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MATERIALS COMPATIBILITY CONCERNS FOR HYDROGEN BLENDED INTO NATURAL GAS

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ABSTRACT

Hydrogen additions to natural gas are being considered around the globe as a means to utilize existing infrastructure to distribute hydrogen. Hydrogen is known to enhance fatigue crack growth and reduce fracture resistance of structural steels used for pressure vessels, piping and pipelines. Most research has focused on high-pressure hydrogen environments for applications of storage (>100 MPa) and delivery (10-20 MPa) in the context of hydrogen fuel cell vehicles, which typically store hydrogen onboard at pressure of 70 MPa. In applications of blending hydrogen into natural gas, a wide range of hydrogen contents are being considered, typically in the range of 2-20%. In natural gas infrastructure, the pressure differs depending on location in the system (i.e., transmission systems are relatively high pressure compared to low-pressure distribution systems), thus the anticipated partial pressure of hydrogen can be less than an atmosphere or more than 10 MPa. In this report, it is shown that low partial pressure hydrogen has a very strong effect on fatigue and fracture behavior of infrastructure steels. While it is acknowledged that materials compatibility with hydrogen will be important for systems operating with high stresses, the effects of hydrogen do not seem to be a significant threat for systems operating at low pressure as in distribution infrastructure. In any case, system operators considering the addition of hydrogen to their network must carefully consider the structural performance of their system and the significant effects of hydrogen on structural integrity, as fatigue and fracture properties of all steels in the natural gas infrastructure will be degraded by hydrogen, even for partial pressure of hydrogen less than 0.1 MPa.

1. INTRODUCTION

Gaseous hydrogen is an important industrial chemical and emerging as a potentially carbon-free fuel. To enable broader use of hydrogen as a fuel and energy carrier, efficient and economic methods are needed to convey hydrogen from sites of production to sites of usage. This is not a new concept; hundreds of miles of dedicated hydrogen pipelines exist around the world [1]. As hydrogen technologies grow, hydrogen pipeline networks will likely grow as well. In the near term, however, many projects around the world are investing in concepts to blend hydrogen into natural gas infrastructure [2].

Gaseous hydrogen is known to degrade fatigue and fracture properties of structural steels; therefore, ASME developed a code for hydrogen pressure piping: ASME B31.12. This code provides guidance for consideration of structural integrity in hydrogen environments, pointing to the ASME Boiler and Pressure Vessel Code (Section VIII, Division 3) for testing and fracture mechanics assessment of pipelines. Despite literature data, anecdotal misinformation persists suggesting that a critical hydrogen content in natural gas is needed before the hydrogen becomes a threat to the structural integrity of the system. This perception ignores the fundamental reality that hydrogen degrades fatigue and fracture properties at any concentration and the physics of degradation depend on the fugacity (partial pressure) of hydrogen.

Structural integrity in a system depends on both the materials properties as well as the mechanical service conditions (e.g., stress) and the details of the service environment (e.g., pressure, temperature, impurities). In this report, we evaluate the effect of low partial pressure of hydrogen on fatigue and fracture of pipeline steel, and we consider the impact of hydrogen-natural gas blends on structural integrity of a transmission pipeline. In addition, this information is extrapolated to distribution piping. Transmission and distribution systems are distinguished by

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operational conditions, namely pressure, as well as the materials of construction. These differences will be described, and the implications assessed in the context of structural integrity.

2. MATERIALS AND TEST METHODS

2.1 Materials

API Grade X52 steel was utilized in this study. Specimens were extracted from pipe with an outside diameter (OD) of 324 mm and wall thickness (t) of 12.7 mm. The composition of this steel was nominally Fe-0.87Mn-0.06C (w%) as reported elsewhere [3]. The yield and tensile strength of this steel are reported as 429 and 493 MPa respectively. The microstructure is predominantly polygonal ferrite with approximately 10% pearlite.

