Recent Activities in ISIJ HLP Research Committee Corrosion Working Group: Proposal of pH Buffer Test Solution for Fitness-For-Purpose HIC Evaluations

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The HLP (High-strength Line Pipe) Research Committee in the Iron and Steel Institute of Japan (ISIJ) has investigated the major issues of line pipes from the viewpoints of corrosion and fracture. Fitness-For-Purpose (FFP) evaluations for Hydrogen Induced Cracking (HIC) have been the subject of considerable study in recent years. FFP is the idea that appropriate materials are chosen depending on the severity of the use environment. Therefore, it is possible that the applicable range of material strength and the flexibility of manufacturing can be expanded by establishing the FFP evaluation approach. This paper reviews the recent activities of the HLP Corrosion Working Group during the past few years.

The current status of the conventional HIC test method and FFP HIC evaluation methods were over-viewed. Based on the results of a variety of experiments, the HLP Research Committee proposed a HIC test solution for FFP evaluations in the NACE Task Group. The proposed solution is a highly concentrated acetate buffer solution that is 5%NaCl+0.93N – (CH₃COOH+CH₃COONa), and is expected to be adopted as the optional solution in NACE TM0284. The solution displayed an excellent pH buffering capacity during a long-term HIC test in comparison with the conventional HIC test solutions. The corrosion rate and hydrogen permeation behavior of the steel specimen in the solution were similar to those in the conventional solutions.

KEY WORDS: the HLP Research Committee; line pipe; HIC; Fitness-for-Purpose; pH buffer.

1. Introduction

The HLP (High-strength Line Pipe) Research Committee of the Iron and Steel Institute of Japan (ISIJ) was established by steel manufacturing companies in Japan in 1978 and started to investigate arrestability against propagation of shear fracture of line pipes by conducting full scale burst tests. At present, the HLP Research Committee has two working groups, the Corrosion WG and Fracture WG. The HLP Committee has investigated several common issues of line pipes from the viewpoints of corrosion and fracture to support the development of new steels and pipe products and evaluation methods.

With continuing development of oil and gas resources, line pipes are exposed to a variety of corrosive environments. Natural gas and oil transported by pipeline often contain hydrogen sulfide and water as contaminants, and hydrogen generated by corrosion reactions involving these

substances is aggressively absorbed into the steel due to the poisonous effect of hydrogen sulfide. The absorbed hydrogen atoms diffuse in the steel and cause various types of environmental cracking, including Hydrogen Induced Cracking (HIC), Sulfide Stress Cracking (SSC) and Stress Oriented Hydrogen Induced Cracking (SOHIC). Damage of pipes due to those phenomena can lead to suspension of pipeline operation. In particular, HIC is a major issue for line pipes laid in the sour environments.

Steel manufactures have made enormous efforts to develop sour resistant line pipe. Advance of technologies in steelmaking process brought cleanliness of slab, and contributed improvement of HIC resistance. Continuous casting process and Thermo Mechanical Control Process (TMCP) also have played an important role to control centerline segregations and microstructure in the development. Sour resistant pipes of API X65 grade have been used in many recent pipeline projects throughout the world, and the development of X70 or higher grades for sour service has also been reported.^{1–3)}

The Corrosion WG of the HLP Research Committee con-

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ducted full scale pipe test in 1980s. Hydrogen permeation behavior and HIC cracking were investigated under high pressure gas conditions that simulated pipeline operation. Based on the correlation of HIC damage between full scale test and laboratory test, it was found that laboratory test was more server than full scale test.⁴⁾ HIC evaluation method and external stress corrosion cracking have been investigated by following previous studies. In recent years, the development of a HIC evaluation method for Fitness-For-Purpose (FFP) has been the major project of the HLP Corrosion WG.⁵⁻⁸⁾ This paper presents an overview of evaluation methods for HIC and the background of the recent activities of the HLP Corrosion WG, and reviews the achievements of the study of the HIC test solution for FFP.

