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## HYDROGEN PRESSURE CYLCING OF SUBSCALE PIPES TO SIMULATE FULL-SCALE TESTING OF TRANSMISSION PIPELINES

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### ABSTRACT

*Full-scale testing of pipes is costly and requires significant infrastructure investments. Subscale testing offers the potential to substantially reduce experimental costs and provides testing flexibility when transferrable test conditions and specimens can be established. To this end, a subscale pipe testing platform was developed to pressure cycle 60 mm diameter pipes (Nominal Pipe Size 2) to failure with gaseous hydrogen. Engineered defects were machined into the inner surface or outer surface to represent pre-existing flaws. The pipes were pressure cycled to failure with gaseous hydrogen at pressures to match operating stresses in large diameter pipes (e.g., stresses comparable to similar fractions of the specified minimum yield stress in transmission pipelines). Additionally, the pipe specimens were instrumented to identify crack initiation, such that crack growth could be compared to fracture mechanics predictions. Predictions leverage an extensive body of materials testing in gaseous hydrogen (e.g., ASME B31.12 Code Case 220) and the recently developed probabilistic fracture mechanics framework for hydrogen (Hydrogen Extremely Low Probability of Rupture, HELPR). In this work, we evaluate the failure response of these subscale pipe specimens and assess the conservatism of fracture mechanics-based design strategies (e.g., API 579/ASME FFS). This paper describes the subscale hydrogen testing capability, compares experimental outcomes to predictions from the probabilistic hydrogen fracture framework (HELPR), and discusses the complement to full-scale testing.*

**Keywords:** Hydrogen Testing, Fatigue, High-Pressure, Subscale Testing

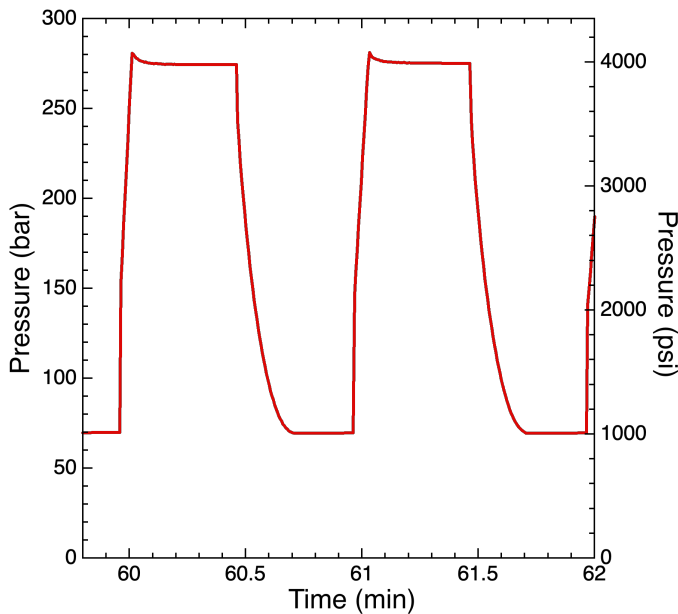
### 1. INTRODUCTION

Laboratory investigations of hydrogen-assisted fatigue crack growth and fracture resistance of line pipe steels has been reported in the literature [1-13]. These tests utilize a standardized test geometry extracted from pipe, such as the compact tension specimen, and standardized test methods to generate design information for hydrogen infrastructure at the laboratory scale. Examples of component and full-scale testing to evaluate failure in real-world configurations (i.e., pipe or pressure vessels) is less common and more costly to execute [14, 15]. Subscale testing of analogous configurations to large-scale hydrogen infrastructure offers an opportunity to perform laboratory scale tests that include important characteristics (e.g., geometry, defect configuration, stress state) of the full-scale application. This work demonstrates the potential for subscale component testing as a complement to both materials testing and full-scale testing.