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2.2 Testing Environment

Testing was conducted in gaseous nitrogen with 3% hydrogen (N_2-$3H_2$ by volume). This gas mixture was chosen for testing to simulate a blended gas environment while eliminating the confounding effects of impurities, such as oxygen. Natural gas generally contains impurities (such as O_2 and CO) that may mitigate some of the effects of hydrogen (although not all); therefore, the nitrogen-hydrogen represents a 'worst case' for the tested partial pressures of hydrogen. Tests in the N_2-$3H_2$ gas mixture were conducted at total pressure of 21 MPa (3,000 psi) and 3.4 MPa (500 psi), representing hydrogen partial pressure of approximately 0.6 MPa and 0.1 MPa, respectively.

2.3 Fatigue and Fracture Test Methods

Fatigue testing was conducted following the procedures in ASTM E647 for fatigue crack growth testing. The compact tension geometry was utilized for this testing with W = 26.4 mm and B = 9.5 mm. Specimens were side grooved prior to precracking, resulting in reduced thickness (B_N) of 8.4 mm. Fatigue testing was conducted with load ratio (R) of 0.1 and frequency of 1 Hz.

Fracture testing was conducted at the conclusion of fatigue testing without removal of the specimen from the test environment. Load was applied monotonically following the procedures in ASTM E1820 and the direct current potential difference method (DCPD) was utilized to monitor crack length. The elastic-plastic fracture resistance was determined from the J-R curves at the intersection with the 0.2 mm offset construction line. The measured values of plane-strain fracture resistance (J_{IC}) are converted to stress intensity factor following standard practice (ASTM E1820) and denoted K_{JIC}.

Additional details of testing in gaseous hydrogen can be found in Ref. [4].

3. RESULTS AND DISCUSSION

3.1 Hydrogen-Assisted Fatigue Crack Growth

The measured fatigue crack growth rate (da/dN) of X52 in N_2-$3H_2$ at total pressure of 21 and 3.4 MPa are provided in Figure 1 as function of the stress intensity factor range (ΔK). The hydrogen partial pressures in these tests are relatively low compared to the special requirements in ASME Section VIII, Division 3 for high-pressure gaseous hydrogen service (Article KD-10 applies to welded vessels with pressure greater than 17 MPa and non-welded construction with pressure greater than 41 MPa). However, hydrogen partial pressure as low as 0.1 MPa results in substantially higher fatigue crack growth rates than in air, by more than an order of magnitude in the high ΔK limit. Additionally, the fatigue crack growth response in the N_2-$3H_2$ mixed gas shows the classic two-part power-law behavior of da/dN versus ΔK (fatigue crack growth curve) that is typically observed for tests conducted in pure gaseous hydrogen environments [5]. At low ΔK, the fatigue curve has a steep slope, whereas at high ΔK, the fatigue curve is comparatively shallower (and similar to the slope in air). The transition between these two portions of the fatigue curve is often referred to as the 'knee'. The 'knee' depends on numerous factors (including load ratio and pressure); for these tests, the transition occurs around ΔK = 20 MPa $m^{1/2}$. In short, fatigue crack growth rate of this X52 API pipeline grade steel is substantially affected by hydrogen at partial pressure as low as 0.1 MPa.

For comparison, literature data [3] evaluated in pure hydrogen at pressure of 21 MPa are also shown in Figure 1. The knee for this higher-pressure data occurs at ΔK closer to 12 MPa $m^{1/2}$. Interestingly, the fatigue curves at pressure of 21 MPa for the pure hydrogen condition and for both N_2-$3H_2$ mixed-gas conditions converge for ΔK greater than 20 MPa $m^{1/2}$. This is consistent with the report of Meng et al. [6], where they determined no significant effect of hydrogen partial pressure on fatigue crack growth of X42 steel in N_2-H_2 mixtures at total pressure of 12 MPa. This trend also demonstrates, as previously reported [5], the broader pressure independence of fatigue crack growth of low-strength ferritic steels at high ΔK (above the 'knee').

