall be adjusted to the measured physical crack depth after the test.

Commentary to Clause 5.1:
The potential difference signal can be postprocessed to represent crack depth using the Johnson's equation. It is recommended however to break open the sample and measure the initial and final crack extension in the test in accordance with the test standard such as ASTM E1820 or ISO 12135 and re-calibrated the measured potential voltage difference signal to these measurements.

Clause 5.2:
The fracture toughness threshold for the onset of crack propagation (more precisely termed the initiation fracture toughness) can be determined as follows:

a) For crack growth measured using the potential difference methods
Option A: At a J corresponding to the $\Delta a \approx 0$mm and termed J_{th} (the equivalent toughness expressed in terms of the stress intensity factor K is $K_{J0mm.}$)
Option B: At a J value for $\Delta a > 0$mm and termed $J_{x.x\ mm}$ where x.x mm represent the Δa value selected (the equivalent toughness expressed in terms of the stress intensity factor K is $K_{Jx.x\ mm.}$).

b) For crack growth measured using the unloading compliance the threshold toughness is recommended to be taken at $\Delta a = 0.05$mm and termed $J_{0.05mm}$ ((the equivalent toughness expressed in terms of the stress intensity factor K is $K_{J0.05mm.}$)

Commentary to clause 5.2:
For Option A the guidance in ASTM E1820 or ISO 12135 is first followed to construct the J-R curve. When the potential difference-based crack growth measurement is used, it is possible to define the threshold of crack propagation as illustrated in the Figure 5.1a) below (blue arrow) at the point where a straight line fitted to the initial portion of the DCPD signal departs from the rising portion of the DCPD signal. Work at SNL has used interrupted tested and heat tinted surface to confirm the point of crack initiation corresponds well with this approach. From such post-processing the associated J-R curve will have a distinct "knee" near the $\Delta a \approx 0$mm as shown in Figure 5.1 b). The threshold fracture toughness at $\Delta a \approx 0$mm can be defined directly from these plots.
An example of a post-processed data by DNV Columbus is shown in Figure 5.2. The normalised potential difference signal versus CMOD signal is plotted and a straight line is fitted to the initial portion of the signal. A 0.2% offset line is fitted and the crack initiation point is defined at the intersection with the data. A subsequent check is performed that the defined initiation point corresponds to the max load value.