Previous materials testing has shown that fatigue crack growth is relatively insensitive to the steel grade (such as X52 versus X65) and to microstructural characteristics [16]. Some modest differences in fatigue response exist in gaseous hydrogen, which likely reflect the modest differences also observed in air. These steels, however, show comparatively more variation in fracture resistance [17] as fracture properties are more sensitive to strength and microstructural characteristics than fatigue crack growth. In short, materials testing has generated foundational understanding of fatigue and fracture properties of steels in gaseous hydrogen. However, some engineering questions cannot be confidently addressed by idealized and carefully controlled tests in the laboratory. For example, real-world defects and damage can be challenging to quantify by analysis and standardized testing. Full-scale testing may offer an opportunity to evaluate dents and other defects, however, a framework for generalizing broad defects classes will be challenging with full-scale testing only, due to the resource intensive nature of testing large diameter pipe. Subscale testing

of small diameter pipe provides a platform to address engineering scale questions with comparatively less resources and to execute significantly more tests than can be achieved in full-scale testing. Moreover, insights from subscale testing can be used to inform and improve testing at the full-scale.

In this brief report, a system for testing relatively small diameter piping (60 mm) is described as a surrogate for full-scale testing of transmission pipe. Preliminary testing is described that compares pressure cycling of pipe with internal and external defects, respectively. These tests were conducted with gaseous hydrogen to assess the role of a pipe's internal surface condition (i.e., mill scale) on hydrogen-induced failure considering both internal and external defects. Additionally, the results are compared to fracture mechanics predictions.



**FIGURE 1: REPRESENTATIVE PRESSURE CYCLES FOR PIPE TESTING IN THIS STUDY. (TEST DATA FROM SPECIMEN E2)**

## 2. MATERIALS AND METHODS

A custom system was designed and constructed for pressure cycling pipe specimens. The pressure system is a closed loop system consisting of (1) a high-pressure reservoir (with pressure regulation for the desired pressure), (2) the test fixture, (3) a low-pressure reservoir, and (4) a compressor (with the suction side connected to the low-pressure reservoir and the outlet connected to the high-pressure reservoir). Feedback from a pressure transducer connected the test fixture controls valves on the inlet and outlet to the test fixture to enable a controlled and approximately trapezoidal pressure cycle, as shown in FIGURE 1.

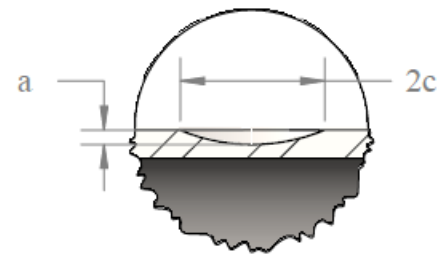
The test fixture consists of free-floating endcaps that seal against the outer diameter of the pipe specimen without contributing an axial load. When the system is pressurized, the endcaps can slide and react axially against strongbacks on either side of the pipe specimen, such that the pipe is not axially constrained, but pressure is maintained. The strongbacks are held

together with two tie rods, which carry the pressure load. Gas enters the pipe specimen through gas ports integrated into the endcaps. Additionally, the endcaps are designed with o-rings on the outside diameter to seal against a secondary vessel. The secondary volume is purged with nitrogen and captures the gas from the pipe when the specimen fails. The secondary volume is vented above the roofline of the facility, such that gases never enter the laboratory when the pipe specimens fail. Additionally, to aid the pressure cycling, an aluminum rod is inserted into the pipe specimen to fill the volume, thus testing requires less gas and compression to achieve the desired pressure cycle.

Test control and monitoring is achieved with an automated, computer control and data acquisition system designed specifically for the test system. The system controls a series of valves to produce the desired maximum and minimum pressure, as well as the dwell time for these pressures. The system includes data acquisition for recording pressure-time information throughout the test, as well as temperature and other signals, such as strain gauges attached directly to the pipe specimens.

Commercial-off-the-shelf pipe was used in this study: ASTM A53 Grade A, Type F (furnace-welded), nominal pipe size (NPS) 2, Schedule 40. The specified outside diameter (OD) and wall thickness (t) of this pipe are 60.3 x 3.91 mm (2.375 x 0.154 in). The minimum yield and tensile strength of Grade A pipe are 205 and 330 MPa (30 and 48 ksi), respectively. The exact composition of the tested pipe was not measured.