As reported in Ref. [5], a wide range of pressure vessel steels show sufficiently similar fatigue crack growth behavior in gaseous hydrogen that a universal design curve was defined in Code Case 2938 of the BPVC. The design curve consists of two power-law relationships characterizing the two regions described above. These relationships account for the load ratio as well, such that the fatigue crack growth in hydrogen can be estimated for any R between at least 0.1 and 0.7. Moreover, a pressure term was proposed for the design curves in Code Case 2938 that adapts the relationship to lower pressure. The pressure-compensated design curves from Code Case 2938 show remarkable consistency with measured fatigue crack growth behavior of a range of common API grade pipeline steels (see Figures 7 and 8 in Ref. [5]).

The pressure effect in the design curves is represented by an empirical scaling term based on the square root of fugacity. This scaling follows from thermodynamic equilibrium since the concentration of hydrogen in a metal is proportional to the square root of the fugacity [7]. The fugacity is the thermodynamic pressure, which represents the activity of hydrogen dissolved in the metal for known gaseous boundary conditions. Curiously, the pressure term applies only to the power law for the low ΔK

regime (below the knee); the resulting design curves are shown in Figure 1 (dashed lines) for the two cases: (1) pure hydrogen at pressure of 21 MPa, and (2) the N_2-$3H_2$ gas mixture at the same pressure, representing hydrogen partial pressure of 0.6 MPa. For ΔK greater than the knee, the design curve is independent of fugacity (i.e. pressure), consistent with the measured fatigue crack growth data. It is important to emphasize that the fugacity is dependent on both the partial pressure of hydrogen and the total pressure; moreover, characterization of the hydrogen environment as a volume percentage is not sufficient to characterize the effect of hydrogen, since the fatigue response scales with fugacity (partial pressure) of hydrogen, not percentage. Details of determining the fugacity of pure hydrogen and hydrogen blends are provided in the Appendix.

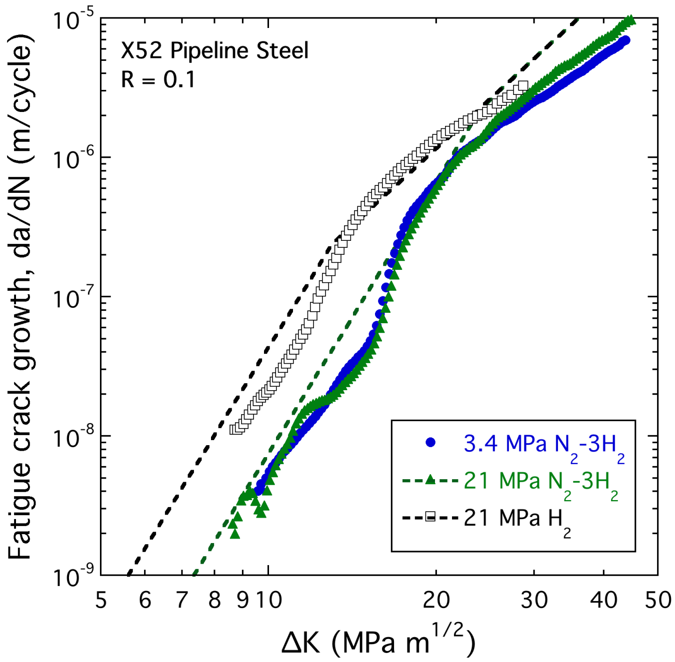

FIGURE 1: FATIGUE CRACK GROWTH CURVES FOR X52 PIPELINE STEEL IN GASEOUS HYDROGEN ENVIRONMENTS. DASHED LINES REPRESENT PRESSURE-COMPENSATED DESIGN CURVES FROM CODE CASE 2938.

The difference in the fatigue response for the pure hydrogen and the mixed gas (both at total pressure of 21 MPa) shows a pressure dependence on fatigue crack growth (Figure 1). However, the two mixed gas cases (hydrogen partial pressure of approximately 0.1 and 0.6 MPa respectively) are not significantly different. It may be that below some critical partial pressure of hydrogen, fatigue crack growth is relatively insensitive to hydrogen partial pressure (but characterized by higher pressure). Alternatively, the similarity may be coincidental and reflect uncertainty in the measurement or gas quality. In any case, the hydrogen effect is clearly evident.