2. Hydrogen Induced Cracking (HIC) and HIC Evaluation Methods

HIC is the major issue of line pipes exposed to sour environments containing H_2S . Cracking occurs around the segregation zone and inclusions in the steel, and susceptibility to cracking has a strong relationship with local hardness, which is determined by the chemical composition and microstructure of the steel. Therefore considerable efforts have been carried out by steel manufactures to develop sour resistant steels and pipes. Decrease in sulfur and calcium treatment in steel making process significantly improved HIC resistance due to suppression of elongated MnS formation. Reduction of inclusions and impurities by slag control made it possible to produce highly clean steels that had low HIC susceptibility due to less crack initiation sites.⁹⁻¹¹⁾ Along with decrease in Manganese and Phosphorous contents, the soft reduction technology in continuous casting process was applied to decrease centerline segregation of the slab.¹²⁾ Those advanced technologies in steelmaking and continuous casting processes extraordinary contributed to development of sour resistant materials for line pipe. The TMCP adopted with accelerated cooling system enable to produce the steels with favorable homogeneous microstructure that satisfies both mechanical properties and sour resistance.¹³⁾

In order to evaluate the HIC susceptibility of steels and pipes, the HIC test specified in NACE TM0284 is widely used as the standard method. NACE TM0284 was first standardized in 1984 and was later revised as TM0284-2011, which is its present form.¹⁴⁾ Solution A (5.0wt%NaCl+0.5wt%CH₃COOH) under 0.1 MPa H_2S is most commonly used to evaluate the HIC susceptibility of different steel samples in a relatively short time. However, it is recognized that the test method in NACE TM0284 is too severe compared with actual service environments. In fact, the NACE TM0284 document itself states as follows, “The test method is not intended to duplicate service conditions. It is intended to provide reproducible test environments capable of distinguishing the susceptibility of different steel samples to HIC in a relatively short time”.¹⁴⁾

In recent years, Fitness-for-Purpose (FFP) HIC evaluation has been studied as an approach in which appropriate materials are chosen for sour environments of different severities.¹⁵⁻²¹⁾ FFP is generally applied to mildly sour conditions that have lower H_2S and/or higher solution pH than solution A in NACE TM0284, since the severity of that method

is much greater than those of actual use environments. Therefore, establishment of the FFP evaluation method is expected to expand the applicable range of material strength and the flexibility of manufacturing, and thus will result in increased sales of high strength steels and/or cost reduction. H_2S partial pressure and solution pH are significant environmental factors that determine HIC cracking damage. It has been reported that HIC susceptibility strongly depends on H_2S partial pressure.^{17,22,23)} Solution pH is also important from the viewpoints of the corrosion reaction and hydrogen supply to the steel surface. **Figure 1** shows effect of pH on HIC occurrence of two different steels under 0.1 MPa H_2S . The critical pH for HIC was between 5.0 and 5.5 for material A and between 3.0 and 3.5 for material B. Those results imply that line pipe steels have different pH dependence of HIC susceptibility and a small shift of the pH value can lead to significant difference of HIC occurrence. Therefore it is essentially important to maintain solution pH during HIC test. On the other hand, there may be cases where the 96-hour test period specified in NACE TM0284 is not long enough to judge HIC susceptibility under mildly sour conditions. Results of long-term HIC tests suggested different test durations for different ranges of H_2S partial pressure.^{17,18,21)} In those HIC tests under mildly sour conditions, it is necessary to maintain the test conditions for extended periods of time. Therefore, the pH stability of the test solution is necessary for test reliability and reproducibility.

The FFP HIC test method and acceptance criteria are specified in NACE MR0175/ISO15156-2.^{24,25)} This method specifies a 5.0 wt% sodium chloride and 0.4 wt% sodium acetate solution, which is also standardized in EFC16 for the SSC test solution. For SSC testing, NACE TM0177 specifies a 5.0 wt% sodium chloride, 0.41 wt% sodium acetate and 2.5 wt% acetic acid solution as solution B.²⁶⁾ The initial pH of the solution is approximately 3.5, and the solution has a pH buffering effect due to the equilibrium between its acetic acid and sodium acetate constituents. The HLP Corrosion WG investigated an appropriate HIC test solution for FFP focusing on the buffering capacity of the acetic acid solution. In the following section, highlight data of recent research that the HLP Corrosion WG conducted, particularly the influence of concentration of acetate in the solution on pH buffering effect and comparison with conventional HIC solution are shown.