Pipe specimens were prepared in approximately 300 mm long sections. Two types of test specimens were prepared: one type with internal (I) notches and the other with external (E) notches. In both cases, two notches were prepared in the center of the length with one notch on the weld seam, and the other notch opposite (i.e., 180 degrees from the seam). Notches, also referred to as engineered defects, were machined using plunge electro-discharge machining (EDM). In both cases, the notches have a nominal depth (a) of 2 mm and a nominal notch length (2c) of 20 mm, where the length extends along the axial direction of the pipe. Therefore, the notch is principally loaded by the hoop stress during pressurization of the pipe. A schematic of the notch is shown in FIGURE 2. After notching the specimens, the ends of the pipe were prepared for o-ring seals by machining a reduced diameter. Other than the notch and the sealing surface at the ends of the pipe specimen, the pipe surfaces were undisturbed.



**FIGURE 2: CUT-AWAY OF THE NOTCH GEOMETRY IN THE WALL OF THE PIPE FOR AN EXTERNAL NOTCH, SHOWING THE DEPTH (a) AND THE LENGTH (2c).**

For all the testing in this study, the nominal minimum and maximum pressure of the cycles were 69 bar (1,000 psi) and 276 bar (4,000 psi) respectively. This pressure cycles corresponds to a stress ratio (R) of 0.25. The shape of the pressure cycles have evolved slightly with fine-tuning of the system; however, the basic cycle remained the same for all tests: about one minute per pressure cycle with pressure dwell at both minimum and maximum pressure as shown in FIGURE 1. The minimum and maximum pressure varied slightly throughout the test and from test to test. Generally, the average maximum pressure was within +3% of the target pressure with standard deviation of less than 1% of the mean value (in other words, the deviation in the maximum pressure from cycle to cycle was relatively small (1%), while the average pressure for all cycles was slightly greater than the target pressure, but within +3%). The average minimum pressure was generally within 1% of the target with a standard deviation of up to 2% of the mean. The standard deviation of all pressure cycles for a given test was typically less than 4 bar for both minimum and maximum pressure (in some cases less than 1 bar).

Tests were conducted with either pure hydrogen (99.9999% purity) or pure nitrogen source gases. The system was carefully purged to remove residual air with a series of pressurization-venting cycles using nitrogen. Prior to testing with hydrogen, the purging procedures were repeated with hydrogen; in the case of nitrogen tests, additional nitrogen purges were conducted such that the total number of purge cycles was the same for tests in hydrogen and nitrogen. The testing of pipe specimens with external defects was repeated twice with hydrogen, all other testing conditions represent single tests.

At the conclusion of testing (i.e., after failure), the pipes were cut to remove a section of the pipe around each defect. These sections were then heated in air at about 275°C for 30 minutes to tint the fracture surfaces. The heat-tinted sections were immersed in liquid nitrogen and broken open to reveal the fracture surface. This cryo-fracturing step ensures that the fracture surface can be revealed without substantial plastic deformation.

### 3. RESULTS

The number of cycles to failure for all pipe tests is summarized in Table 1. For external defects, testing with nitrogen resulted in an order of magnitude greater cycles to failure than the tests in hydrogen. The test with hydrogen was repeated, and the cycles to failure for the two tests are within about 4% of the average value. Testing of pipe with internal defects resulted in a substantially smaller number of cycles to failure in hydrogen, than pipe with external defects. In nitrogen, however, the number of cycles to failure was substantially greater for test specimens with internal (engineered) defects than for specimens with external defects.

Example fracture surfaces for both external and internal engineered defects are shown in FIGURE 3. The full wall thickness is shown in these images, although the perspective varies slightly such that the thickness dimension is distorted and not consistent for all images (but the physical thickness is the

same). The pair of images for each pipe specimen (E1 and I1) represent the two notches in the specimen: notch on the seam weld and the notch opposite the seam. In these two cases (and all others), the specimen failed at the notch opposite of the seam weld. The dotted red line marks the internal surface of the pipe. The arrow emphasizes the direction of crack propagation: outside to inside of the pipe for external engineered defects and inside to outside for internal engineered defects.

