

Research Report on Characterization and Monitoring of Cracking in Wet H₂S Service

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Washington, D.C. 20005



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Research Report on Characterization and Monitoring of Cracking in Wet H₂S Service

FOREWORD

In 1990 cracking of refinery process equipment in wet H₂S service was being widely reported. As a result, committees and task groups of industry organizations, including API, NACE and the Materials Properties Council, were actively seeking improved understanding of the phenomenon. Of particular concern were conditions leading to cracking and blistering, the incidence of cracking, the consequences of such damage, and the efficacy of NDE methods for detection and monitoring.

The Program reported herein was one of several significant industry-wide efforts on this subject. It was intended to supplement other activities by examining, in a large-scale test vessel, issues which could not be addressed satisfactorily either with conventional small-scale laboratory specimens or with in-situ exposures in refineries.

The construction of a welded steel pressure vessel with a replaceable "window" for test purposes was proposed by MPC to the API Subcommittee on Corrosion and Materials. Its Task Group on Materials and Corrosion Research developed the program to study the effect of variables on cracking and the capabilities of certain NDE monitoring methods. At that time, it was realized that the results would not be directly translatable to field application because the environment to be used would be extremely severe, and the thickness of the steel studied was limited to 0.5 inch. Nevertheless the guidance to be obtained would be valuable.

The results of the program have helped to provide validation of observations of conventional laboratory test specimens and clarify the roles of the variables considered. The work on acoustic emission monitoring was particularly enlightening. However, it must be realized that the information obtained is to be viewed in the context of specific service demands before application in the plant should be attempted. For example, pressure vessels usually have thicker shells than used in the test which will affect NDE capabilities, hydrogen permeation and material behavior.

A large measure of the credit for the success of this program goes to the members of the API Subcommittee and Task Group and the MPC Sponsor Committees who contributed their ideas and vast experience.

M. Prager



Research Report on Characterization and Monitoring of Cracking in Wet H₂S Service

M. S. Cayard, R. D. Kane, L. Kaley, and M. Prager

CONTENTS

1.0 Executive Summary	1	3.3.3 Evaluation of the Repair of a HIC Damaged Vessel	10
2.0 Introduction	2	3.3.4 Simulation of the Cracking Behavior of Thick Plate	10
2.1 Background	2	3.3.5 Evaluation of HIC Resistant Plate/I.D. Surface Cleaning/Severe Hydrogen Charging Conditions	10
2.2 Goal	3	3.3.6 Evaluation of Nozzle Attachments/Effect of PWHT	11
2.3 Technical Approach	3	3.3.7 Verification of AE Signature for Hydrogen Blistering and SSC	11
2.4 Terminology	3	4.0 Results and Discussion	12
2.4.1 Wet H₂S Cracking Mechanisms	3	4.1 Materials Selection	12
2.4.1.1 Hydrogen Blistering	3	4.2 Fabrication	13
2.4.1.2 Hydrogen Induced Cracking (HIC)	3	4.3 Inspection	17
2.4.1.3 Stress Oriented Hydrogen Induced Cracking (SOHIC)	4	4.4 Vessel Design and Integrity	28
2.4.1.4 Sulfide Stress Cracking (SSC)	4	5.0 References	31
2.4.2 Steels	4	Appendix I—Serviceability of HIC Damaged Steel	33
2.4.2.1 Conventional Steel	4	Appendix II—Serviceability of Hard Welds	45
2.4.2.2 Low Sulfur Conventional Steel	4	Appendix III—Evaluation of Weld Repair/PWHT	57
2.4.2.3 “HIC Resistant” Steel	4	Appendix IV—Simulation of Thick Plate Behavior	75
2.4.2.4 Ultra-Low Sulfur Advanced Steels	4	Appendix V—Environmental Staging/Effect of Cleaning	93
2.4.3 General Terminology	4	Appendix VI—Serviceability of Nozzle Attachments and PWHT	121
2.4.3.1 Crack Length Ratio (CLR)	4		
2.4.3.2 Crack Thickness Ratio (CTR)	5	1.0 Executive Summary	
2.4.3.3 Crack Sensitivity Ratio (CSR)	5	This report presents the experimental methods and findings of a research program of MPC entitled “Characterization and Monitoring of Cracking in Wet H ₂ S Service.” The program was supported by the Refining Division of the American Petroleum Institute (API) and by The Materials Properties Council (MPC) and its Fitness-for-Service group-sponsored program.	
2.4.3.4 Longitudinal-Transverse (LT) Section	5	The two main objectives of the program were to study the performance in wet H ₂ S of welded steel plate of various qualities and microstructures, and evaluate the effectiveness of NDE techniques to characterize and monitor the cracking. To accomplish these objectives, a series of large scale exposure tests were conducted with steel panels (referred to herein as “windows”) containing welds and attachments welded into a fabricated steel vessel filled with a	
2.4.3.5 Transverse-Longitudinal (TL) Section	5		
3.0 Experimental Procedures	5		
3.1 Materials Evaluated	5		
3.2 Specimen Configurations	5		
3.3 Experimental Overview	7		
3.3.1 Evaluation of Plate Containing Pre-existing HIC Damage	9		
3.3.2 Evaluation of Plate Containing Hard Welds (HRC 22–30)	9		

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pressurized H₂S containing solution prepared in accordance with NACE Standard TM0177-90, Method A. Experiments were performed using windows comprised of conventional, low sulfur, ultra-low sulfur and advanced thermo-mechanically controlled processed (TMCP) steels per the ASTM A516-70 and A841 specifications using various weld fabrication methods.

Characterization and monitoring of internal cracks resulting from Hydrogen Induced Cracking (HIC), Stress Oriented Hydrogen Induced Cracking (SOHIC) and Sulfide Stress Cracking (SSC) was accomplished at various pressures, solution pH, H₂S content, and time. Methods utilized included: (1) manual and automated ultrasonic testing (UT), (2) wet fluorescent magnetic particle testing (WFMPT), and (3) acoustic emission (AE). The results of these nondestructive techniques were confirmed using metallographic sectioning following exposure.

It was found that resistance to blister-type HIC was greater in lower sulfur steels than in the higher sulfur conventional steels. Maximum resistance to HIC was found in ultra-low sulfur advanced steels produced by thermo-mechanically controlled processing (TMCP) with ultra-low sulfur contents (≤ 0.001 wt. percent) and processed to produce controlled homogeneous microstructures free from ferrite/pearlite banding.

Based on all materials evaluated in this program, the resistance to through-wall crack propagation also increased with decreasing sulfur content and decreasing microstructural banding. However, under very severe hydrogen charging conditions, i.e., two to three times the NACE TM0177 solution charging levels, all steels evaluated exhibited through-wall cracking to depths ranging from approximately 30–50% of the plate thickness irrespective of either the sulfur content or degree of banding. These data suggest that there is a threshold level of hydrogen charging above which the resistance of these materials, even those processed to optimize resistance to HIC, breaks down. At such severe hydrogen charging conditions, the use of stainless steel clad vessels may be more appropriate.

One of the most notable findings of this program was the significant impact of surface cleaning and removal of protective sulfide films on the subsequent cracking behavior of carbon steel equipment. It was found that removal of surface films on the internal surface of carbon steel equipment, using techniques typically used prior to WFMPT, increased the hydrogen flux and likelihood of wet H₂S cracking during operation prior to the reformation of a protective sulfide scale.

Nondestructive evaluation techniques (i.e., WFMPT, UT and AE) were useful to varying degrees in identifying and monitoring HIC, SOHIC and SSC. Automated ultrasonic testing (AUT) T-scan adequately identified in-plane, blister type cracking caused by HIC in a semi-quantitative manner which

correlated reasonably well with metallographic sectioning. AUT P-scan was only able to qualitatively identify through-wall SOHIC. This may have, in part, been due to the relatively thin section size of the plate used in this study.

Manual UT was required to size through-wall flaws in plate specimens resulting from SOHIC and sulfide stress cracking (SSC). However, manual UT crack sizing measurements in the regions around nozzle attachment welds and surface crack indications obtained using WFMPT typically did not accurately identify areas of significant through-wall cracking from SOHIC as confirmed by metallographic sectioning. Many of the indications obtained from WFMPT were limited to surface cracks and imperfections.

AE monitoring was able to detect and characterize damage during periods of active cracking resulting from HIC, SOHIC and SSC. AE also differentiated cracking related to in-plane (blister-type) HIC growth from through-wall crack growth resulting from SOHIC and SSC. AE conducted during hydrotesting of HIC/SOHIC damaged material required relatively high levels of internal pressure, beyond the normal operating levels for the vessel, to produce a significant AE response.

In general, the observations of material behavior found in this study were consistent with the findings of the previously conducted laboratory testing conducted in the MPC group-sponsored Wet H₂S Research Program. The results of the present study validated the wet H₂S test methods developed previously in that work and showed their applicability to refinery wet H₂S service conditions.

2.0 Introduction

Presented herein is the final report for a research program conducted at CLI *International* as contractor to The Materials Properties Council, Inc. (MPC). The program was jointly funded by MPC, its Fitness-for-Service sponsor group and the Refining Division of the American Petroleum Institute (API). DNV Industry Inc. provided nondestructive services for WFMPT, UT and AE inspection as well as nondestructive test data analysis. Chicago Bridge and Iron fabricated the wet H₂S full-scale test vessel. This report contains a comprehensive summary of the test facilities and experimental methods, pertinent findings and analysis of the test results.

2.1 Background

Refinery equipment in wet H₂S service is characterized by exposure to aqueous process environments containing hydrogen sulfide. Systematic inspection programs conducted by petroleum companies have shown that wet H₂S refinery processes can provide conditions for hydrogen charging of steel and widespread cracking of carbon steel equipment.^{1,2} The results of operating experience surveys and technical investigations have described situations where carbon steel equipment exposed to wet H₂S environ-

ments may be susceptible to cracking via hydrogen induced cracking (HIC), stress oriented hydrogen induced cracking (SOHIC) and/or sulfide stress cracking (SSC).³⁻⁶ In some cases, cracking has been found to be minimal, resulting in no significant effect on equipment integrity or serviceability. In other cases, widespread cracking initiates and/or cracks propagate to a substantial degree thus limiting the residual load and pressure capabilities of the affected equipment.

Prior to the initiation of this program, MPC organized a research program on wet H₂S cracking of steels sponsored by more than twenty major petroleum companies, steel manufacturers and equipment fabricators. This program was aimed at (1) the development of screening procedures for evaluation of steels, (2) the determination of the influence of metallurgical processing and welding variables, and (3) the better understanding of the roles of stress, environment composition and temperature. It has provided valuable fundamental information which has improved both the awareness of the causes of wet H₂S cracking and potential solutions in terms of both new construction and repair and remediation of existing equipment. However, there was a desire to validate the findings and conclusions of that program and to explore the complex interrelations of variables that can affect the actual behavior of large scale equipment used in wet H₂S service.

The present study was conducted to provide important information regarding the serviceability of welded steel equipment in wet H₂S service. Specifically, situations exist in refinery operations where it is necessary to assess equipment based on the fitness for continued service versus repair or replacement because of wet H₂S damage. Such an assessment requires information regarding (1) the nature of wet H₂S crack propagation, (2) the operational conditions that may affect wet H₂S damage, and (3) the ability to use nondestructive methods to assess the degree of cracking in operating equipment.

2.2 Goal

The overall goal of this program was to demonstrate the ability to characterize and monitor various aspects of crack propagation in pressurized process equipment exposed to wet H₂S environments. Specific aspects of wet H₂S cracking and crack monitoring were closely examined, namely:

1. Identification of the mechanical/environmental effects such as the role of internal pressure, pressure cycling and environmental severity.
2. Identification of active cracking sites in pressurized equipment using nondestructive evaluation (NDE) methods.
3. Assessment of the relative abilities of various NDE methods such as acoustic emission (AE), ultrasonics (UT), automated ultrasonics (AUT) and wet florescent magnetic particle techniques

(WFMP) versus destructive examination methods such as metallography.

4. Evaluation of fabrication and repair techniques with regard to their ability to reduce or prevent wet H₂S cracking, and to identify any procedures which may increase the susceptibility to cracking.

2.3 Technical Approach

To accomplish the goal and objectives of this program, a series of large scale exposure tests were conducted with a fabricated steel vessel [36 in. (90 cm) nominal outer diameter; 6 ft (1.8 m) long] made to ASME design requirements. The tests conducted during this program utilized a novel approach involving test specimens or "windows" fabricated from steel plates, welds, fittings and attachments using typical practices utilized in the construction and maintenance of refinery equipment. These windows were welded into the test vessel which contained pressurized wet H₂S test media.