In the discussion above, we idealize the fatigue response as a two-part power law, but the fatigue curves of the mixed gas in the low ΔK regime show more curvature than a simple power law. Such curvature is generally absent from testing in pure hydrogen. One possible explanation is oxygen impurities, which tend to mitigate the effects of hydrogen (reducing fatigue crack growth rates); however, tests with controlled impurities tend to have a much larger effect. We believe this behavior is related to a combination of low pressure and oxygen impurity, although more testing is required to quantify these relationships. Regardless of these subtle perturbations in the fatigue curves, to first order, the fatigue response in X52 in the mixed gas follows the basic trends from established empirical predictions of fatigue crack growth rates.

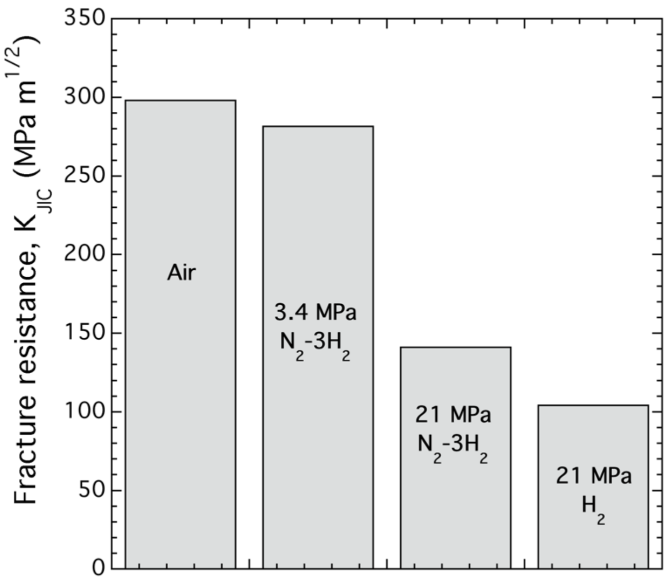

FIGURE 2: FRACTURE RESISTANCE (K_{JIC}) OF X52 PIPELINE STEELS IN GASEOUS HYDROGEN ENVIRONMENTS.

3.2 Hydrogen-Assisted Fracture

The elastic-plastic fracture resistance of the X52 depends on partial pressure of hydrogen as shown in Figure 2. The air reference data and pure hydrogen data are from Ref. [8]. The fracture resistance of pipeline steels in terms of K was hypothesized in the literature to be inversely proportional to the equilibrium concentration of hydrogen based on data at higher pressure [9]. Since hydrogen concentration is proportional to the square root of fugacity (Sievert's Law), K would be proportional to $f^{-1/2}$. However, if the elastic-plastic fracture resistance (J) is assumed to scale inversely with the equilibrium hydrogen concentration, then $K \propto f^{-1/4}$ (since K is proportional to the square root of J). The data in Figure 2 follow approximately this scaling ($K \propto f^{-1/4}$), which imply that fracture resistance is a steep function of fugacity/pressure at low partial pressure of hydrogen, and a relatively shallow function of fugacity/pressure for higher partial pressure of hydrogen (greater than about 2 MPa). A similar trend was observed in the literature for an API X70 steel [10]: the fracture resistance was substantially reduced in hydrogen at low pressure, but the difference in fracture resistance was modest for hydrogen partial pressure between 0.1 MPa and 8 MPa. More testing at different hydrogen partial pressures and testing rates (as well as replicate testing) will be needed to clarify the fugacity/pressure dependence of fracture resistance in hydrogen environments. However, we can state definitely that even hydrogen partial pressure of 0.1 MPa (represented by the measurement in N_2-$3H_2$ mixed gas at pressure of 3.4 MPa) has a measurable effect on fracture resistance. Fracture resistance is further decreased at higher partial pressure of hydrogen. That is not to say that the materials become brittle; in pure hydrogen at pressure of 21 MPa the fracture resistance is about 100 MPa $m^{1/2}$ (units of K), consistent with similar measurements of pipeline steel at this pressure [4, 9, 11]. Incidentally, this fracture resistance is greater than the minimum fracture resistance required by ASME B31.12.