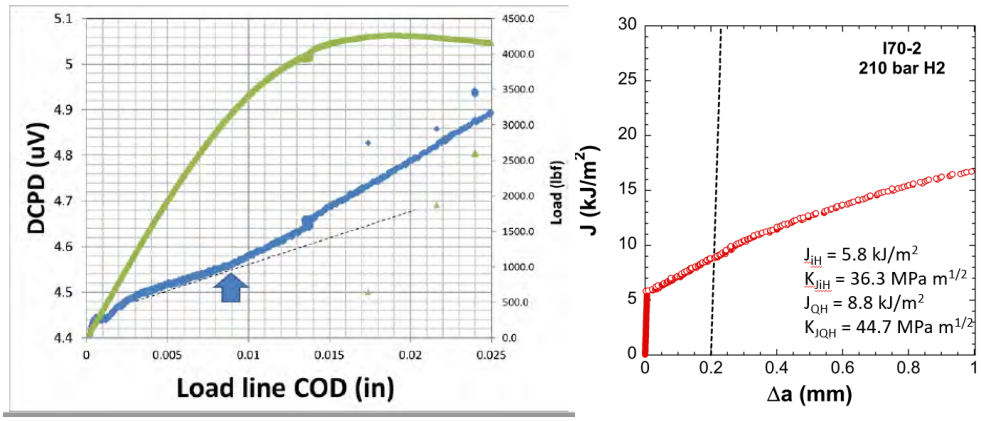
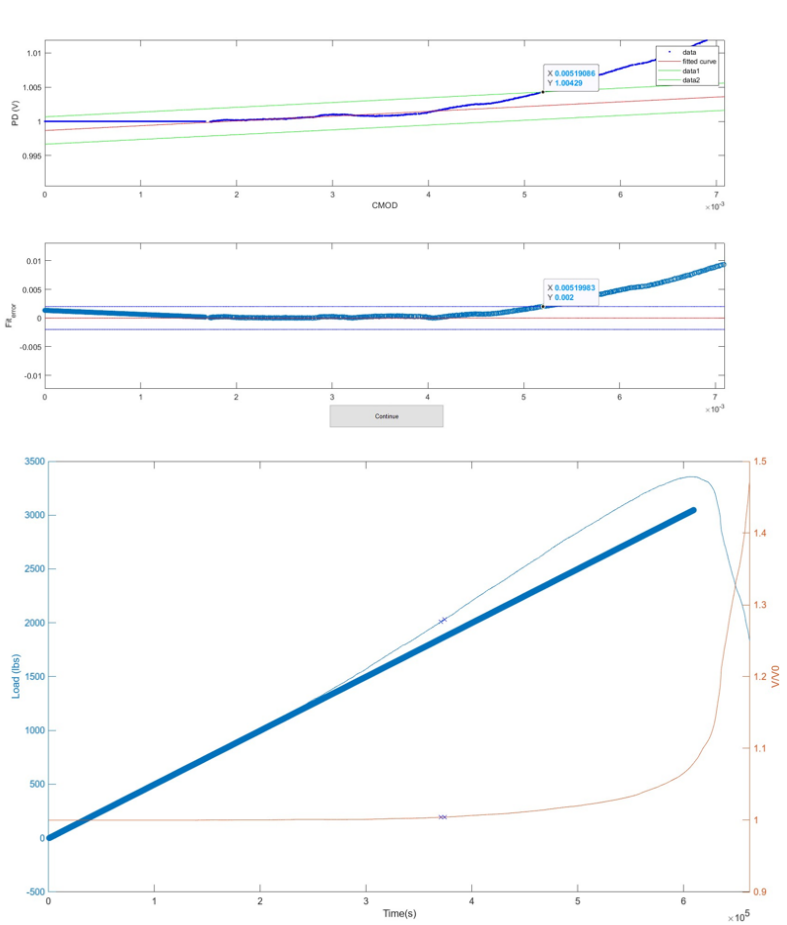


Figure 5.1 Illustration of the potential drop based crack growth and the associated J-R curve based on work of Sandia National Laboratories



- Normalized Voltage (V_i/V_{ref}) → Normalized sample voltage with a reference sample
- Normalized voltage plotted against the CMOD
- A linear equation is fitted to the "Early" portion of the curve
- A line with a 0.2% offset of the slope is applied → Nominally to cover the noise of the data
- Initiation is selected at the intersection of the 0.2% offset line with the normalized voltage
- Selected initiation point is checked in the load trace.

Figure 5.2 Illustration of the potential drop based initiation of crack from DNV Columbus

For Option B the $J_{x.x\ mm}$ value can be determined following the procedure of ASTM E1820 with the offset line (reference ASTM E1820 Figure A9.1 definitions) set at the respective Δa value. With this option if Δa is chosen > 0.05mm a supplementary test to demonstrate the crack stability at a chosen J value should be conducted such as by a constant load test or by assessment of the crack stability in the pipeline at the chosen J value.

When the unloading compliance approach is used to monitor crack growth it is usually not expected that accurate crack depth will be inferred from this method for short crack growth near $\Delta a \approx 0$ mm because the method relies on detecting the change in stiffness of the test sample with a growing crack. The resulting J-R curve is usually constructed by fitting a curve-fit function to the generated discreet J vs Δa points as illustrated in Figure 4.2 and the initial portion of the curve ($\Delta a \rightarrow 0$mm) is extrapolated. Because this method does not have the required resolution in the initiation region (near $\Delta a \approx 0$ mm) and by default the curve fit function fixes J =0 at Δa=0, an engineering definition of the threshold fracture toughness is required. Based on experience from wet sour service and from assessment of various test data performed in hydrogen gas, the toughness value set at Δa=0.05mm is recommended to be used as an engineering approach of the threshold fracture toughness at this point in time. Further comparative testing is required to calibrate the unloading compliance method based results with the potential difference based results. The $J_{0.05\ mm}$ value can be determined following the procedure of ASTM E1820 with the offset line (reference ASTM E1820 Figure A9.1 definitions) set at the Δa=0.05 mm value.