2.4 Terminology

2.4.1 Wet H₂S Cracking Mechanisms. Wet H₂S cracking is a complex and often misunderstood phenomenon involving several fundamental cracking mechanisms. The complexities involved in developing a global understanding of wet H₂S cracking revolve around the fact that each cracking mechanism has different controlling metallurgical and environmental parameters as well as specific modes of attack. To properly present and discuss the results of this program, it is first necessary to clearly set forth the basic terminology related to the various mechanisms of wet H₂S cracking.

Wet H₂S cracking involves four types of mechanisms:

1. Hydrogen Blistering
2. Hydrogen Induced Cracking (HIC)
3. Stress Oriented Hydrogen Induced Cracking (SOHIC)
4. Sulfide Stress Cracking (SSC)

A brief discussion of each of these cracking mechanisms is presented below.

2.4.1.1 Hydrogen Blistering. Hydrogen blistering is the development of internal blisters in a steel caused by the accumulation of molecular hydrogen. The blisters usually occur at sites of large non-metallic inclusions, laminations or other large metallurgical discontinuities in the steel. The blisters are oriented parallel to the surfaces of the steel. The molecular hydrogen which acts to initiate and propagate these blisters arises from the absorption and diffusion of atomic hydrogen produced on the steel surface by the sulfide corrosion process. No externally applied stress is required to produce hydrogen blistering.

2.4.1.2 Hydrogen Induced Cracking (HIC). HIC is a form of internal hydrogen damage caused by the development of small cracks oriented parallel to the surfaces of the steel. These cracks tend to link up with

other cracks due to a build-up of internal pressure in the hydrogen damage zones in the steel and the resultant stress fields around the zones. This link-up of the cracks tends to produce the characteristic stepwise crack appearance. Similar to hydrogen blistering, no externally applied stress is required for the formation of HIC.

The link-up of the small blister cracks on different planes in the steel is often referred to as "stepwise cracking" to describe the characteristics of the crack appearance. The stepwise linkage of these cracks can have a major or minor effect on reducing the load (pressure) capabilities of the equipment depending on the nature of the linkage. HIC is commonly found in steels with moderate to high impurity levels which have a high density of elongated sulfide inclusions often found in fully (Al-Si) killed steels.

2.4.1.3 Stress Oriented Hydrogen Induced Cracking (SOHIC). SOHIC is the development of arrays of short cracks which are linked in the through-thickness direction. These arrays of cracks are typically aligned perpendicular to the tensile stress which can be produced by both applied mechanical and residual tensile stresses. SOHIC is commonly observed to occur in the heat affected zone (HAZ) microstructures in the base metal associated with fabrication and attachment welds. They may also be produced at high stress concentration points such as crack-like flaws, the tip of cracks produced by SSC in hard HAZ's or where HIC intersects the weld HAZ area.

2.4.1.4 Sulfide Stress Cracking (SSC). SSC is brittle cracking produced by a form of hydrogen embrittlement cracking under the combined action of tensile stress and aqueous corrosion in the presence of hydrogen sulfide. SSC usually occurs in high strength steels or in high hardness regions of welds and HAZ's. SSC involves the interaction of the absorbed atomic hydrogen produced by the sulfide corrosion process with internal sites in the metal lattice. Such sites can be grain boundaries and inclusions. However, SSC is usually differentiated from HIC because it does not require the recombination of atomic hydrogen to form molecular hydrogen and the build-up of pressure at sites inside of the steel.

2.4.2 Steels. The present investigation involves the evaluation and testing of several types of steels which can be differentiated by the type of metallurgical processing which they receive during manufacturing. The following steels were tested:

1. Conventional Steel
2. Low Sulfur Conventional Steel
3. "HIC Resistant" Steel
4. Ultra-Low Sulfur Advanced Steel

The basic attributes of each of these steels is described below:

2.4.2.1 Conventional Steel. A conventional steel is a commercially produced steel which is either hot rolled or normalized (e.g., ASTM A516-70). It has generally moderate to high levels of impurities, par-

ticularly sulfur (i.e., ≥ 0.010 wt. percent sulfur). This type of material generally has a high susceptibility to HIC in most hydrogen charging environments even under moderate exposure conditions.

2.4.2.2 Low Sulfur Conventional Steel. A low sulfur conventional steel is a commercially produced material which contains lower than normal levels of sulfur (i.e., 0.003–0.010 wt. percent). This material can exhibit improved mechanical properties over conventional steels, but typically has not been processed to specifically exhibit high resistance to HIC. These steels can still show significantly high susceptibility to HIC even in moderate service environments.

2.4.2.3 "HIC Resistant" Steel. The term "HIC resistant" steel is used by manufacturers and users to denote conventional grades of steel (e.g., ASTM A516-70) which have been metallurgically processed to enhance their resistance to HIC. Such processing typically includes ultra-low sulfur levels (i.e., ≤ 0.002 wt. percent sulfur), normalizing heat treatments to modify the hot rolled microstructure and possibly Ca additions to produce sulfide shape control. Shape control is important in that it produces sulfides of spherical morphology which reduce localized stresses in the vicinity of the inclusion, compared to the elongated stringers found in conventional steels. These steels are often tested to evaluate HIC resistance using conventional or modified NACE TM0284 methods for the purposes of lot acceptance or for supplemental information. These steels typically have improved resistance to HIC as compared to conventional steels; however, they may still show some degree of susceptibility to HIC and SOHIC in severe wet H_2S service conditions.

2.4.2.4 Ultra-Low Sulfur Advanced Steels. Ultra-low sulfur advanced steels are those made by modern steelmaking and processing techniques. These steels typically have ultra-low levels of sulfur (e.g., ≤ 0.002 wt. percent sulfur) and low carbon equivalents compared to conventional steels of comparable tensile strengths (i.e., ASTM A516-70). Steels in this category are currently made to ASTM A841 by thermo-mechanically controlled processing (TMCP) and/or accelerated cooling techniques. Also, they have reduced carbon levels as compared to conventional steels to produce ferritic or ferritic/bainitic microstructures with little or no microstructural banding.

2.4.3 General Terminology. The following terms are used throughout the context of this report and are defined here for clarity.

2.4.3.1 Crack Length Ratio (CLR). The crack length ratio or CLR provides a measure of the materials resistance to HIC as defined in NACE Standard TM0284-87. CLR is determined by summing the lengths of each crack array and dividing by the section width and multiplying by 100 to express it as a percentage. This is shown schematically in Fig. 2-1.

2.4.3.2 Crack Thickness Ratio (CTR). The crack thickness ratio or CTR also provides a measure of the materials resistance to HIC as defined in NACE Standard TM0284-87. CTR is determined by summing the thicknesses of each crack array and dividing by the section thickness and multiplying by 100 to express it as a percentage. This is shown schematically in Fig. 2-1.

2.4.3.3 Crack Sensitivity Ratio (CSR). The crack sensitivity ratio or CSR also provides a measure of the materials resistance to HIC as defined in NACE Standard TM0284-87. CSR is determined by summing the products of the length and thicknesses of each crack array and dividing this sum by the product of the section length and thickness and multiplying this value by 100 to express it as a percentage. This is shown schematically in Fig. 2-1.

2.4.3.4 Longitudinal-Transverse (LT) Section. A longitudinal-transverse or LT section is a metallographic section in which the perpendicular to the polished face is parallel to the longitudinal or rolling direction.

2.4.3.5 Transverse-Longitudinal (TL) Section. A transverse-longitudinal or TL section is a metallographic section in which the perpendicular to the polished face is perpendicular to the longitudinal or rolling direction.

3.0 Experimental Procedures

The materials evaluated in this program along with specimen configurations, general conditions of expo-

sure, and post-exposure evaluations conducted are summarized below. Specific conditions of exposure time, pressure, and environmental severity of each test are presented in the respective appendix (Appendices I-VI) for each particular vessel exposure.

3.1 Materials Evaluated

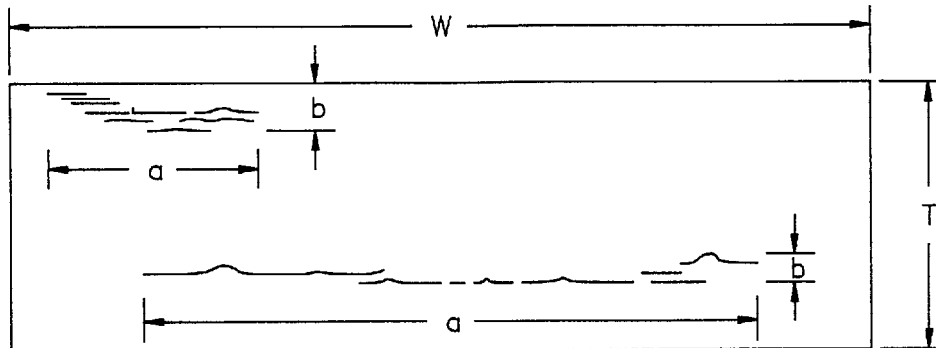
The present investigation involved the testing and evaluation of the following steels:

1. Conventional Steel
2. Low Sulfur Conventional Steel
3. "HIC Resistant" Steel
4. Ultra-Low Sulfur Advanced Steel
5. ASTM A53 ERW Pipe
6. ASTM A234 WPB Weld Cap
7. ASTM A105 Threadolet

The basic attributes of each of the first four steels listed were previously described in Section 2.3.2. The evaluation of the ASTM A53 ERW pipe, ASTM A234 WPB weld cap and ASTM A105 threadolet materials was limited to the final test to study the effect of nozzle attachments. The material compositions and mechanical property data for each of the base plate materials are presented in Table 3-1.

3.2 Specimen Configurations

As previously mentioned, the goals and objectives of this program were accomplished using a series of large scale tests on a 36 in. (90 cm) nominal diameter pressure vessel approximately 6 ft (1.8 m) long. The



$$\text{Crack Sensitivity Ratio, CSR} = \frac{\sum (a \times b)}{W \times T} \times 100$$

$$\text{Crack Length Ratio, CLR} = \frac{\sum a}{W} \times 100$$

$$\text{Crack Thickness Ratio, CTR} = \frac{\sum b}{T} \times 100$$

Method Of Measuring Stepwise Cracks

Fig. 2-1—HIC damage evaluation formulas given in NACE TM0284-87

Table 3-1—Material Summary

Steel No. CLI# Grade	A 2278 A516-70	B 3201 A516-70	C 2279 A516-70	D 2098 A516-70	E 3249 A841	F 3250 A841	G 2280 A516-70
C.E.	0.410	0.427	0.460	0.400	0.332	0.329	0.420
C	0.220	0.210	0.220	0.210	0.120	0.090	0.220
S	0.017	0.001	0.019	0.007	0.001	0.001	0.020
P	0.020	0.005	0.014	0.013	0.005	0.003	0.027
Mn	1.090	1.090	0.950	1.080	1.110	1.180	1.110
Cu	...	0.060	...	...	0.190	0.010	...
Ni	...	0.050	...	...	0.190	0.230	...
Cr	...	0.120	...	...	0.010	0.020	...
Mo	...	0.020	...	...	0.000	0.070	...
Ti	...	0.003	...	...	...	...	...
V	...	0.001	...	...	0.000	0.040	...
Nb	...	0.002	...	...	0.026	0.020	...
Si	0.260	0.260	0.190	0.210	0.270	0.250	0.270
Al	0.035	0.020	0.010	0.039	0.037	0.029	0.031
Ca	...	...	...	...	...	...	...
Y.S.	48.2	49.6	57.3	47.6	61.4	67.0	52.0
U.T.S.	78.9	76.2	78.4	78.8	74.2	79.0	76.9
% Elong	25.0	27.0	23.0	45.0	26.0	25.0	42.0

vessel, shown in Fig. 3-1, was fabricated by Chicago Bridge and Iron (CBI Na-Con, Inc.) at its Houston location.

Each large scale test incorporated the use of a “window” specimen measuring approximately 2 ft by 2 ft (0.6 × 0.6 m). This approach is detailed schematically in Fig. 3-2. With the exception of the test window, the entire I.D. surface of the vessel was coated to protect the remaining vessel from damage. The coating chosen for this program was T31, ECTFE material. The T31 process is comprised of a primer

and multiple topcoats of a partially fluorinated copolymer. The T31 coating is a true thermoplastic and was applied in this application in the thickness range of 0.015–0.025 in. (0.38–0.64 mm).