The light areas represent the cryo-fracture, produced after the pipe had failed. The darkest areas are the machined notch. The yellowish area (E1) and purplish area (I1) extending from the notches are the regions of fatigue crack propagation due to pressure cycling. A crack extends from the notch on the E1-seam but was arrested due to failure at the opposite notch. A crack did not initiate from the notch on the seam in I1 (bottom most image). In all cases, the crack extended from the notch profile without lengthening; in other words, the crack length (2c) appears to have remained approximately constant throughout the test. In fact, it appears that the crack extending from E1-seam notch did not extend to the ends of the notch, unlike the cracks that grew through wall.

**TABLE 1: SUMMARY OF TESTING CONDITIONS AND MEASURED PRESSURE CYCLES TO FAILURE.**

Specimen		Gas	Measured cycles to Failure	Estimated cycles to $a/t = 0.8$
internal	I1	Hydrogen	636	347
	I4	Nitrogen	18,880	13,229
external	E1	Hydrogen	1,097	–
	E2	Hydrogen	1,191	–
	E3	Nitrogen	13,356	–

### 4. DISCUSSION

#### *Surface condition*

The importance and role of the mill scale on hydrogen-assisted fatigue and fracture of real-world pipes motivated the comparison of failure in pipe specimens with internal and external engineered defects. In contrast, laboratory fatigue crack growth and fracture test specimens are precracked in air, prior to incorporation into the hydrogen environment. Thus, the crack surfaces have a relatively thin native oxide prior to immersion in the hydrogen environment. This oxidized surface is sometimes falsely considered a barrier to hydrogen uptake; published work shows that hydrogen can permeate through oxide surfaces [18], although the surface condition may affect the measurement of diffusion [19]. Once the crack extends in hydrogen, ‘clean’ metal surface is exposed to hydrogen, which is expected to promote

hydrogen uptake into the metal. Therefore, standard pre-cracked specimens do not necessarily capture the behavior of engineering surfaces prior to the initiation of a crack in hydrogen.

Pipe specimens with an internal notch can have a mostly intact mill scale on the inside of the pipe. Care was taken in the present specimens to minimize damage to the as-received mill scale inside the pipes. This mill scale in the internally notched pipe specimens is only disrupted at the notch (engineered defect). However, the surface of the notch is a recast layer (giving it a pebbly appearance) that is also not characteristic of a clean (albeit oxidized) metal surface. Therefore, the influence of the EDM surface is also an open question. When the specimen is pressurized, localized strain at the notch could crack the mill scale, thus revealing metal surface to hydrogen and facilitating hydrogen ingress into the metal. However, as mentioned, oxidized surfaces do not necessarily prevent hydrogen diffusion [18] as hydrogen can dissociate on iron oxide surfaces [20]. Even if cracking of the native oxide were required for hydrogen uptake, it seems unlikely that localized cracking the native oxide would expose enough metal surface to induce significant hydrogen effects on short laboratory time scales.

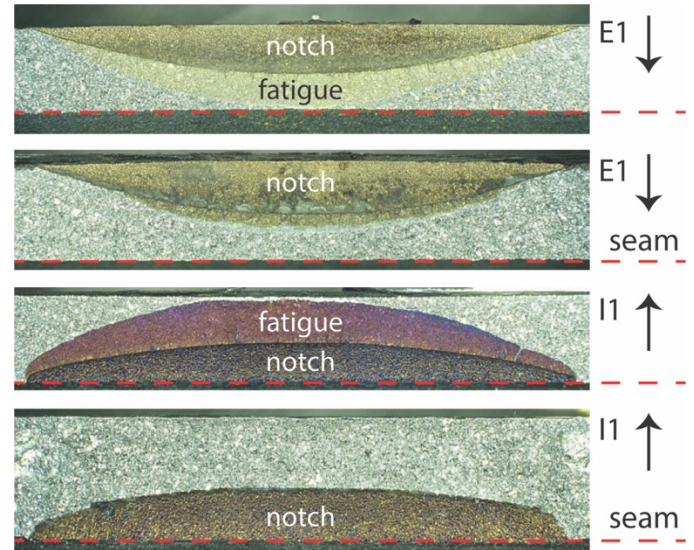
To probe these concepts, pipe with external defects were used to minimize the local damage of the native oxide. In this case, the internal mill scale remains undisturbed at the beginning of the test. Admittedly, the local strains may still be somewhat high on the internal surface opposite of an external defect, compared to without the external defect. Nevertheless, the externally notched specimen was used to evaluate the effectiveness of the mill scale on preventing hydrogen-assisted fatigue and fracture in the absence of engineering damage (e.g., an internal gouge or notch).

