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Standard
Test Method

Laboratory Test Procedures for Evaluation of SOHIC Resistance of Plate Steels Used in Wet H_2S Service

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Foreword

Carbon steel plates employed in welded pressure vessels may be susceptible to one or more forms of environmental cracking when exposed to wet H_2S service conditions. These include (1) sulfide stress cracking (SSC) of hard welds and heat-affected zones (HAZ); (2) hydrogen-induced cracking (HIC) in the base metal; and (3) stress-oriented hydrogen-induced cracking (SOHIC), for example, in the region adjacent to weldments of nominally acceptable hardness. (Definitions of these forms of cracking are presented in Paragraph 1.3.) Extensive work has been conducted over many years to understand various fundamental and applied aspects of these phenomena. Due to recent experiences in refinery wet H_2S operations, attention has been particularly directed to understanding SOHIC and the various metallurgical and environmental parameters that govern its occurrence.

Recent technical publications[1-7] and NACE technical committee reports[8, 9] and standards[10] have focused on several issues related to the serviceability of carbon steel equipment in wet H_2S service. One of these issues is the evaluation of steels to determine their resistance to SOHIC. Other test methods have been standardized for evaluation of SSC and HIC; NACE Standards TM0177[11] and TM0284[12] are extensively utilized in the evaluation and qualification of steels for determination of resistance to SSC and HIC, respectively. However, neither of these test methods deals directly with the specific mechanism and mode of cracking inherent to SOHIC.

The following situation illustrates the shortcomings of the existing test methods in the evaluation of SOHIC. Cracking observed in steel equipment resulting from SOHIC appears to be mechanistically related to HIC because it involves the formation of small blisters in the steel (from the recombination of atomic hydrogen to molecular hydrogen) and the development of interconnecting cracks that link adjacent blisters on different planes in the steel. The small blisters characteristic of HIC typically form parallel to the plate surface (see Figure 1). However, in SOHIC, the orientation of these blisters is much different. The presence of an applied or residual tensile stress in the steel (typically adjacent to the weld HAZ) produces stacked arrays of these blisters and the interconnecting cracking is oriented in the through-thickness direction (see Figure 2). NACE Standard TM0284, while being an acceptable test method for evaluation of HIC, lacks the application of tensile stress and therefore cannot provide an evaluation of this through-thickness cracking process involved in SOHIC.

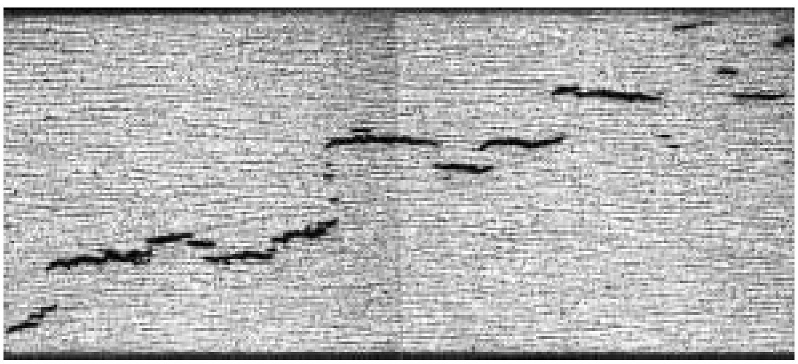

FIGURE 1—HIC in Carbon Steel Under No Applied Stress

The test method defined by NACE Standard TM0177 utilizes stressed specimens and has been effective in evaluating SSC. It is particularly useful for defining threshold stresses below which rupture by SSC does not occur for specific steels and conditions of steels. The TM0177 test method was originally developed and is extensively utilized to evaluate SSC susceptibility in high-strength, low-alloy steels and other materials, which usually occurs without extensive internal cracking. In some cases, blistering is observed. The TM0177 test method has been shown to produce extensive through-thickness arrays of blisters with interconnecting cracks (i.e., SOHIC) in some steels. But, in other cases, test specimens with extensive internal cracking do not rupture. Therefore, differentiation in susceptibility of materials to SOHIC using TM0177 can be somewhat ambiguous.