3.3 Structural Integrity Assessment: Transmission Pipelines

The laboratory fatigue and fracture testing of X52 demonstrates significant degradation of mechanical properties when these materials are concurrently exposed to gaseous hydrogen. However, these assessments can be misleading in the context of structural integrity. In general, the stress in pressurized cylindrical shells (pipes) can be relatively low, thus hydrogen embrittlement may not be a substantial structural concern even in the presence of large defects. Consider, for example, the X52 pipe from which the test specimens were extracted in this study. For the purposes of a simple structural analysis, we consider a maximum pressure of 10 MPa (1,450 psi) and a minimum pressure of 5 MPa (725 psi), corresponding to a pressure differential (ΔP) of 5 MPa. This maximum pressure induces a hoop stress in the pipe of about 25% of the reported tensile strength of the steel, which is a reasonable operating condition. For simple demonstration purposes, we assume a 'thumbnail' flaw with a 3:1 aspect ratio as prescribed in ASME BPVC Section VIII, Division 3 for fracture mechanics assessment. The driving force for crack growth (ΔK) is calculated based on these boundary conditions using the closed form solutions of the stress intensity factor (K) for a thumbnail crack from Ref. [12]. The evolution of the crack depth (a) is determined by numerical integration of the crack growth per cycle (da/dN) from the design curve described in Ref. [5] and corrected for the maximum pressure (10 MPa).

The evolution of the initial defect is shown in Figure 3 for the conditions described above and for two initial defect sizes of 25% and 50% of the wall thickness (a is defect depth, and t is thickness). These starting defects are exceedingly large from a practical standpoint, but this analysis serves to demonstrate the integrity of this pipeline for these operating conditions. An initial defect of 25% of the wall is essentially stable and will not significantly grow during the 100,000 cycles shown in Figure 3. An initial defect of 50% of the wall will grow over this time scale, extending to about 85% of the wall thickness. To place this into context, 100,000 cycles represents over 250 years at 1 cycle per day, meaning the pressure would cycle between 10 and 5 MPa once per day. Moreover, this analysis assumes pure hydrogen at total pressure of 10 MPa, which presents a greater hydrogen partial pressure than a pressure medium of 20% hydrogen in natural gas.

Additionally, a sizeable through-wall crack would be required for K to exceed the fracture resistance of the material in hydrogen. A thumbnail crack at 80% of the wall thickness equates to a driving force (K) of <25 MPa $m^{1/2}$ compared to the fracture resistance of ~100 MPa $m^{1/2}$ in hydrogen (at hydrogen partial pressure of 21 MPa). In simple terms, the resistance of the material to crack extension is more than 3 times the 'driving force' applied by the pressure on an almost through-wall crack (with the thumbnail configuration). In other words, the pipe will not rupture in hydrogen due to internal pressure, even if a fatigue crack grows essentially through the wall.

This analysis demonstrates that the X52 pipe configuration described herein is not compromised for pure hydrogen service and reasonable operating conditions despite the material properties being strongly degraded by exposure to hydrogen environments. Of course, the specifics matter, meaning the structural integrity of piping and pipelines in hydrogen environments will depend on the environmental and mechanical operating conditions and specific design requirements for the pipe. Additionally, other configurations, geometries and scenarios may be important, and this simple analysis of a cylindrical shell (or pipe) should not be considered a substitute for a comprehensive system analysis. For example, in this simple analysis the effect of welded microstructural and residual stresses was not considered (hydrogen-assisted fatigue and fracture properties of pipeline welds can be found in Refs. [8, 13-16]).