3. Recent Activities of HLP Corrosion WG

3.1. pH Buffering Effect of Highly Concentrated Acetate Solution

In this study, API X65 grade non-sour carbon steel plates for manufacturing welded line pipes were used. The plate was produced by Thermo Mechanical Controlled Process (TMCP) and the microstructure was uniform bainitic ferrite. The pH stability of the test solution is essential for FFP HIC evaluation since pH drift during the HIC test can change the severity of the test environments. The influence of the total amount of acetate on pH stability was investigated by comparing the pH stability among three types of solutions, as shown in **Table 1**. In order to improve the pH stability of the solution, the total amount of acetic acid and sodium acetate was increased to 0.93 N that is double in the solu-

tion B. Note that solution B in NACE TM0177 uses a 0.47 N acetate solution and the conventional FFP HIC test solution in NACE MR0175 is equivalent to a 0.05 N acetate solution. **Figure 2** shows the pH change of those solutions under 0.1 MPa H_2S during a 14-day HIC test. The initial pH was 3.5, and the specific solution volume to the surface area of specimens (hereafter specific solution volume) was 3.0 mL/cm², which was the lower limit specified in NACE TM0284. The solution pH gradually increased with increasing test time. However, the pH increase of the solution was suppressed with increasing concentration of acetate in the solutions. The pH of the 0.05 N acetate solution reached around 4.0 after 14 days, while that of the 0.93 N acetate buffer solution was lower than 3.6 and its shift from the initial value was less than 0.2 even after 14 days. **Figure 3** shows the comparison of the pH shift after 14 days from the initial value between the 0.05 N and 0.93 N acetate

solutions under 0.1 MPa H_2S . The initial pH of the 0.93 N acetate solution was adjusted by changing the ratio of acetic acid and sodium acetate. On the other hand, the initial pH of the 0.05 N solution was adjusted by adding hydrochloric acid (HCl) or sodium hydroxide (NaOH). The pH of both solutions increased during the HIC test when the initial pH was 3.0 and 3.5, whereas the pH decreased when the initial pH was greater than 5.0. The 0.93 N acetate buffer solution had excellent pH stability in the range of pH 3.5 to 5.5, and its shift was within 0.15. On the other hand, the pH shift of the 0.05 N acetate solution was greater than that of the 0.93 N acetate buffer solution and reached approximately 0.5 when the initial pH was 3.5. This large pH shift can affect HIC occurrence of the specimen that has different

Fig. 1. Effect of pH on HIC occurrence of two different steels.

Fig. 2. Influence of concentration of acetate in solution on pH shift during HIC test.

Fig. 3. Comparison of pH stability in a range of initial pH values under 10^{-1} MPa H_2S .

Fig. 4. Comparison of pH stability between 0.05 N and 0.93 N solutions under 10^{-3} MPa H_2S (1% H_2S balanced with N_2).

Table 1. HIC test solutions for FFP evaluation.

Normal of acetic acid and sodium acetate	Composition of test solution	Remark
0.05 N	5%NaCl + 0.4%CH ₃ COONa + HCl	NACE MR0175/ISO15156-2
0.47 N	5%NaCl + 0.4%CH ₃ COONa + 2.5%CH ₃ COOH	solution B/NACE TM0177
0.93 N	5%NaCl + 0.8%CH ₃ COONa + 5.0%CH ₃ COOH	new candidate

pH susceptibility.