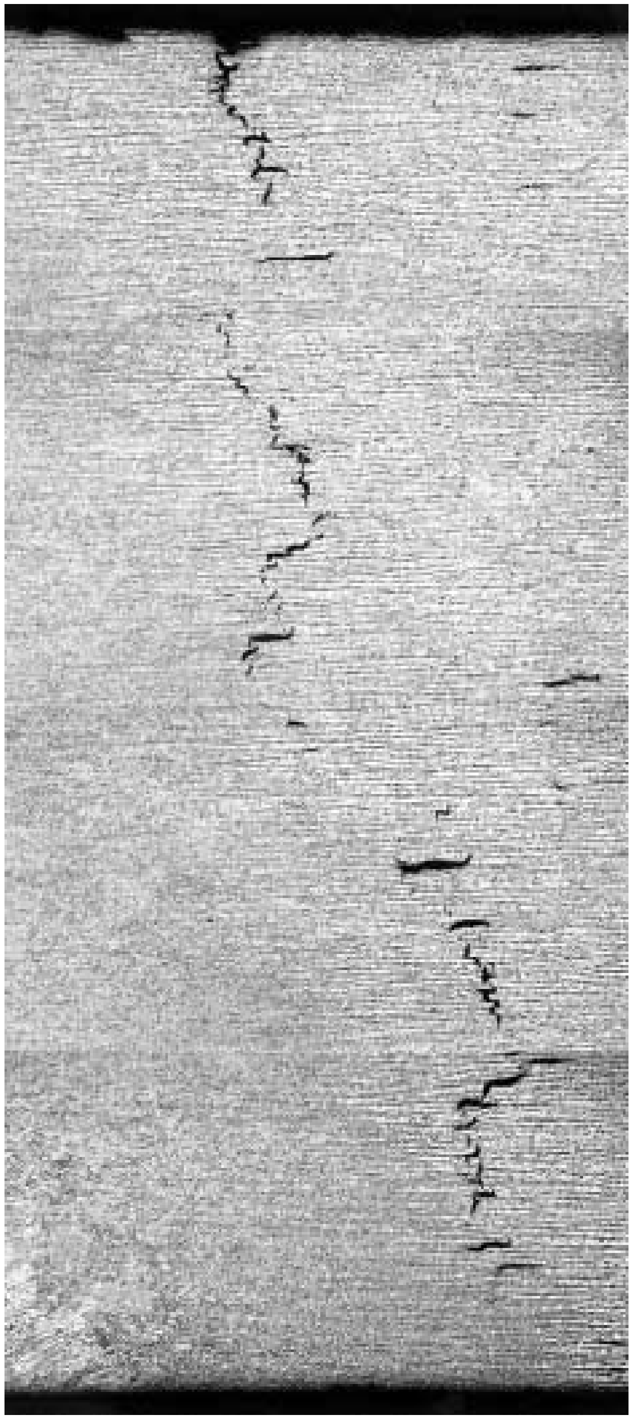

FIGURE 2—SOHIC in Carbon Steel Under Applied Stress. Direction of tensile stress is normal to cracks linking the blisters.

In this SOHIC test method, an attempt has been made to utilize the results of research studies on SOHIC that have been specifically directed toward (1) the evaluation of various H_2S cracking test methods including TM0284, TM0177, and various modifications of these methods; (2) the development of enhanced procedures and techniques for evaluation of the resistance of steels to SOHIC; and (3) investigation of the role of metallurgical and fabrication-related variables on SOHIC resistance of steels with varying processing histories. These studies have identified cases in which a SOHIC test method would be beneficial and in some instances necessary.

Based on the results of the above-mentioned studies, susceptibility to HIC has been generally shown to be greatest in high-sulfur steels and decrease with decreasing sulfur content of the steel. By comparison, some of these studies[2-7, 13] also show that, in severe hydrogen-charging environments (e.g., those that have hydrogen-charging capabilities equivalent to TM0177 solution at pH 3), SOHIC susceptibility can increase as the sulfur content in the steel decreases from the level commonly found in conventional plate steels until very low levels of sulfur are reached, when susceptibility to SOHIC can decrease. However, at very low sulfur contents, several other variables in the steel may play a role in determining SOHIC susceptibility as well. These include the presence of ferrite-pearlite banding, carbide phase hardness, calcium treatment, and inclusion morphology, among others. This complex situation makes prediction of SOHIC susceptibility difficult and is a major reason for conducting SOHIC evaluations of steels. Therefore, it appears that SOHIC testing may be most applicable to newer steels (used in repair and new construction) that tend to have lower sulfur levels by virtue of modern steel-making practices. Furthermore, it has been found that these lower-sulfur steels can exhibit substantial susceptibility to SOHIC under tensile loading even when little or no HIC has been observed in the TM0284 test conducted without externally applied stress. Therefore, SOHIC testing may be needed for steels that have been developed for HIC resistance but that may still be susceptible to SOHIC by virtue of their metallurgical processing and resulting microstructures.

It has also been shown that welding may influence susceptibility to SOHIC in the steel. There may be specific situations in which the SOHIC susceptibility of the transformed region of the base metal adjacent to the weld HAZ is increased. Therefore, specific welding procedures and postweld heat treatments may need to be tested to evaluate their influence on SOHIC resistance. Research studies have shown that this may be the case in steels over a wide range of sulfur contents (i.e., 0.002 wt% sulfur and higher).[4]

This standard presents a test method that can be used by those who produce, use, or specify carbon steels as a basis for laboratory evaluation of SOHIC in these materials. It provides for evaluation of SOHIC based on a crack-arrest methodology. In this procedure, conditions that promote the initiation of SOHIC have been instituted and the response of the steel in terms of crack arrest is monitored. The basic procedures given in the body of this standard have been streamlined specifically to provide a relatively simple testing procedure with respect to the SOHIC resistance of steels. However, there are many other applications of these procedures that may involve nonroutine testing and materials evaluation for research and development applications. Information relevant to the testing of welds, supplemental crack analyses, and testing in alkaline sour water solutions with cyanide has been separated from the more routine procedures and is presented in Appendices A and B.