During insertion of the test window into the vessel, the coated area in the vicinity of the weld underwent localized damage. The weld around the window and any additional damage areas caused by excessive heat, arc strikes, etc., were repaired with a modified thermoplastic hand-applied coating. Both coatings utilized in this program were successful in protecting

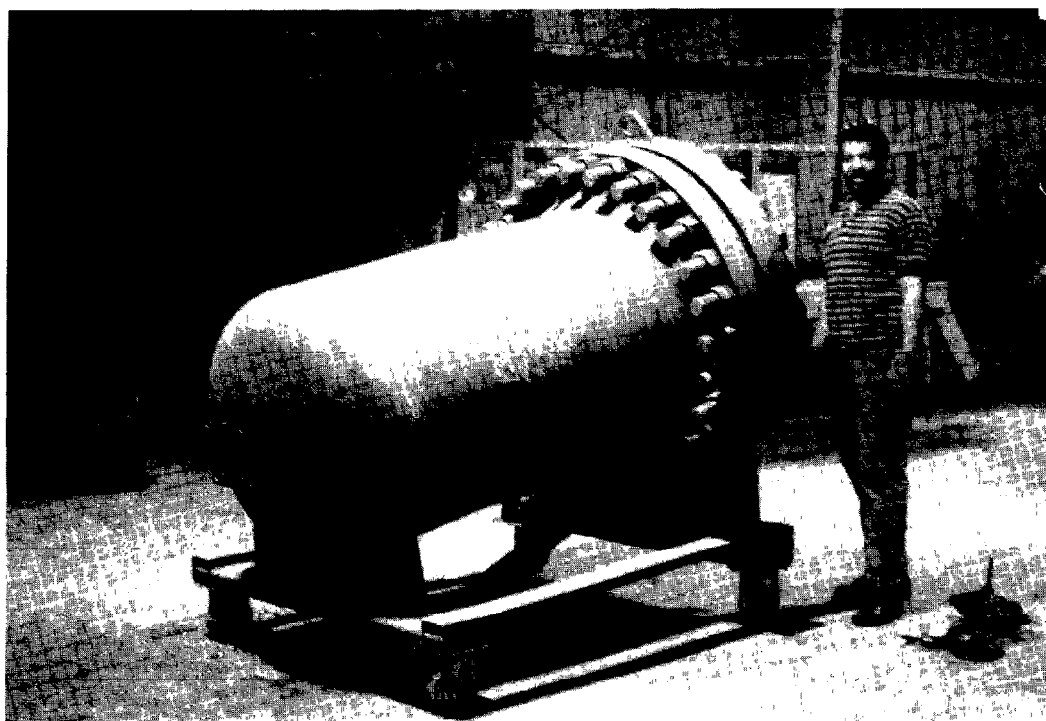


Fig. 3-1—Pressure vessel used for this study (PN 3040-1)

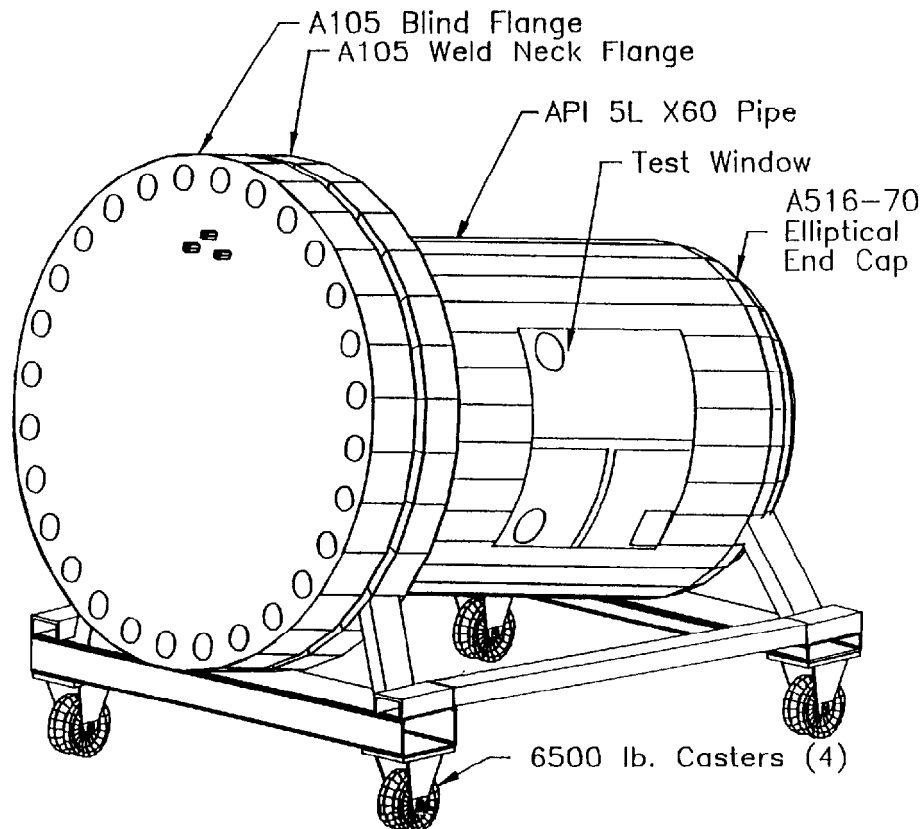


Fig. 3-2—Schematic of pressure vessel detailing materials used

the vessel from damage for the total duration of testing.

The test windows were fabricated as follows. The plate steels were cold rolled to the appropriate radius, and tacked together with strips or “strong-back” welds. Each window typically had a 2 ft (0.6 m) longitudinal weld and a 1 ft (0.3 m) girth or circumferential weld. The parameters used for welding are documented in Table 3-2.

The various test windows also contained: (1) Charpy notches and low heat input weld beads to assist in initiating cracking, (2) Cu plating on the O.D. surface of the window to act as a hydrogen barrier, (3) external hydrogen charging, (4) penetrations to allow cathodic charging on the I.D. surface, and (5) nozzle attachments. The details of the individual test windows evaluated are presented in the six appendices attached to this report.

Limited experiments using standard NACE TM0177 SSC specimens (see Fig. 3-3) and one-side exposed 6 in. $\times$ 6 in. (15 $\times$ 15 cm) plate samples (see Fig. 3-4) were also utilized to assist in obtaining the characteristic cracking AE responses of SOHIC and HIC, respectively.

3.3 Experimental Overview

Several aspects of the experimental procedures are common to all of the experiments such as solution, temperature, etc. These common variables are first addressed followed by a brief discussion of the particular variables which are unique to each experiment.

The test solution for all but one of the evaluations corresponded to NACE TM0177-90, Method A solution (0.5 wt. percent glacial acetic acid, 5.0 wt. percent sodium chloride in distilled water). The solution was saturated with H_2S and maintained at a hydrogen sulfide concentration of approximately 1600–2000 ppm throughout the test duration. One of the evaluations conducted slowly increased the severity of the test environment in several stages by decreasing the pH and increasing the H_2S concentrations of the test solution. The details of this environment will be described later.

All of the evaluations were conducted at room temperature. The test durations ranged from less than 4 days for conditions of rapid failure to 5 weeks for the staged and prolonged experiments.

Efforts were made to measure the residual strain from initial window fabrication to insertion into the test vessel. However, problems with the strain gages during fabrication and welding of the windows prevented quantitative measures. Biaxial strain measurements were conducted on both the vessel and window during the hydrotest to calculate the corresponding outer fiber stresses of the window and the vessel.

The test pressure was applied hydrostatically. Operating pressures in the range of 0–850 psi were examined. H_2S concentration and solution pH were monitored throughout the test period. No depletion in H_2S concentration was observed during the tests due to the high ratio of solution volume to exposed

Table 3-2—Welding Summary

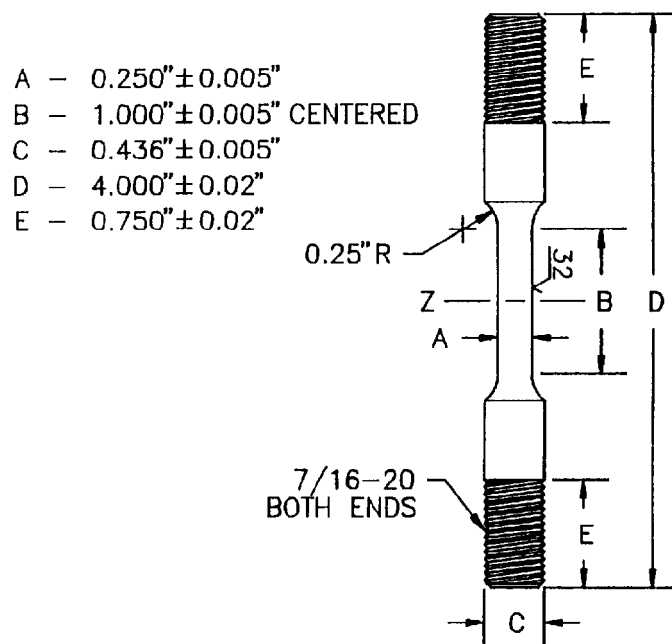
Procedure Specification						
Material Specification			A516-70			
Welding Process			FCAW/SAW/GMAW			
Manual or Machine			Manual and Machine			
Position of Welding			Flat			
Filler Metal Specification			A5.29/A5.23			
Filler Metal Classification ^(a)			E71T1/E76A6-EG-G/ER-705-2			
Flux			Lincoln 880M			
Shielding Gas			75% CO ₂ /25% AR			
Flow Rate			40 cfh			
Single or Multiple Pass			Multiple			
Single or Multiple Arc			Single			
Welding Current			DC			
Polarity			RP			
Welding Progression			N/A			
Root Treatment			Back gouge/Grind to clean metal			
Preheat or Interpass Temperature			100°F min.–150°F max.			
Postheat Treatment			N/A			
Welding Procedure						
	Pass No.	Electrode Size	Welding Current		Travel Speed	Joint Detail
			Amperes	Volts		
Side 1	1–2	0.052	200	25	7.54 IPM	B-12C-2
	3–4	3/32	300	31	14 IPM	
Side 2	1	0.052	200	25	7.5 IPM	
10 KJ Weld	1–2	0.035	110	8	12	

^(a) Applicable only when filler metal has no AWS classification: FCAW Trimark E71T1; SAW LA71 880 M; GMAW ER-705-2.

steel surface area used in these experiments. The solution pH on all but one of the exposures ranged from 2.7 at the initiation of the test to 3.5 at test completion. Hydrogen flux was monitored with a patch probe affixed to the vessel window.

The nondestructive evaluation services included AE, UT, AUT and WFMP. The AE monitoring was used to assess cracking activity both in terms of locating active sites and distinguishing between blister cracking and through-wall cracking. Fig. 3-5

NACE TENSILE SPECIMEN DRAWING # CSD01001



NOTES:

- 1 — MACHINING OF THE SPECIMEN MUST BE DONE CAREFULLY TO AVOID OVERHEATING AND COLD WORKING IN THE GAGE SECTION.
- 2 — THE FINAL TWO PASSES SHOULD REMOVE NO MORE THAN 0.002" OF THE METAL.
- 3 — THE GAGE SECTION MUST BE A UNIFORM DIAMETER WITHIN THE ± 0.005 " TOLERANCE.
- 4 — THE SPECIMEN IS FINISHED TO A SURFACE ROUGHNESS OF 32 MICRO-INCH OR FINER.
- 5 — TOLERANCE OF DIMENSIONS ± 0.005 " UNLESS OTHERWISE SPECIFIED.
- 6 — CONFORMS TO NACE TM0177-90.
- 7 — MINIMUM GAGE DIAMETER (DIMENSION A) MUST BE LOCATED WITHIN 0.125" OF CENTERLINE Z.