As the results in TABLE 1 demonstrate, hydrogen-assisted failure is apparent in test specimens both with internal surface defects and with external surface defects. In the case of the external engineered defect (notch), hydrogen must diffuse through the mill scale and the metal to reach the stress concentration at the external notch and induce a crack that then propagates inward toward the internal surface of the pipe. It is apparent from the images in FIGURE 3 that the crack evolves from the notch root in much the same way as for the internal notch. In the example shown, cracks formed at both external notches due to the pressure-induced stresses and propagated inwardly. The driving force for crack extension (characterized by the stress intensity factor,  $K$ ) will generally be highest at the deepest point of the crack, thus the crack should evolve at the deepest point and grow both in the depth direction and laterally as shown in the top three images of FIGURE 3. Failure also occurred in the sample pressure cycled with nitrogen, but the influence of hydrogen was clear, accelerating failure by an order of magnitude.

It is worth noting a few additional characteristics of the external notch. The boundary condition at the external surface of the pipe specimen is nominally zero hydrogen fugacity (provided no barrier to hydrogen recombination) as the outside surface is exposed to a nitrogen purged volume. Therefore, the hydrogen content near the notch root is relatively low, although a gradient

of hydrogen is expected, although the gradient will be affected by the stress field associated with the notch/defect. In contrast, the hydrogen content near the notch root of the internally notched specimen is directly exposed to hydrogen and potentially much higher. This description suggests that small amounts of hydrogen are effective at promoting hydrogen-assisted fatigue, consistent with materials testing [21]. These results are also notionally consistent with observation that precracking fracture specimens in air or in hydrogen have little, if any, effect on fracture tests in gaseous hydrogen [5]; in other words, native oxides on metal surfaces do not mitigate the influence of hydrogen. Similar conclusions can be drawn from the loss of tensile ductility in gaseous hydrogen [22] (irrespective of the surface strain, as strain is a necessary condition for fatigue and fracture with or without hydrogen).

In short, the undisturbed mill scale of the pipe specimens with external notches was insufficient to mitigate hydrogen-assisted fatigue. Therefore, it should not be hypothesized that mill scale or native oxides will prevent hydrogen-assisted fatigue and fracture.



**FIGURE 3: FRACTURE SURFACE AFTER CRYO-FRACTURING FAILED PIPE SECTIONS. THE RED DASHED LINE REPRESENTS THE INNER SURFACE OF THE PIPE, AND THE ARROWS SHOW THE DIRECTION OF CRACK PROPAGATION. THE PERSPECTIVE DIFFERS SLIGHTLY IN THE IMAGES (SURFACE IS INCLINED WITH RESEPECT TO IMAGING PLANE), SUCH THAT THE WALL THICKNESS IS NOT TO SCALE. IN BOTH SPECIMENS (E1 AND I1), FAILURE OCCURRED ON THE NOTCH OPPOSITE THE SEAM WELD.**

#### *Fatigue life predictions*

In this study, commercial ASTM A53 pipe was used, which is significantly different from transmission pipe. In particular, the strength of ASTM A53 pipe is substantially lower than API 5L transmission pipe, for example. However, previous work has shown that fatigue crack growth in gaseous hydrogen is relatively insensitive to the strength of the steel [16]. Moreover, the microstructural characteristics of ASTM A53 pipe is similar

to API 5L steels. For these reasons, we anticipate that ASTM A53 steel will display similar fatigue crack growth in gaseous hydrogen as API 5L steels. Therefore, the hydrogen-assisted fatigue crack growth design curves from ASME B31.12 Code Case 220 can be used to predict the life of ASTM A53 pipe.