This standard was prepared by NACE Task Group (TG) 175 on Stress-Oriented Hydrogen-Induced Cracking (SOHIC). This TG is administered by Specific Technology Group (STG) 62 on Science and Engineering Applications and Methods of Corrosion Monitoring and Measurement. It is also sponsored by STG 32 on Oil and Gas Production—Metallurgy, and STG 34 on Petroleum Refining and Gas Processing. This standard is issued by NACE under the auspices of STG 62.

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Contents

Section 1: General

1.1 This standard covers the testing of carbon steels for the evaluation of their susceptibility to cracking in aqueous hydrogen sulfide (H_2S) environments by the phenomenon known as SOHIC. Tests for cracking in these environments are commonly conducted at room temperature where cracking susceptibility is typically high. However, other situations that can produce conditions that are more or less severe from the standpoint of corrosion, hydrogen permeation, and cracking severity than found in room-temperature tests may be present.

1.2 This standard specifies the reagents, test specimens, testing apparatus, base material and test specimen properties, test solutions, test procedures, and test specimen evaluation procedures for a NACE standard double-beam (DB) test for SOHIC. Sections 1 through 9 cover the DB test and focus on the evaluation of resistance of the material to through-thickness cracking in the presence of a tensile stress and a stress concentrator used to initiate cracking. Section 10 gives an alternative SOHIC test procedure using the existing NACE Standard TM0177 Method A (Tensile Test). This section provides additional requirements on the TM0177 Method A procedures for evaluation of SOHIC when time-to-failure data or a threshold stress approach is needed. Other recommended and optional procedures and related information that may be useful in the testing of carbon steels and weldments for SOHIC are given in Appendices A through D.

The DB test provides for the evaluation of through-thickness internal cracking of carbon steels under conditions of applied tensile stress that may result in SOHIC in plate steels. It utilizes a four-point bent DB specimen. It has been observed that an increased amount of through-thickness internal cracking (due to the presence of SOHIC) can occur when a tensile stress is applied to a test specimen. More specifically, stressed test specimens can exhibit cracking even when unstressed test specimens exposed to the same environment show no cracking.

The test procedure can be summarized as follows: Stressed DB specimens are immersed in an aqueous test solution saturated with H_2S at ambient pressure and temperature. The test solution can be selected to produce conditions of moderate to high levels of hydrogen charging in the steel, which may produce moderate to high severity of cracking in susceptible steels. The standard test solutions are acidic. However, an optional alkaline cyanide-containing test solution that has been shown to produce significant hydrogen charging of steels and internal cracking in susceptible steels is described in Appendix A. It is generally not necessary to simulate the actual conditions of service environments, but rather to choose a test solution that results in a severity of hydrogen charging that is appropriate for the evaluation under consideration.

Procedures for making measurements of the extent of through-wall cracking under a slot in the tension surface in the DB test specimen are given herein. Optional methods that can be used to further characterize and analyze the location and extent of internal cracking that may be related to HIC and/or SOHIC are also provided. These test methods can be extended to elevated temperatures and pressures; however, that is beyond the scope of this standard.

1.3 The following terms, definitions, and descriptions assist in differentiating between the various forms of internal cracking that can occur in steels under hydrogen charging:

1.3.1 **Sulfide Stress Cracking (SSC)**—Cracking of a metal under the combined action of tensile stress and corrosion in the presence of water and H_2S (a form of hydrogen stress cracking [HSC]).

Corrosion of steel by H_2S liberates atomic hydrogen at the metal surface. The H_2S also poisons the recombination of the atomic hydrogen into molecular hydrogen, thus promoting the absorption of atomic hydrogen by the steel. The atomic hydrogen then diffuses through the steel and tends to accumulate at areas of high metal hardness and high tensile stress (either applied or residual) and embrittles the steel. Therefore, the SSC mechanism involves hydrogen embrittlement. The cracking mode is primarily transgranular in low-strength steels, but can be mixed-mode or even intergranular in localized hard regions and in higher-strength (i.e., martensitic or bainitic) steels.

1.3.2 **Hydrogen-Induced Cracking (HIC)**—Stepwise internal cracks that connect adjacent hydrogen blisters on different planes in the metal, or to the metal surface (also known as *stepwise cracking*).

No externally applied stress is needed for the formation of HIC. The driving force for crack propagation is high stresses at the circumference of the blisters caused by buildup of internal pressure in the blisters. Interaction between these high-stress fields tends to cause cracks that link blisters on different planes to develop. The linkup of blisters on different planes in steels has been referred to as *stepwise cracking* to characterize the nature of the crack appearance.

1.3.3 **Stress-Oriented Hydrogen-Induced Cracking (SOHIC)**—A stacked array of small blisters joined by hydrogen-induced cracking that is aligned in the through-thickness direction of the steel as a result of high localized tensile stresses.

SOHIC is a special form of HIC that usually occurs in the base metal, adjacent to the HAZ of a weld, where there are high residual stresses from welding. It may also occur at other high-stress points such as the tip of