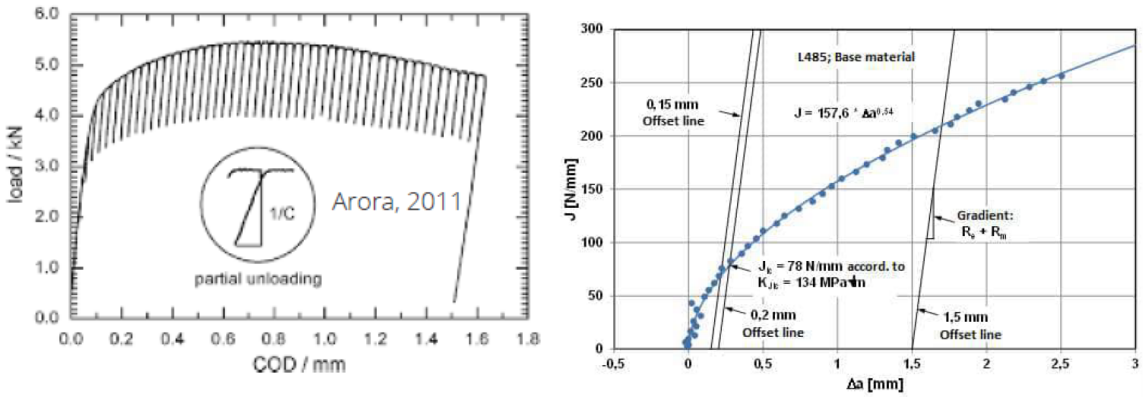


Figure 5.2 Illustration of the unloading compliance method and the associated example J-R curve from SyWestH2 project

Clause 5.3:
It is not required to generate the J-R curve beyond the crack growth depth required to establish the threshold fracture toughness.

Commentary to clause 5.3:
It is not recommended to use the rising portion of a J-R curve in the fracture assessment of a hydrogen gas pipeline due to a different crack growth mechanism (quasi – cleavage) in hydrogen compared to ductile void formation and growth in-air where a

longer extent of the J-R curve can be used. The same guidance is advised for wet sour conditions in DNV RP F108 Appendix C. With this consideration in mind there is no need for constructing the J-R curve beyond the crack depth required for establishing the threshold fracture toughness.
Generating a J-R curve for crack growth over several millimetres also takes a substantial length of time at appropriately selected slow loading rates.
It is suggested that for a potential difference based crack growth monitoring technique the fracture test can be stopped at the attainment of the maximum load in the test. This gives over 0.5mm of crack growth which is sufficient to define the transition from crack blunting to crack propagation.
For an unloading compliance based approach to monitor crack growth the requirements in ASTM E1820 or ISO 12135 need to be followed to generate sufficient data points over the sufficient length of crack growth for fitting the J-R curve and extrapolation to origin.

Clause 5.4:
The reporting shall follow the requirements of ANSI/CSA CHMC-1 and shall include the amount of crack growth Δa associated with the reported J_{th} value.

Conclusions

This document provides current best practice views on performing fracture toughness in hydrogen gas. It is intended to supplement the available standards, namely ANSI/CSA CHMC-1 and provide the current best practice for pipeline application specific guidance. Some clauses presented in this document may already be covered in other publications and standards and are intentionally repeated here for completeness to provide salient guidance in one place. Several gaps still remain to be resolved in the future. The document will be updated with new learnings periodically to strive towards the evergreen state of the art guidance document on the subject. Future revision will also include guidance on the fatigue crack growth and tensile slow strain rate (SSRT) testing in hydrogen gas.

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EPRG
European Pipeline Research Group e.V.
Ehinger Straße 200
D-47259 Duisburg
Germany

www.eprg.net