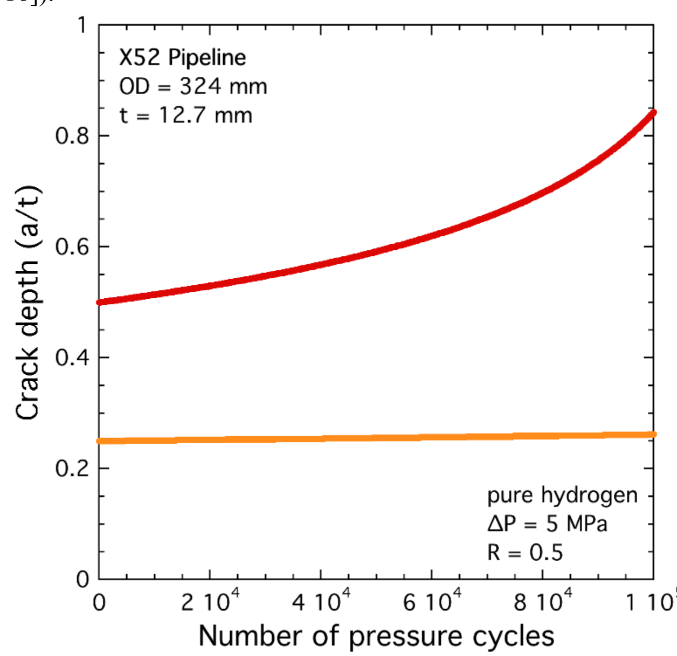

FIGURE 3: CRACK EVOLUTION IN X52 PIPE PRESSURE CYCLED WITH PURE GASEOUS HYDROGEN BETWEEN 5 AND 10 MPA WITH INITIAL DEFECT DEPTH OF: 25% OF THE WALL THICKNESS (ORANGE) AND 50% OF THE WALL THICKNESS (RED).

3.4 Structural Integrity Assessment: Distribution Piping

The analysis of transmission pipe suggests that transmission of high-pressure gaseous hydrogen is entirely feasible. Distribution systems differ in that the pressure is much lower and the pipes are much smaller. Both characteristics generally reduce the stress in the materials making them less susceptible to pressure-driven failure. We demonstrate this by considering standard pipe with nominal outer diameter of 168 mm and wall thickness of 7 mm (corresponding to schedule 40, 6-inch Nominal Pipe Size (NPS), ASTM A53). Pressure in transmission systems is generally very low (often <1 MPa), but we will consider an excessively high pressure of 3.4 MPa. For a typical ASTM A53 Grade A black pipe material used for natural gas service, the hoop stress for this pipe dimension and pressure is a little over 10% of the specified minimum tensile strength (and <20% of the specified minimum yield strength). The fatigue and fracture properties of black pipe in gaseous hydrogen are similar to API X52 in this study [17]. For the purpose of these simple estimates, we assume that black pipe has the same fatigue and fracture properties in hydrogen as API grades discussed above.

To estimate the structural integrity of black pipe in gaseous hydrogen service, a similar fracture mechanics assessment is conducted as for the transmission pipe. First, consider the resistance of the pipe to rupture. For a thumbnail defect in this pipe with a depth of 80% of the wall, the maximum driving force (K) is a little over 5 MPa $m^{1/2}$. In other words, the mechanical 'force' on the crack is an order of magnitude less than the material's resistance to crack extension assuming the properties of X52 in gaseous hydrogen. Thus, hydrogen-induced rupture is not a threat to this pipe for these conditions.