Fig. 3-3—Standard SSC specimen per NACE TM0177-90, Method A

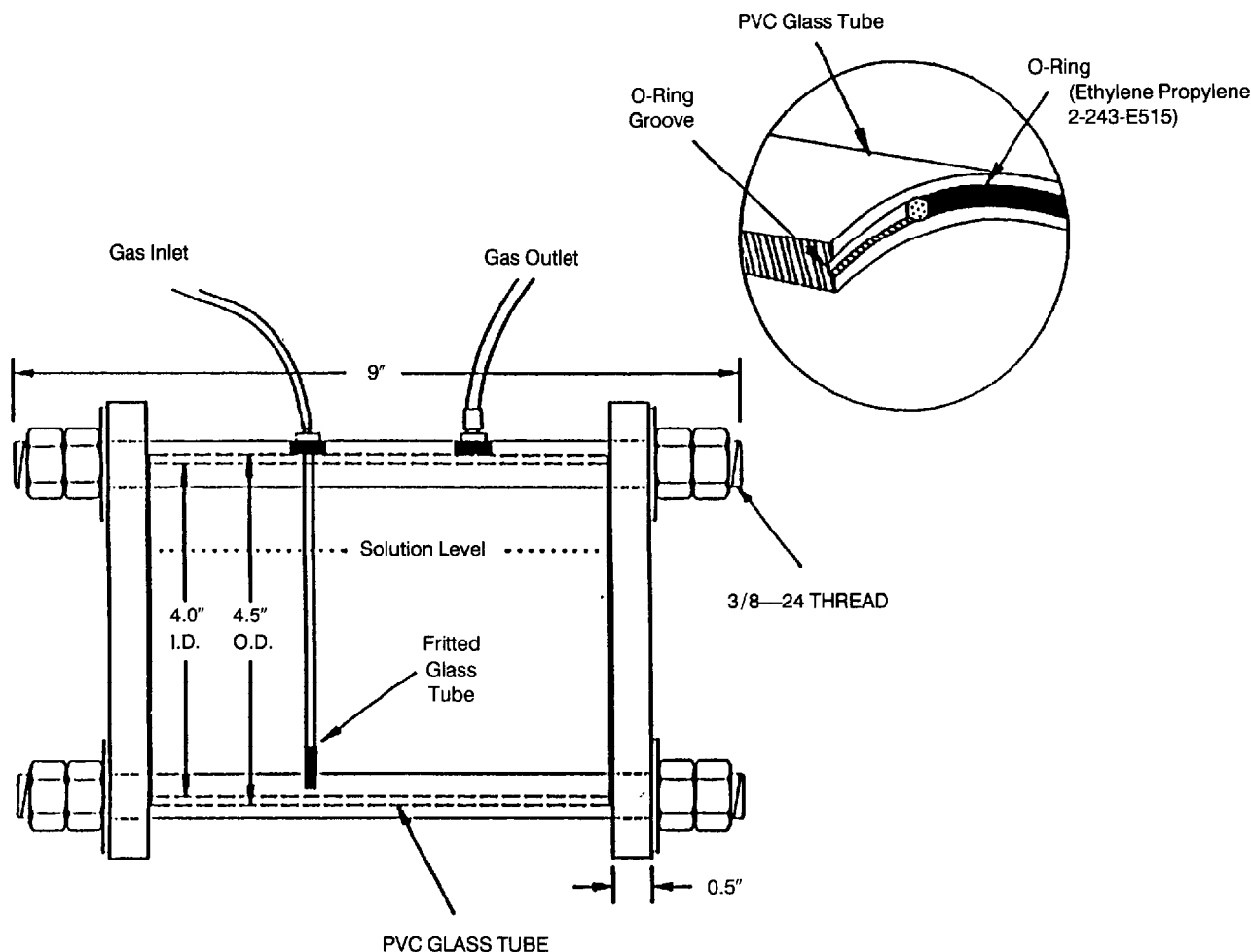


Fig. 3-4—Test fixture used to isolate HIC AE response

shows the AE instrumentation and external hydrogen patch probe used on the test window. Periodic inspection of the window with AUT (T-scan and P-scan) was also conducted to monitor the crack propagation in terms of affected area and through-wall severity. When necessary, crack sizing was conducted using manual sizing UT methods. WFMPT was conducted after cleaning the I.D. surface with a 60-grit flapper wheel to reveal surface crack indications.

Cathodic charging was utilized on two of the evaluations to further increase the severity of the hydrogen charging at the I.D. of the test window or to increase the hydrogen content at the O.D. surface of the test window. Using internal hydrogen charging, levels of charging were produced that were between two to three times those normally obtained in NACE TM0177-90, Method A solution.

3.3.1 Evaluation of Plate Containing Pre-existing HIC Damage. The test window used in this evaluation (see Appendix I) was pre-exposed for 30 days to NACE TM0177-90, Method A solution under one-side exposure conditions to produce pre-existing HIC damage prior to testing in the vessel configuration. The exposure was one-sided in nature and was successful in creating extensive HIC damage

from the I.D. to mid-wall. Metallographic sections were taken from the edge of the pre-exposed window prior to insertion in the test vessel. These sections were evaluated for HIC damage using the formulas given in NACE TM0284 for the calculation of crack length ratio (CLR), crack thickness ratio (CTR) and crack sensitivity ratio (CSR) (see Fig. 2-1). Crack ratios were calculated and location codes given based on cracking observed in the full thickness, $\frac{2}{3}$ thickness and $\frac{1}{3}$ thickness as shown in Fig. 3-6 using techniques developed in prior MPC sponsored research.

Following exposure in the test vessel at pressures ranging from 400–800 psi over a period of 14 days, strips were sectioned from the plate for further metallography and to measure the remaining strength in both the longitudinal and hoop directions. The remainder of the test window was left intact for an ultrasonic round robin program to be conducted later.

3.3.2 Evaluation of Plate Containing Hard Welds (HRC 22-30). The test window used in this evaluation contained hard welds in both the longitudinal and hoop directions of a conventional steel plate. No pre-exposure was conducted prior to testing; consequently no pre-existing HIC damage was pre-

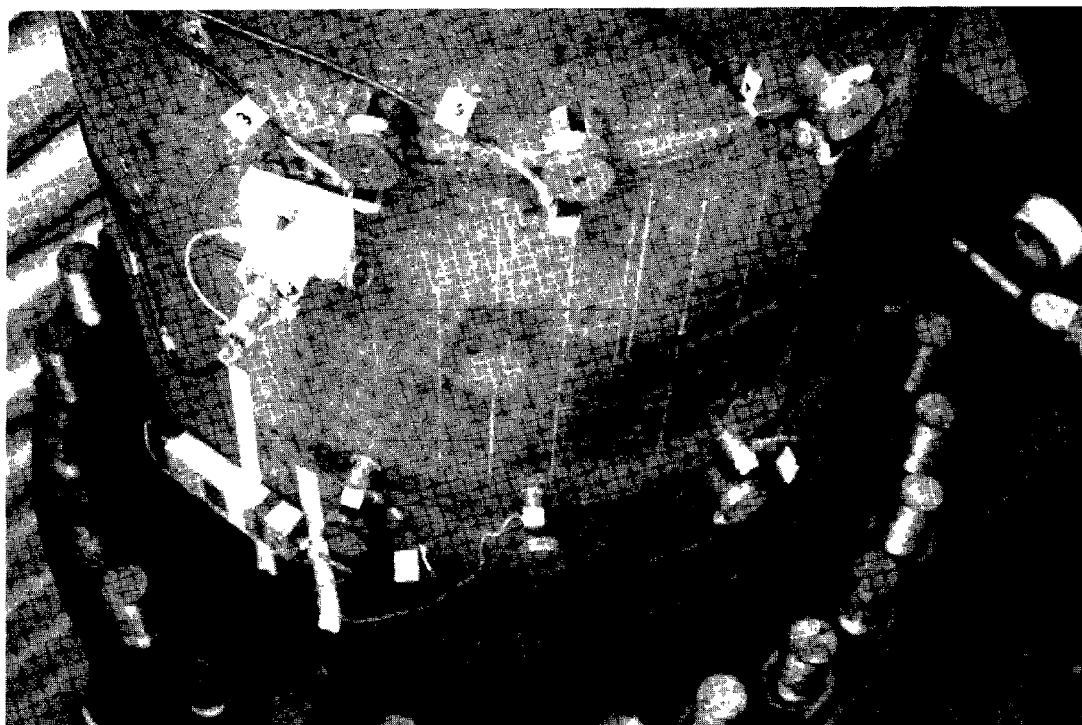


Fig. 3-5—AE instrumentation and external hydrogen patch probe

sent in the window. A schematic of the test window is presented in Appendix II.

The test was initiated and pressurized to 400 psi and allowed to hold for several days. In less than four days a crack emanated through-wall along the girth weld of the window. The crack was completely contained within the hard weld deposit.

A patch containing the full crack was removed and sectioned for metallographic examination and crack measurements. A failure analysis was conducted on the section.

3.3.3 Evaluation of the Repair of a HIC Damaged Vessel. In order to attain information on the repair of H_2S damaged steel vessels, a patch of "HIC resistant" steel, which was found to exhibit a limited degree of cracking susceptibility in prior laboratory tests, was welded into the previous window. The remaining longitudinal weld which was not contained within the removed patch was ground out $\frac{1}{8}$ in. from the I.D. to remove the hard weld deposit and re-welded with E7018. The heat from this welding also served to temper and reduce the hardness of the remaining $\frac{3}{8}$ in. of weld deposit. A schematic of this window is shown in Appendix III.

The window was exposed in the vessel for 28 days. Following the exposure of this window in the test vessel, the window was WFMPPT to reveal any I.D. connected cracks. Extensive sectioning was also conducted to quantify the extent of cracking.

3.3.4 Simulation of the Cracking Behavior of Thick Plate. In order to simulate the effects of thicker material, an electroplated Cu patch was put on the window O.D. which should act as an external

hydrogen barrier and increase the hydrogen concentration under the plating. The expectation was that this would allow HIC to progress past the mid-wall and potentially simulate the hydrogen gradient and cracking response of a thicker wall vessel. The materials chosen and window configuration are presented in Appendix IV.

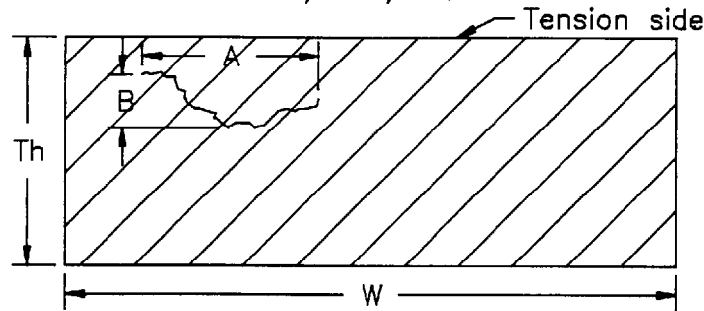
This window was exposed in the vessel for 28 days. Following testing, the window was extensively sectioned and metallographically examined. Strip tensile and Charpy specimens were also removed and tested to determine residual properties.

3.3.5 Evaluation of HIC Resistant Plate/I.D. Surface Cleaning/Severe Hydrogen Charging Conditions. The window chosen for this evaluation is presented in Appendix V. The material chosen represents HIC resistant material, including a TMCP grade steel. The window also contained two electroplated Cu patches to simulate the behavior of thicker plate.

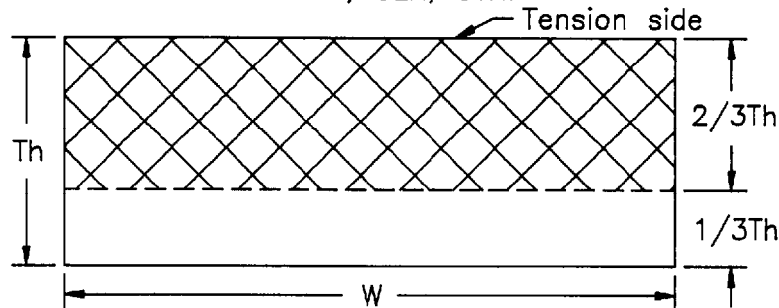
The environmental severity of this test was slowly increased over the 33 day test duration. The increase was conducted in eight stages. The solution ranged from pH 4.4 at the beginning of the exposure (Stage 1) to pH 3.0 at test conclusion (Stage 8). The test was interrupted between Stages 7 and 8 at which point the I.D. surface of the window was cleaned, WFMPPT, and re-exposed to fresh TM0177-90, Method A test solution saturated with H_2S at ambient temperature and pressure. Extensive AUT and AE monitoring were conducted during this exposure sequence. Following the test, the window was extensively sectioned and metallographically examined.

Full Section Analysis:

All cracks included in calculation of CSR, CLR, CTR.

**2/3 Section analysis:**

Only cracks in top 2/3 of section (shaded area) included in calculation of CSR, CLR, CTR.

**1/3 Section analysis:**

Only cracks in top 1/3 of section (shaded area) are included in calculation of CSR, CLR, CTR.