The pH stability of the test solutions was also compared under lower H_2S partial pressure. The comparison of pH change between the 0.05 N and 0.93 N acetate solutions under 10^{-3} MPa H_2S (1% H_2S balanced with N_2) is shown in **Fig. 4**. Similarly to the 0.1 MPa H_2S condition, the pH change of the 0.93 N acetate buffer solution during the 14-day HIC test was smaller than that of the 0.05 N acetate solution. From those results, it was concluded that 0.93 N acetate solution is effective for maintaining the solution pH during an extended HIC test. The influence of the balance gas, *i.e.*, N_2 or CO_2 , on the pH stability of the 0.93 N acetate buffer solution was also investigated. Although the pH drift behavior was different, excellent pH stability was observed regardless of the species of balance gas.⁸⁾

In addition, the pH shift of the solution under 10^{-3} MPa H_2S was greater than that under 0.1 MPa H_2S . It is believed that the pH shift of the test solution is caused by a local concentration change of the chemical species near the surface.

Fig. 5. Influence of specific solution volume to surface area on pH shift in 0.05 N solution during HIC test under 10^{-3} MPa H_2S .

Fig. 6. Influence of specific solution volume to surface area on pH shift in 0.93 N solution during HIC test under 10^{-3} MPa H_2S .

The mechanism of the pH shift attributed to the formation of iron sulfide was discussed in the previous literature.⁷⁾

The pH change during the HIC test can also be suppressed by increasing the specific solution volume to surface area of specimens. Therefore, the influence of the specific solution volume on pH shift was also compared between the 0.05 N and 0.93 N acetate buffer solutions. **Figure 5** shows the result of the pH change in the 0.05 N acetate solution under 10^{-3} MPa H_2S under different conditions of specific solution volume. An increase of the specific solution volume tended to suppress pH shift. However, even when the specific solution volume was 30 ml/cm^2 , a large pH change from 3.5 to 4.3 was observed. Thus, it does not seem to be practical to increase the specific solution volume in order to achieve strict control of the solution pH during the HIC test. **Figure 6** shows the influence of the specific solution volume on the pH shift in the 0.93 N acetate buffer solution under 10^{-3} MPa H_2S . In the 0.93 N solution, the influence of increasing specific solution volume on pH stability was small since the solution has intrinsically high pH stability and the pH shift was very small, even when the specific solution volume was at lower limit of 3 ml/cm^2 . For this reason, the 0.93 N acetic acid and sodium acetate buffer solution does not need an increase in the specific solution

Fig. 7. Relationship between corrosion rate and time weighted average pH in 0.05 and 0.93 N acetate solutions under 10^{-1} MPa H_2S .

Fig. 8. Hydrogen permeation test results in three different solutions under 10^{-3} MPa H_2S .

volume in order to maintain the aimed solution pH during longer period than normal test period of HIC test (4 days).

Figure 7 shows the average corrosion rate determined by the weight loss of the specimens as a function of the time-weighted average pH under 0.1 MPa H₂S. The specific solution volume was 3 ml/cm² and test duration was 14 days. The time-weighted average pH was calculated as the sum of products of the average pH value of two measurement points and interval time of measurements divided by total test duration. In other words, the time-weighted average pH (pH_{twa}) can be calculated as follows: $\text{pH}_{\text{twa}} = (\text{pH}_{\text{ai}} \cdot t_i + \text{pH}_{\text{ai+1}} \cdot t_{i+1} + \dots) / (t_i + t_{i+1} + \dots)$. Here, pH_{ai} is average pH of *i* and *i*+1 measurement points, *t_i* is interval time of *i* and *i*+1 measurement points. The first pH measurement was conducted before H₂S bubbling, 2nd was after H₂S bubbling, 3rd and the final one were after 4 and 14 days. Test duration normally does not include the period for 60-minute H₂S introduction, however, pH value before H₂S introduction was taken into account for calculation of pH_{twa} since influence of pH variation on HIC occurrence was investigated in this study. Even though the initial pH was the same, the pH_{twa} of the 0.05 N and 0.93 N acetate solutions was different due to their different pH buffering capacities. However, the corrosion rate increased with decreasing pH_{twa} regardless of the concentration of acetate in the solution, and it seemed that the corrosion rate had a good correlation with pH_{twa}. **Figure 8** shows the time dependence of the hydrogen permeability of the steel measured by an electrochemical hydrogen permeation test in the 0.05 N and 0.93 N acetate solutions (pH 4.5). The specimen of 1 mm thickness was set in Devanathan-Stachurski type cell and the permeation current was measured in 0.1 N NaOH solution.²⁷⁾ Hydrogen permeability is defined as the product of permeation current and specimen's thickness. The permeation behaviors in the three acetate solutions showed no remarkable difference as long as the solution pH values were approximately the same. Since pH 4.5 was the most stable pH range for the acetate buffer solution, the pH of all the solutions was nearly constant during the measurements.⁶⁾ The hydrogen permeability gradually decreased with increasing time since a corrosion product formed and grew on the exposed surface. In general, HIC occurs inside steel, unlike SSC, which occurs from the surface. Therefore, it is considered that the hydrogen supply rate from the surface of the steel is the most critical factor determining the occurrence of HIC. Since corrosion rate seemed to be controlled by pH and hydrogen permeation behavior was similar regardless of acetate concentration as shown in Figs. 7 and 8, it is believed that the 0.93 N acetate solution can provide similar corrosion and hydrogen entry mechanism to the conventional HIC test solutions.