Following previous work on full-scale testing of steel pressure vessels [14, 15], a simplified formulation was used to predict the fatigue life, using the closed form solution for the stress intensity factor ( $K$ ) of an elliptical crack oriented in the longitudinal orientation from Anderson [23]. For these calculations, the engineered defect/notch is assumed to be a sharp crack. At the time of this article, this closed-form  $K$ -solution is hard coded into an open-source software called HELPR (Hydrogen Extremely Low Probability of Rupture), therefore this tool was used to make life predictions for the geometry of pipe and defects used in this study (additional  $K$ -solutions are actively being developed for inclusion into HELPR, including  $K$ -solutions from API 579/ASME FFS-1). Since this  $K$ -solution is only valid for fractional crack depth less than 80% of the wall thickness ( $a/t < 0.8$ ), for the purposes of this assessment, the failure criterion is established when the crack reaches this depth. The predictions from HELPR for the conditions of testing are shown in Table 1. However, at present, HELPR assumes the aspect ratio of the crack ( $a/2c$ ) remains constant. As shown in Figure 3, a better approximation would be  $2c$  remains constant. The life prediction was repeated analytically with this assumption and the predicted cycles to failure was almost 40% greater (485 cycles to  $a/t = 0.8$ , compared to 347 cycles from HELPR). Longer predicted life is expected for a shorter crack with constant  $2c$ , compared to a lengthening crack with constant  $a/2c$ , since the driving force for crack extension is greater for longer cracks. In both cases, the prediction is still conservative relative to the measured cycles to failure of 636.

We attribute the difference between the predictions and the measured cycles to failure to the cycles required to initiate a growing crack from the engineered defect (notch), similar to observation of full-scale testing of pressure vessels with hydrogen [14, 15]. A similar result is obtained for pressure cycling with nitrogen (assuming crack growth in air), except the life prediction is more than an order of magnitude greater (Table 1).

HELPR does not yet include solutions for the external crack. However, simple solutions for infinite (long) cracks predict that an external crack will have nearly twice the life as an equivalent crack on the internal surface (all else being equal). While not quantitative, this difference approximately scales with the measured cycles to failure of the specimens with external engineered defects compared to the internal defects in gaseous hydrogen. In contrast, the measurements in nitrogen do not seem to support the hypothesis that external cracks will have a longer life. Of course, the number of tests here is limited, and the role of intrinsic defects in the pipe is unknown but could also play a role. Alternatively, the initiation of the crack in nitrogen compared to hydrogen may play a role; a recent study suggests, rather counter intuitively, that hydrogen may suppress crack

initiation [24]. Finally, it should be noted hydrogen must diffuse through the pipe wall to the defect to induce hydrogen-assisted fracture, which also contributes for the difference between internal and external defects when testing in hydrogen. Future testing on different time scales will help clarify the importance of diffusion on failure from external defects.

## 5. SUMMARY

Full-scale testing of hydrogen pipelines is resource intensive and challenging. Testing pipe at the subscale is proposed in this study as an alternative to full-scale testing. To demonstrate the utility of testing at subscale, we evaluate the role of mill scale on mitigating hydrogen-assisted fatigue by testing pipe with internal and external engineered defects respectively. For the configuration employed in this study, the number of pressure cycles to achieve failure in a pipe with hydrogen was relatively similar with internal and external engineered defects (factor of 2 difference) and significantly less (factor of  $>10$ ) than when tested with nitrogen. Therefore, oxides and mill scale do not mitigate the effects of gaseous hydrogen exposure, since the mill scale was intact during the testing of specimens with external defects. These conclusions could not be achieved with conventional laboratory scale testing (since specimens do not maintain mill scale); moreover, special steps to address the mill scale in full-scale testing appear unnecessary. Finally, the measured cycle life is compared to fracture mechanics predictions and shown to be conservative.

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