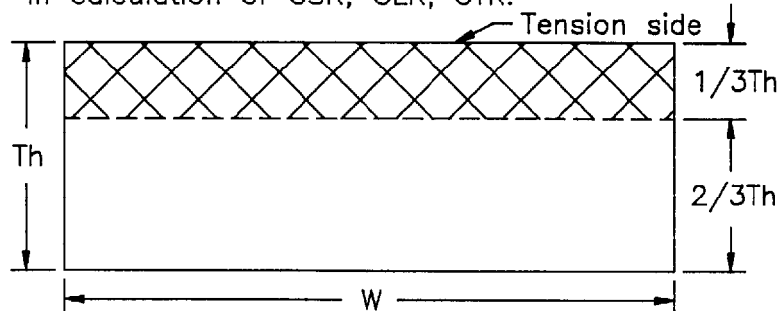


Fig. 3-6—Location coding used to present detailed HIC data

3.3.6 Evaluation of Nozzle Attachments/Effect of PWHT. The window chosen for this evaluation is presented in Appendix VI. This window incorporated several test variables including PWHT versus non-PWHT nozzle attachments, PWHT of the longitudinal seam weld, single-V versus double-V weld geometries of the nozzle attachments (see Fig. 3-7), and as-welded threadlets on the nozzles and weld caps. The environment corresponded to standard NACE TM0177-90, Method A solution. The test exposure lasted 24 days. Following testing, the window was WFMPPT, manually sized for flaws using UT, and extensively sectioned for metallographic examination.

3.3.7 Verification of AE Signature for Hydrogen Blistering and SSC. In conjunction with the API full scale vessel testing, an additional experiment was conducted which measured the AE behavior of an unstressed plate material subjected to a sour environment. In this experiment, sections of plate steel similar to that used in the vessel experiments were exposed to NACE TM0177-90, Method A solution. This exposure was conducted on one side only with no applied stress. Under these conditions, the cracking should have been predominately HIC. The AE data from this experiment were compared to those obtained from previous vessel tests with applied stress. This analysis was used to help distinguish between

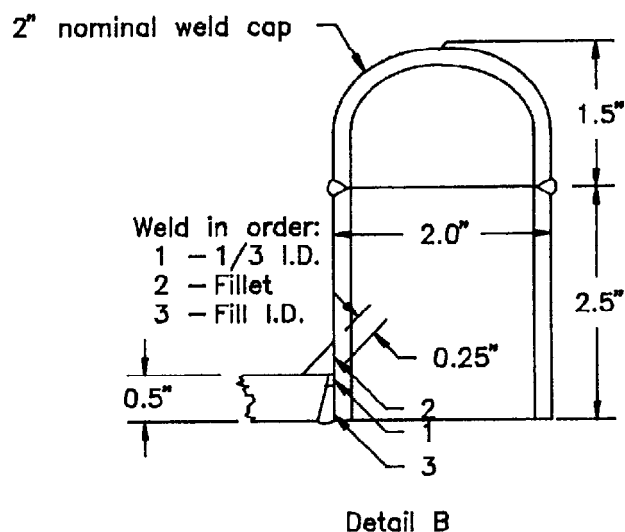
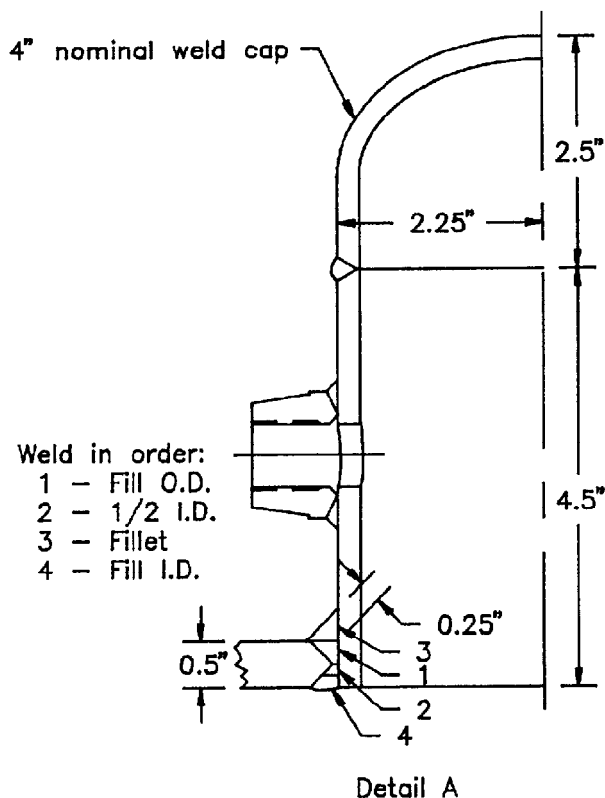


Fig. 3-7—Single-V and double-V attachment weld configurations

HIC and through-wall cracking by SOHIC. A schematic of the experimental set-up was previously shown in Fig. 3-4.

Another experiment was conducted to measure the AE behavior of SSC specimens subjected to an applied tensile stress and exposed to a NACE TM0177-90, Method A environment. The AE data from this experiment were compared to those obtained from previous vessel tests with applied stress.

4.0 Results and Discussion

The findings of this investigation have been evaluated based on their relevance to the serviceability of refinery wet H₂S equipment. For clarity, the test results and the findings they support have been separated into the following categories:

1. Materials Selection
2. Fabrication
3. Inspection
4. Vessel Design and Integrity

4.1 Materials Selection

4.1.1. Ultra-low sulfur steels (i.e., ASTM A516-70 and A841 with ≤ 0.002 wt. percent sulfur) exhibited higher resistance to HIC than conventional and low sulfur conventional steels.

It was shown that the ultra-low sulfur steels had generally fewer HIC indications, as observed by both AUT T-scan (Figs. 4-1a and 4-1b) and by metallographic sectioning (Appendix V and VI) than found in conventional steels which were very susceptible to

HIC in the NACE TM0177 solution environment. The ultra-low sulfur ASTM A516-70 steel was found to have small isolated areas of HIC while the ASTM A841 steel produced by thermo-mechanically controlled processing (TMCP) was essentially free of HIC in the bulk plate. By comparison, the number of HIC indications found in low sulfur conventional ASTM A 516-70 steel was considerably greater. In conventional ASTM A516-70 steel with even higher sulfur content, the cracks produced by HIC were larger than in the lower sulfur materials.

4.1.2. The susceptibility of the base metal to HIC and SOHIC decreased with a corresponding decrease in sulfur content and decrease of microstructural banding. This was observed at the standard NACE TM0177 hydrogen charging conditions and at very severe hydrogen charging conditions, i.e., two to three times NACE TM0177 levels. The exposure to very severe hydrogen charging conditions did produce substantial SOHIC adjacent to the welds in all materials including the ultra-low sulfur steels. This is further discussed in Sections 4.2.6 and 4.4.3.

Fig. 4-2 shows the percent CTR as a function of steel type, sulfur content and banding index in accordance with ASTM E1268. While two data points were not available to complete the spectrum, there still exists a strong correlation amongst the variables. The maximum base metal CTR values observed decreased with a corresponding decrease in sulfur content and decrease of microstructural banding at both the

standard NACE TM0177 hydrogen charging conditions and at very severe hydrogen charging conditions, i.e., two to three times NACE TM0177 levels. The maximum resistance to cracking was observed in the TMCP A841 steels with a sulfur content of 0.001 wt. percent and banding index of 0.20.

The above mentioned trends only represent the behavior of the base metal at stress levels ranging from 60–80% of the specified minimum yield strengths. The behavior of the steels at areas of high residual tensile stress such as at weldments or areas containing local stress concentrations may in many situations produce more severe cracking with respect to SOHIC. Fig. 4-3, obtained from the literature,⁷ shows the effect of sulfur content on the time-to-failure of linepipe steels under various levels of applied tensile stress. It indicates that the susceptibility to SOHIC, as determined by time-to-failure, increases with decreasing sulfur content, in particular at the higher applied stress levels. Therefore, the lower sulfur content, while decreasing susceptibility to HIC, may under very severe environments and high applied or residual tensile stress produce an increased susceptibility to SOHIC. This topic is addressed further in Sections 4.2.6 and 4.4.3.

4.1.3. Based on both UT and metallographic sectioning, the only steels to exhibit no HIC in the base metal were the TMCP steels (i.e., ASTM A841). Both low sulfur and ultra-low sulfur A516-70 steel exhibited low levels of HIC which increased with sulfur content.

As previously shown in Figs. 4-1a and 4-1b, supported by metallographic sectioning results (Appendix V and VI), the TMCP appears to resist HIC even under very severe conditions of hydrogen charging. Under similar conditions, the HIC resistant and low sulfur conventional steels exhibited significant levels of HIC.

4.1.4. ASTM A 53 ERW pipe used in the nozzles did not exhibit HIC. However, the ASTM A234 WPB weld cap material did exhibit substantial HIC.

Fig. 4-4 shows the nature of the HIC observed in the ASTM A234 WPB weld cap. The cracking was at a significantly high level as determined by metallographic sectioning (Appendix VI), but had a different character than the HIC found in conventional steels made to similar impurity levels. The HIC was not restricted to planar cracks as commonly observed in plate materials. Due to the variations typically found in the ASTM A 53 steels, the crack-free behavior of the ASTM A 53 evaluated in this study may not be typical of all materials made to this specification.

4.2 Fabrication

4.2.1. Welds made with low heat input (i.e., single pass welds made with heat input ≤ 10 kJ/in.) were found to increase the presence of SOHIC in the HAZ of the base metal adjacent to the low heat input welds.

In the present study, through-thickness cracking resulting from SOHIC was predominantly found to occur in the base metal adjacent to the weld in the

heat affected zone rather than in the bulk plate material. Where low heat input welds were tested without PWHT, an increase in susceptibility to SOHIC was observed. Fig. 4-5 shows the comparison of the cracking severity in terms of CTR values in the vicinity of the low heat input welds on the I.D. of the specimen versus that observed for the fabrication weld without the low heat input weld. The CTR value on the I.D. $\frac{1}{3}$ section of the plate in the area of the low heat input weld was over eight times greater than the value in the comparable location without the low heat input weld. The reason for this increase appears to be related to at least two factors: (1) the locally high hardness which can produce a stress concentrator resulting from SSC, and (2) the locally high residual tensile stress which can also exist in the area of the low heat input weld.

4.2.2. SSC initiating from localized hard weld zones on the exposed surface of conventional steels was found to propagate into the softer base metal via SOHIC. However, these cracks only propagated to the extent of the HAZ.

Fig. 4-6 shows an example of the situation mentioned above. As represented in Fig. 4-6, many instances of cracking were observed where low heat input welding initiated cracks on the I.D. of the vessel as a result of SSC. However, the extent of subsequent propagation of these cracks in conventional steels by SOHIC was generally limited to a region within the HAZ of the base metal adjacent to the weld.

4.2.3. SSC initiating from localized hard zones on the exposed surface of "HIC Resistant" and TMCP steels was also found to propagate into the softer base metal via SOHIC. However, in these steels the cracks generally propagated *past* the extent of the HAZ.

Ultra-low sulfur steels (i.e., "HIC Resistant" A516-70 and A841 TMCP Steels) tested in this program also exhibited propagation of SSC cracks from low heat input welds. However, in contrast to the cracking behavior for conventional steels mentioned previously (Section 4.2.2), subsequent crack growth via SOHIC in these materials propagated beyond the extent of the HAZ (See Fig. 4-7).

4.2.4. When hard welds (i.e., > 22 HRC or > 240 BHN) were used in the vessel fabrication, the cracking mechanism which led to complete through-wall failure was SSC. Cracking occurred rapidly (< 4 days) without producing SOHIC.