To assess fatigue, we consider complete depressurization as the lower bound of the fatigue cycle. This is an unrealistic condition, since a distribution system is rarely depressurized, but it represents an absolute worst-case situation. The ΔK in this case is the same as the K evaluated above, which is exceptionally small for unreasonably large defects: ΔK ~5 MPa $m^{1/2}$ for a thumbnail defect extending to a depth of 80% of the wall thickness. Even under this condition the defect will not advance over any reasonable timeframe, since da/dN is on the order of 10^{-10} m per pressure cycle (ΔP = 3.4 MPa). In short, it is difficult to imagine a scenario where hydrogen will enable fatigue crack growth in a typical distribution pipe configuration. Other system configurations (such as elbows with significant stress concentrations for example) may need further consideration, but the stresses appear to be so low as to be inconsequential. Therefore, external loading and damage is a more likely threat to these structures. Whereas hydrogen could amplify external threats, since hydrogen clearly degrades fracture properties, industry has operated hydrogen transmission and distribution systems for many decades without issue.

4. CONCLUSIONS

In this brief study, the fatigue crack growth rate and fracture resistance of X52 pipe were measured in N_2-$3H_2$ mixed gas. These properties are substantially degraded in this environment compared to air and generally depend on the partial pressure of hydrogen. At high driving force (i.e., high ΔK), however, the fatigue crack growth is independent of pressure and an order of magnitude greater than in air, even for hydrogen partial pressure as low as 0.1 MPa. Overall, the basic trends on fatigue and fracture due to testing in this mixed gas environment follow the trends established in pure hydrogen at high pressure. In particular, the fatigue crack growth rate in low partial pressure can be predicted from design curves in the ASME BPVC (Code Case 2938), at least to hydrogen partial pressure of about 0.6 MPa.

Whereas the fatigue and fracture properties are clearly degraded in low-pressure hydrogen environments, simple assessment of structural integrity of both transmission and distribution pipes show that the structural integrity of the pipe can be maintained with respect to hydrogen pressure-induced failure. Hydrogen may play a role in failure that is induced by other factors, such as welding or external loading and damage, but such scenarios were not analyzed in this study.

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Appendix

The thermodynamic behavior of an ideal gas is characterized by the partial pressure of gas. However, the fugacity characterizes the thermodynamic behavior of real gases. The fugacity of the gas depends on the equation of state. Hydrogen is a well-behaved, non-ideal gas that can be characterized by the Abel-Noble equation of state over a wide range of temperature and pressure [7, 18]. The fugacity of a single-component, Abel-Noble gas is expressed as:

$$\frac{f}{P} = \exp\left(\frac{P}{RT}b\right) \tag{A.1}$$

where f is the fugacity, P is the pressure, R is the universal gas constant, T is temperature and b is the co-volume constant (= 15.84 cm^3/mol for hydrogen).

For an ideal mixture of real gases, the fugacity of the i-th component is related to the fugacity at the total pressure by the mole fraction of the i-th component (x_i) [18]:

$$f_i = x_i f \quad \text{(A.2)}$$

Combining these two equations gives:

$$\frac{f_i}{P} = x_i \exp\left[\left(\frac{P}{RT}\right) b_i\right] \quad \text{(A.3)}$$

where P is the total pressure. It is important to emphasize that the mole fraction of the i-th component depends on the partial molar volume (v_i), but for non-ideal gases the compressibility (Z_i) must also be considered as:

$$x_i = \frac{{p_i}/{Z_i}}{\Sigma_j\left({p_j}/{Z_j}\right)} = \frac{v_i}{\Sigma_j(v_j)} \quad \text{(A.4)}$$

where the compressibility for the Abel-Noble equation of state is: $Z_i = 1 + b_i\left(\frac{p_i}{RT}\right)$. Since $P > p_i$ in a gas mixture and $b > 0$ for a non-ideal gas, the fugacity in a gas mixture will be greater than the fugacity of the pure gas at the same pressure. Since hydrogen effects are generally pressure/fugacity dependent, hydrogen in a gas mixture has a greater activity than the hydrogen by itself. The thermodynamic effect to enhance the potency of hydrogen in gas mixtures will generally be small in practice, even though it can amount to 10-25% in a relative sense ($f/P \sim 1$ for pure hydrogen at pressure of 0.6 MPa, but $f_H/P_H \sim 1.2$ for N_2-$3H_2$ at total pressure of 21 MPa).

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