3.2. Proposal of Test Solution for FFP HIC Test for NACE TM0284

It was confirmed that the 0.93 N acetate solution had an excellent pH buffering capacity compared with the conventional HIC test solution. The conventional solution requires pH readjustment by adding an acid or alkaline solution when the solution pH exceeds the acceptable limits. The pH value of the conventional test solution can change even as a result of H₂S gas pre-bubbling before starting the HIC test. Frequent pH readjustment of the test solution is not neces-

sary if the 0.93 N acetate buffer solution is used for the HIC test. The ratio of acetic acid and sodium acetate is adjusted so as to obtain the aimed pH of the solution, and the pH of the 0.93 N acetate buffer solution does not change greatly due to H₂S gas pre-bubbling or during the HIC test in the range of initial pH 3.5 to 5.5.⁶⁻⁸⁾ It is believed that the pH stability of the solution contributes to less additional work during test operations.

Although it is also possible to improve the pH stability of the test solution by increasing the specific solution volume, it reduces the maximum number of specimens that can be placed in the test vessel. For instance, if the thickness of specimen is 25 mm and the specific solution volume is 30 mL/cm², only 3 specimens can be set in a 10 liter vessel. In contrast, due to the excellent pH stability of the 0.93 N acetate buffer solution, it is possible to use a low specific solution volume and, as a result, a large number of specimens can be set in the test vessel when the 0.93 N acetate buffer solution. More than 25 specimens can be set in a 10 liter vessel if the specific solution volume is 3.0 mL/cm². Thus, the highly concentrated 0.93 N acetate buffer solution is also advantageous from the viewpoint of test efficiency.

On the basis of those advantages, the HLP Corrosion WG has proposed the 0.93 N acetate buffer solution as the optional test solution for the FFP HIC test specified in NACE TM0284. Revision of the NACE TM0284 has been under discussion in the TG082 Subcommittee in NACE STG32. In order to gain wide recognition of the applicability of the proposed test solution, further investigations to clarify corrosion mechanism and hydrogen entry behaviors are necessary from the view points of electrochemistry and characterization of corroded surface.

4. Conclusions

The recent activities in the HLP Corrosion WG and the present status of HIC evaluation methods were reviewed. The pH buffering capacity of a highly concentrated (0.93 N) acetate buffer solution during the HIC test was superior to that of the conventional HIC test solution within the range of the test conditions. The pH stability of the newly-proposed solution provides reliability and reproducibility of the HIC test. The proposed solution has a big advantage of less frequent readjustment of the solution pH during the test. On the basis of those advantages, the 0.93 N acetate solution has been proposed as the optional HIC test solution in NACE TM0284. In order to gain wide recognition that the solution is applicable in place of conventional solutions, further investigation of the corrosion mechanism and hydrogen entry behavior and comparison with those of the conventional solutions will be necessary in the future.

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Figures and Table in the following Refs. 6) to 8) are permitted.

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