In one experiment, the test window was fabricated by a procedure which produced a hard weld metal deposit. The hardnesses were in the range of HRC 22–30. As shown in Fig. 4-8, complete through-wall cracking was observed which was completely confined to the weld metal. Crack propagation by SSC was rapid at relatively low levels of internal vessel pressure (i.e., 400 psi), and resulted in loss of pressure of the H_2S containing solution. This is the only case in the present study where a through-wall crack was obtained. No through-wall cracking was observed in other tests even where (1) substantial amounts of

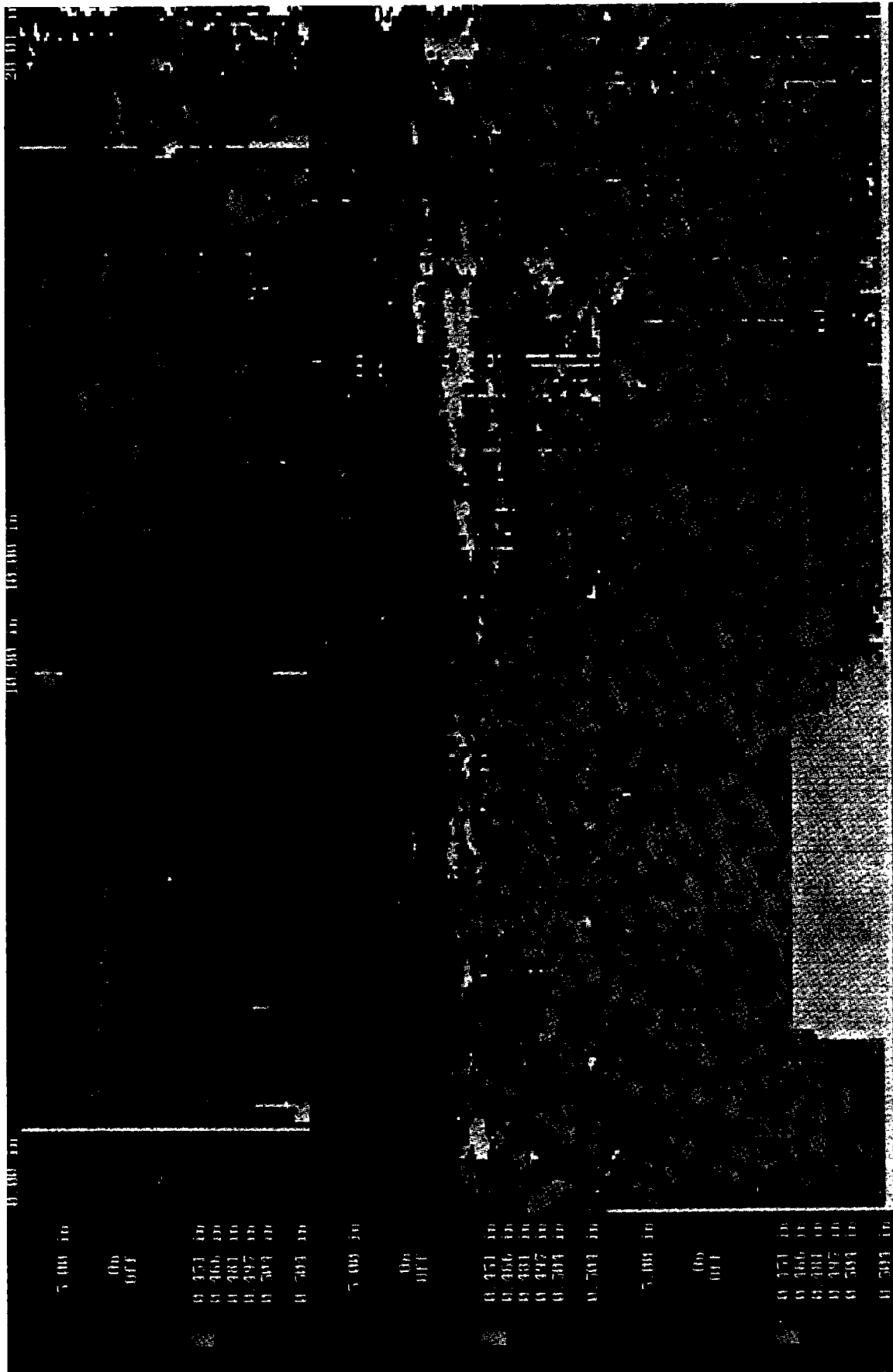


Fig. 4-1a—AUT T-scan data taken after Stage 7 of the environmental staging experiment

AUT T-SCANS

The print-outs of the AUT T-scans shown
in Figures 4-1a, 4-1b, and 4-15
are shown in full color on the following three pages.

Cracking in Wet H₂S Service

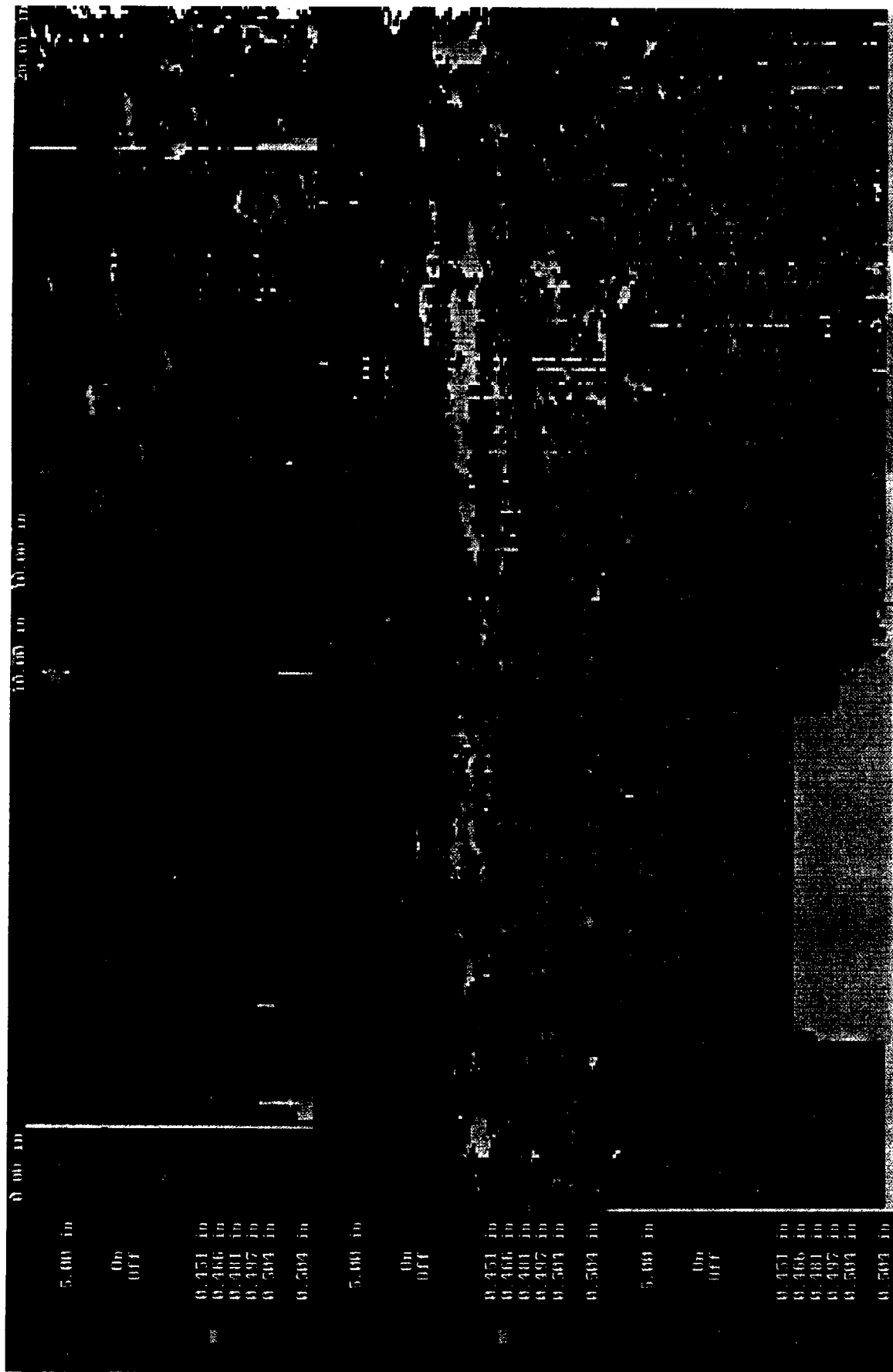


Fig. 4-1a---AUT T-Scan Data Taken After Stage 7 of the Environmental Staging Experiment

WRC Bulletin 396

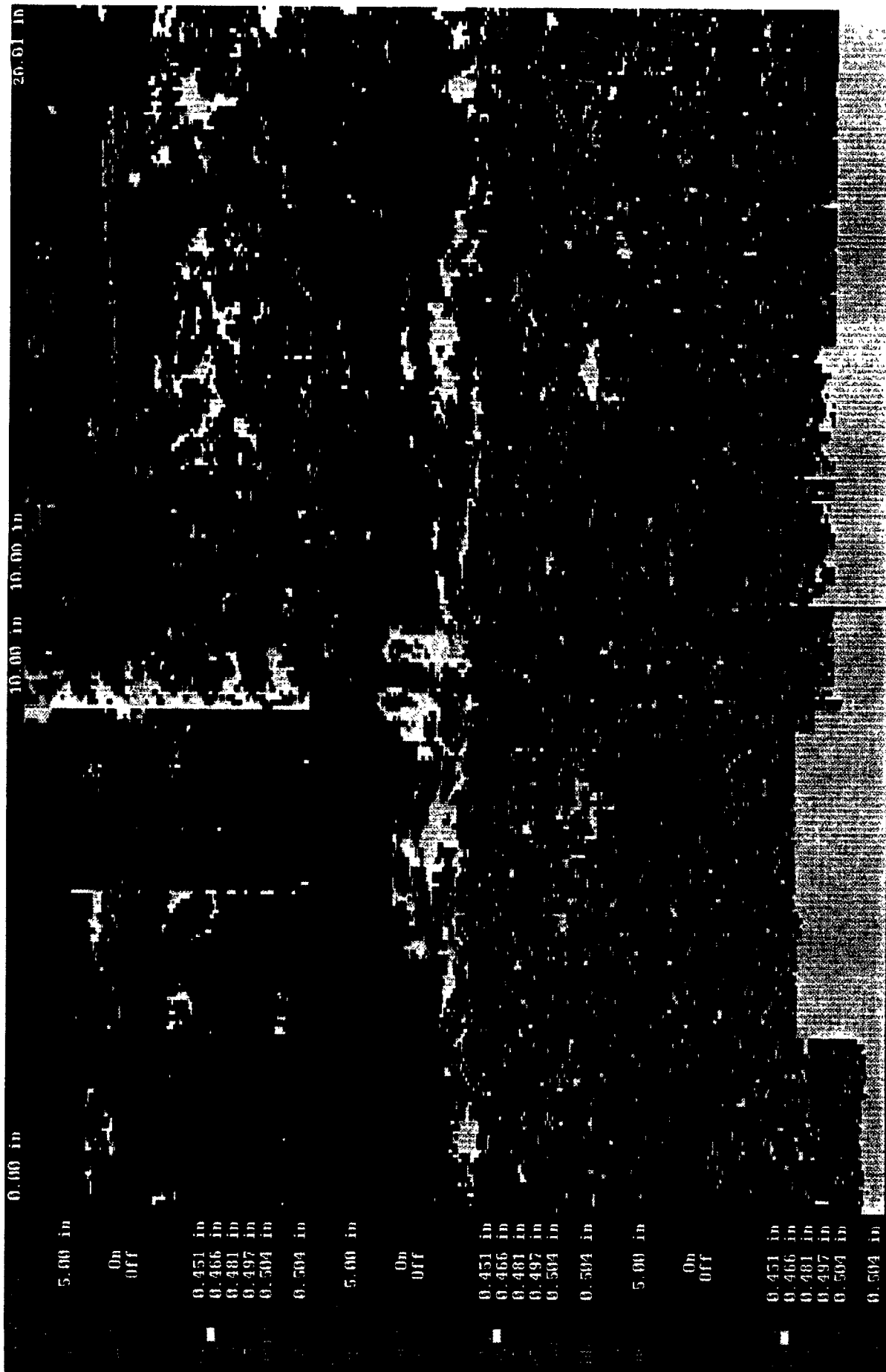


Fig. 4-1b—AUT T-Scan Data Taken After Stage 8 of the Environmental Staging Experiment

Cracking in Wet H_2S Service



Fig. 4-15 -AUT T-Scan Data Taken at the Conclusion of the Simulation of Thick Plate Behavior Evaluation

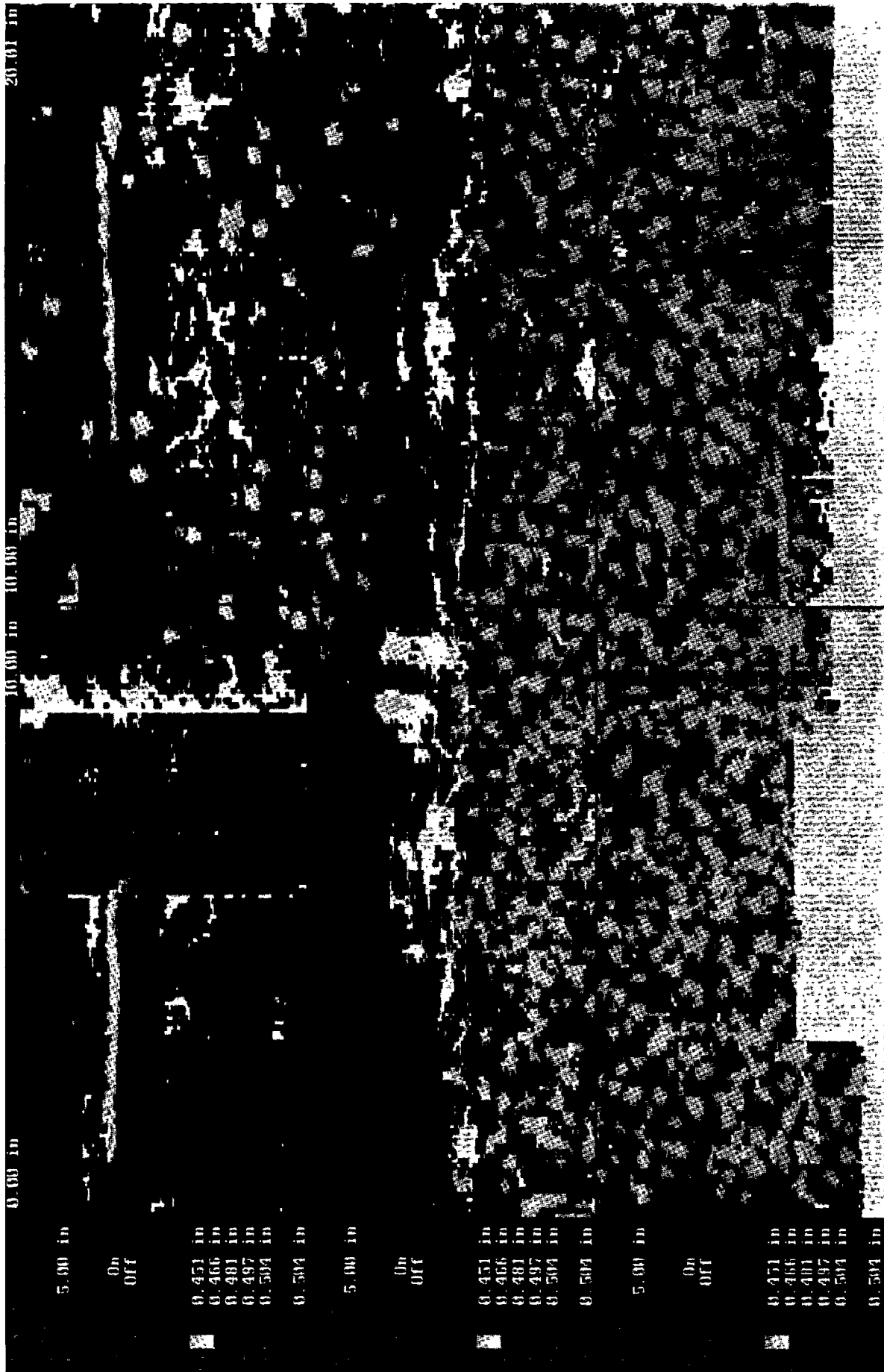


Fig. 4-1b—AUT T-scan data taken after Stage 8 of the environmental staging experiment

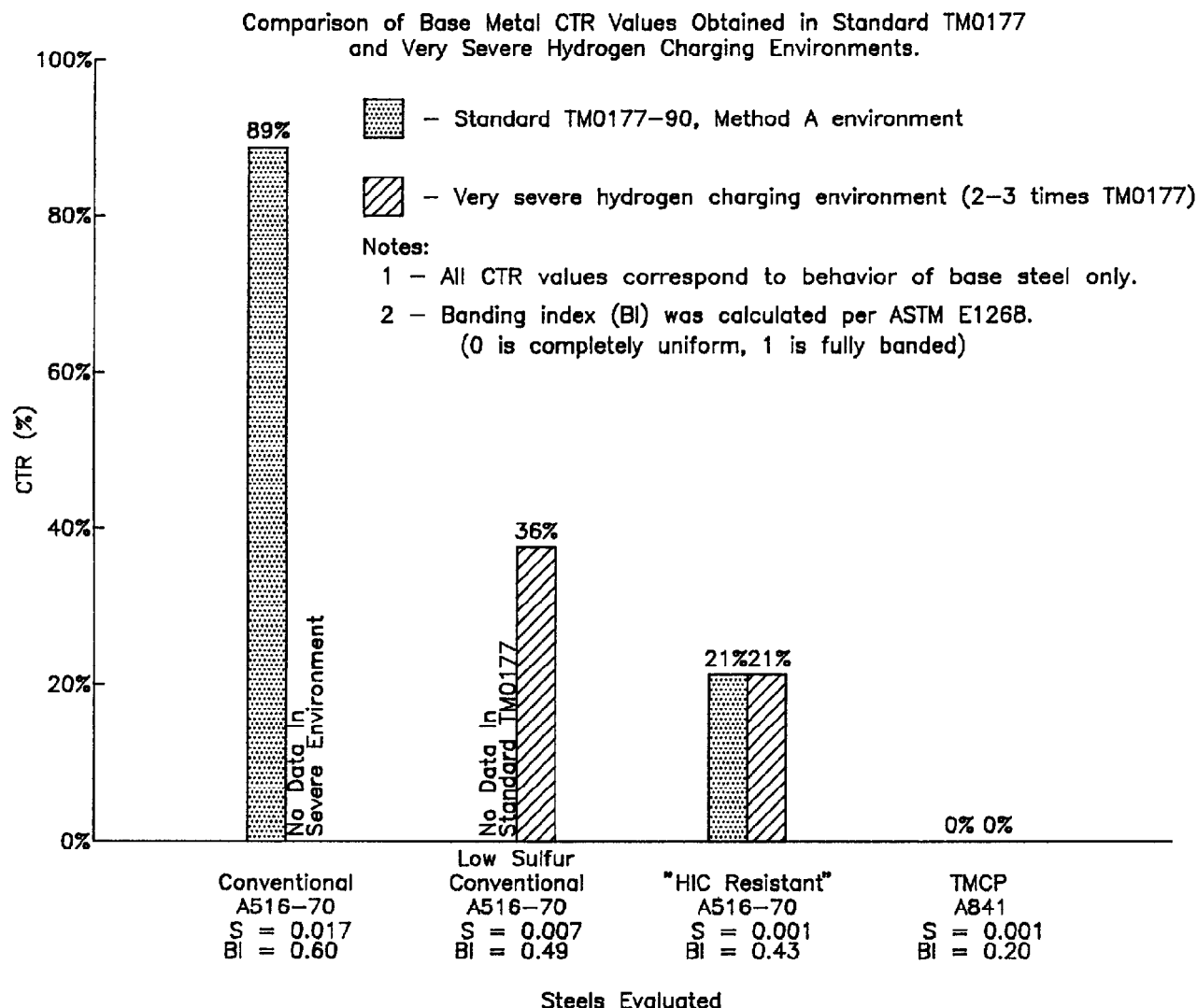


Fig. 4-2—Comparison of base metal CTR values obtained in standard NACE TM0177 and very severe hydrogen charging environments

HIC and SOHIC (i.e., up to 60% of the wall thickness) were noted, and (2) high levels of internal vessel pressure (i.e., 850 psi) were combined with severe hydrogen charging levels during exposure.

4.2.5. Short cracks produced by SSC and regions of residual tensile stresses were found to be effective initiation sites for SOHIC.

The most significant areas in terms of susceptibility to through-wall crack propagation from SOHIC were regions immediately adjacent to low heat input welds (See Fig. 4-5). SOHIC easily initiates from these low heat input weld locations. Factors which cause this phenomenon are related to (1) the formation of small cracks from SSC in hard weld zones and (2) the tensile residual stresses in these regions.

4.2.6. SOHIC initiated from low heat input welds in the conventional, low sulfur conventional, "HIC Resistant," and TMCP steels at the standard NACE TM0177 hydrogen charging conditions and at very severe hydrogen charging conditions, i.e., two to three times NACE TM0177 levels. The absolute through-wall crack penetration observed at the stan-

dard NACE TM0177 hydrogen charging conditions decreased with a decrease in sulfur content and decrease in microstructural banding. However, under very severe hydrogen charging conditions, the low sulfur conventional, "HIC Resistant," and TMCP steels exhibited through-wall crack penetrations ranging from approximately 30–50%, irrespective of the sulfur content or degree of microstructural banding.

Fig. 4-9 compares the through-wall crack penetration observed at both the standard NACE TM0177 hydrogen charging conditions and very severe hydrogen charging conditions, i.e., two to three times NACE TM0177 levels. All of the values represent cracking along the longitudinal seam weld at low heat input weld bead locations, except the "HIC Resistant" steel in the standard NACE TM0177 environment which corresponded to the longitudinal weld only. At the standard NACE TM0177 hydrogen charging conditions, a decrease in the through-wall crack penetration is observed with a decrease in sulfur content and decrease in microstructural banding. However, under the very severe conditions of hydro-

INFLUENCE OF SULFUR CONTENT ON HYDROGEN INDUCED FRACTURE IN LINEPIPE STEELS

Met. Trans. A, Vol. 10A,
Nov. 1979, p 1691

Makio Iino (NSC)

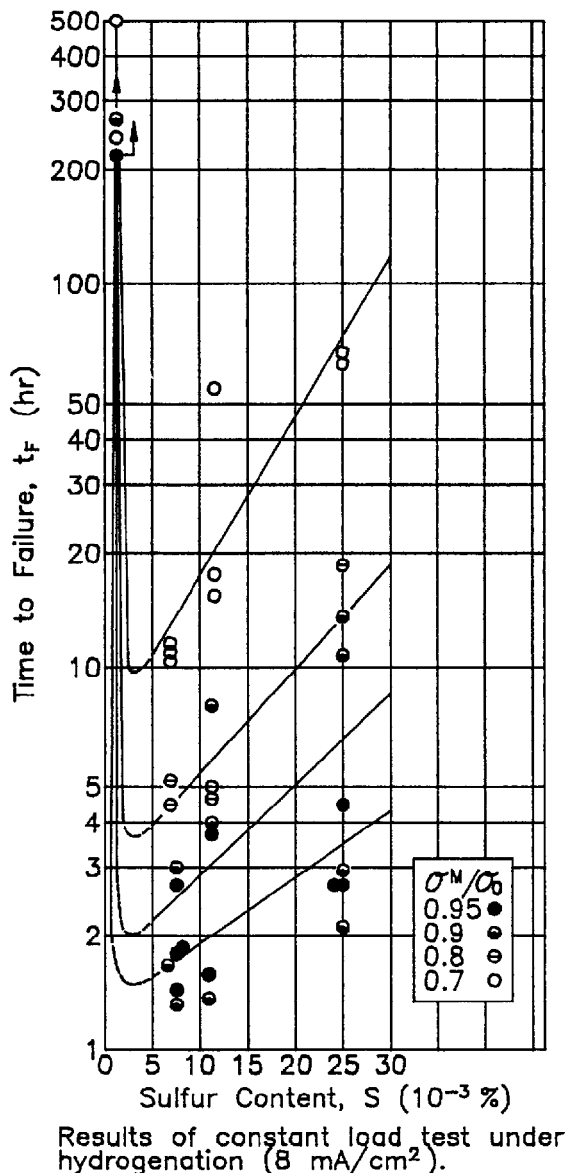


Fig. 4-3—Results of constant load tests under hydrogenation showing time to failure as a function of sulfur content

gen charging, no relationship was observed between the sulfur content, degree of microstructural banding, and through-wall crack penetration. Through-wall cracks were observed to penetrate approximately 30–50% in the low sulfur conventional, “HIC Resistant” and TMCP steels.

Additional factors which may control the depth of HIC and SOHIC in wet H₂S environments include (1) the differences in threshold hydrogen concentrations in the various steels to produce cracking and (2) the natural hydrogen concentration gradient in the steel resulting from the I.D. exposure of the vessel to the

wet H₂S environment. This situation is shown schematically in Fig. 4-10. The hydrogen concentration in the steel is a maximum at the I.D. surface exposed to the test solution resulting from the charging of hydrogen produced by the sulfide corrosion reaction. At the O.D. surface, the hydrogen concentration is zero because there is no barrier to the egress of hydrogen from the steel on this surface.

The depth to which cracking can occur in the steel is dependent on the hydrogen gradient through the wall of the vessel. Once the hydrogen concentration becomes less than the threshold hydrogen concentration to produce cracking, the cracks will tend to arrest unless the driving force (i.e., stress or hydrogen concentration) is increased further. The results of this program show that the threshold hydrogen concentration to produce cracking in the ultra-low sulfur advanced steels is higher than for conventional steels. Therefore, the depth of cracking is restricted to varying degrees as shown in Fig. 4-10. The relationship is not simply linear, but is complicated by the changes in cracking susceptibility through the thickness of the material caused by microstructural differences such as centerline segregation or banding. In these cases, cracks can develop and propagate, in particular steels to a further extent than indicated in the figure.

4.2.7. No difference was observed in the cracking behavior of attachment welds using single-V and double-V weld configurations made per ASME specifications. Both were very resistant to cracking in this study even in the severe NACE TM0177 environment. Therefore, weld configuration did not appear to have a significant effect on susceptibility to cracking.

As shown in Appendix VI, the results from metallographic sectioning of attachment welds did not show significant SOHIC susceptibility using either single-V or double-V welds under what was considered to be severe conditions of hydrogen charging. While these welds showed AE responses and manual UT indications of potential cracking, no through-wall cracks were found in these regions by the metallographic sectioning (See Section 4.3.3.).

4.3 Inspection

4.3.1. Wet Fluorescent Magnetic Particle Testing (WFMP) was able to identify HIC and SOHIC which intersected the I.D. surface of the plates. HIC indications were located in the bulk plate material whereas SOHIC was primarily found in the HAZ region of the base metal. In most cases examined, the depths of the surface cracking identified by WFMP were shallow and not associated with SOHIC of significant through-wall extension.

Metallographic sectioning was performed in regions inspected by WFMP that were found either to have crack indications or no crack indications. Fig. 4-11 gives the results of these examinations. It can be seen that WFMP was able to identify surface cracking as evidenced by the high CTR values in the 1/3

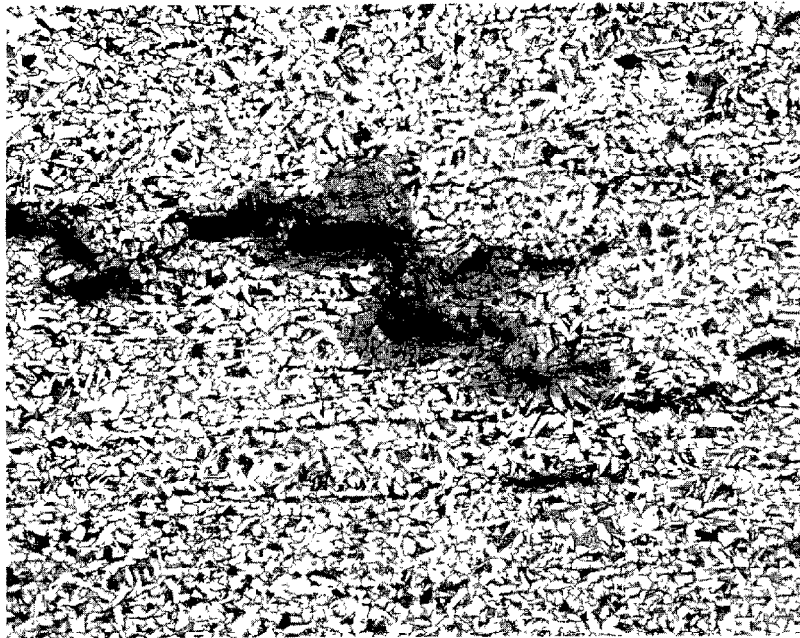


Fig. 4-4—Morphology of typical HIC observed in the ASTM A234 WPB material; magnification 100× (PN 3343-13)

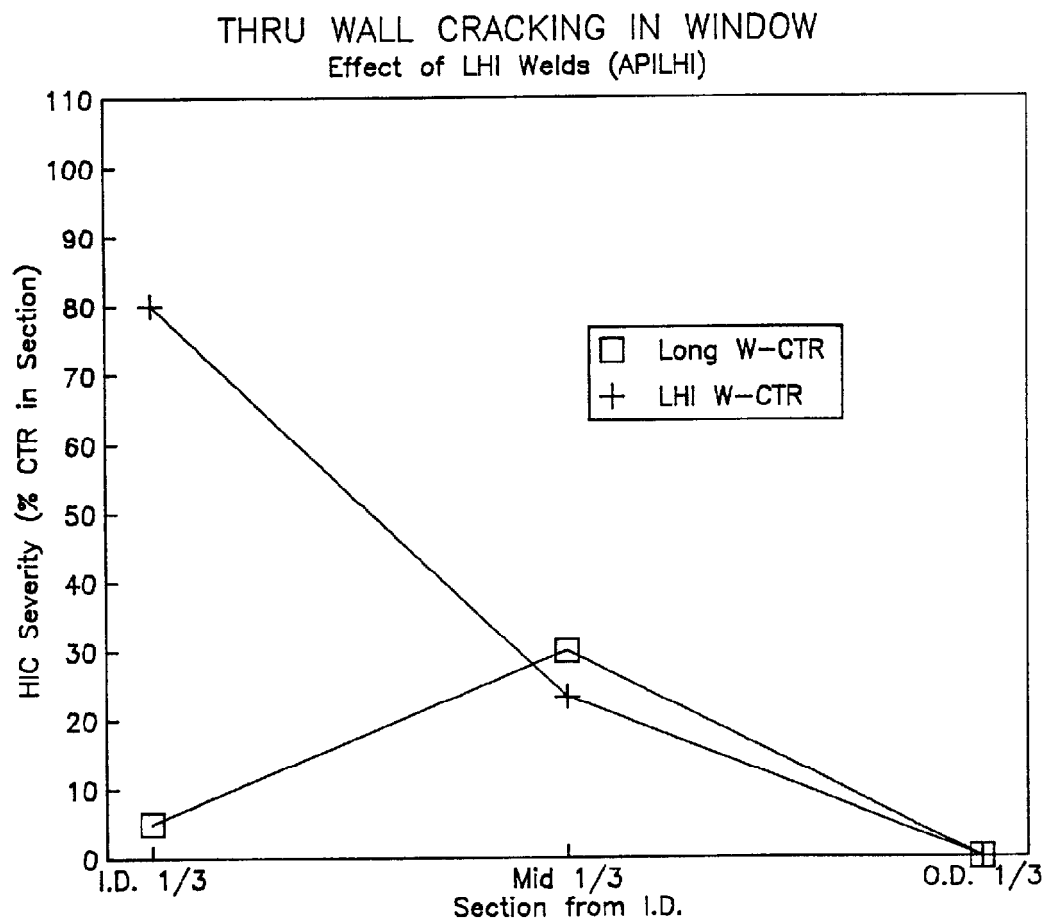


Fig. 4-5—Through-wall cracking at LHI weld locations along longitudinal weld seam

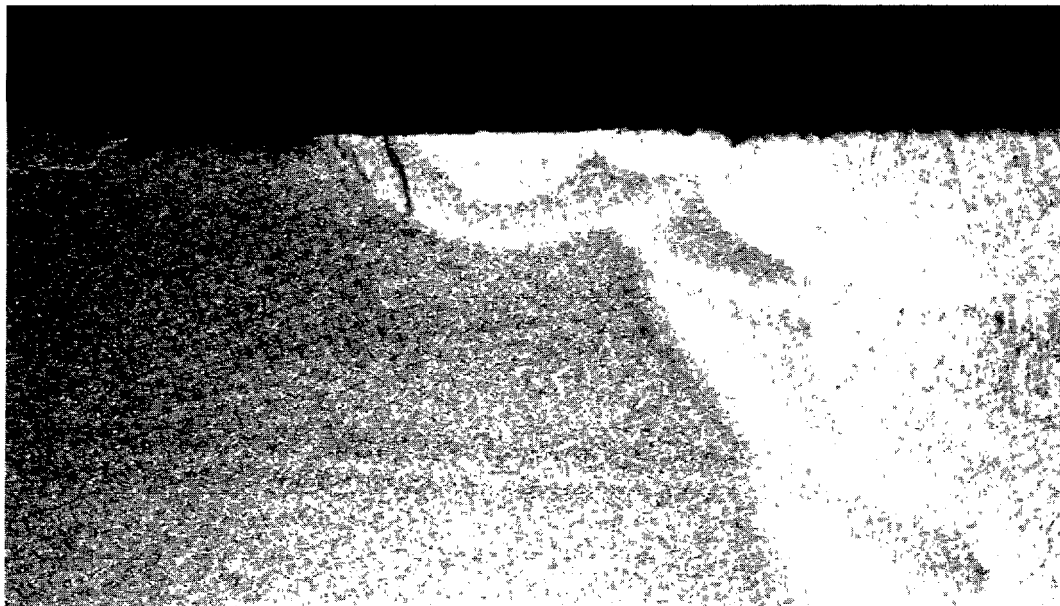


Fig. 4-6—Photograph of SSC at LHI weld in conventional steel: crack arrests at extent of HAZ (PN 3343-3)

thickness from the I.D. surface. The indications in the bulk plate were from HIC which intersected the I.D. surface while the indications in the weld area were more likely to be related to cracks from SSC and SOHIC. However, in most cases, the WFMPT indications were associated with limited surface cracking and not with regions of substantial through-wall cracking via SOHIC.

4.3.2. I.D. surface cleaning required for WFMPT resulted in an increase in SOHIC upon re-exposure of the vessel to the wet H_2S environment. This appeared to be caused by the removal of the protective sulfide films by cleaning and surface preparation prior to WFMPT.

An experiment specifically designed to observe the effect of surface cleaning on cracking susceptibility was conducted. The results are shown in Figs. 4-1a and 4-1b (AUT), Fig. 4-12 and Appendix V. In this experiment, a test window was exposed to an increasingly more severe environment using the sequence given in Table 4-1. Upon completion of the seventh exposure sequence, the test was stopped, the solution drained from the vessel and the I.D. surface of the window cleaned for WFMPT. The I.D. surface was found to have a thick sulfide scale which was removed prior to inspection. Upon completion of the cleaning and inspection, the test was restarted with fresh NACE TM0177 solution saturated with H_2S at ambient temperature and pressure.

Immediately upon restarting the test, it was apparent that an increase in cracking was occurring. As shown in Fig. 4-12, the upper band AE response, typical of SSC and SOHIC, was more active than in the previous exposure sequences of this test. Additionally, AUT P-scans conducted after this eighth exposure sequence showed more through-wall cracking oriented adjacent to both the longitudinal and trans-

Table 4-1—Environmental Staging Description

Stage	Number of Days	H_2S Conc. (ppm)	Solution pH	Acetic Acid (wt. %)	HCL (wt. %)	Current Density (μA)
1 ^(a)	5	325	4.4	—	—	—
2 ^(a)	6	2100	4.2	—	—	—
3	1	2100	4.2	0.05	—	—
4 ^(a)	5	2100	4.1	0.50	—	—
5 ^(a)	7	2100	4.2	0.50	—	55
6	3	2100	4.2	0.50	—	145
7 ^(a)	6	2100	2.9	0.50	0.02	145
Solution was removed and the exposed surface of the window was cleaned. A new batch of standard NACE TM0177 solution was immediately added.						
8 ^(a)	11	2100	3.0	0.50	—	145

^(a)An AUT scan was conducted at the conclusion of the stage.

The pressure was maintained at 50 to 500 psi throughout the first seven stages. The pressure varied from 500 to 850 psi during stage 8.

verse fabrication welds in the window than was found in previous scans. However, the extent of HIC extension, as determined by AUT T-scan, did not appear to increase noticeably between the seventh and eighth exposure periods.

The increase in through-wall oriented cracking was confirmed by metallographic sectioning. It was identified to be caused primarily by SOHIC.

4.3.3. SOHIC locations with through-wall extension that were not revealed by WFMPT were found by automated shear wave (P-scan) ultrasonic testing. Manual relative arrival time UT methods were used for sizing of defects in the bulk plate areas and were confirmed by metallographic sectioning. Neither manual UT or WFMPT proved to be fully reliable for the identification of cracks in attachment welds for nozzle penetrations as revealed by metallographic sectioning.

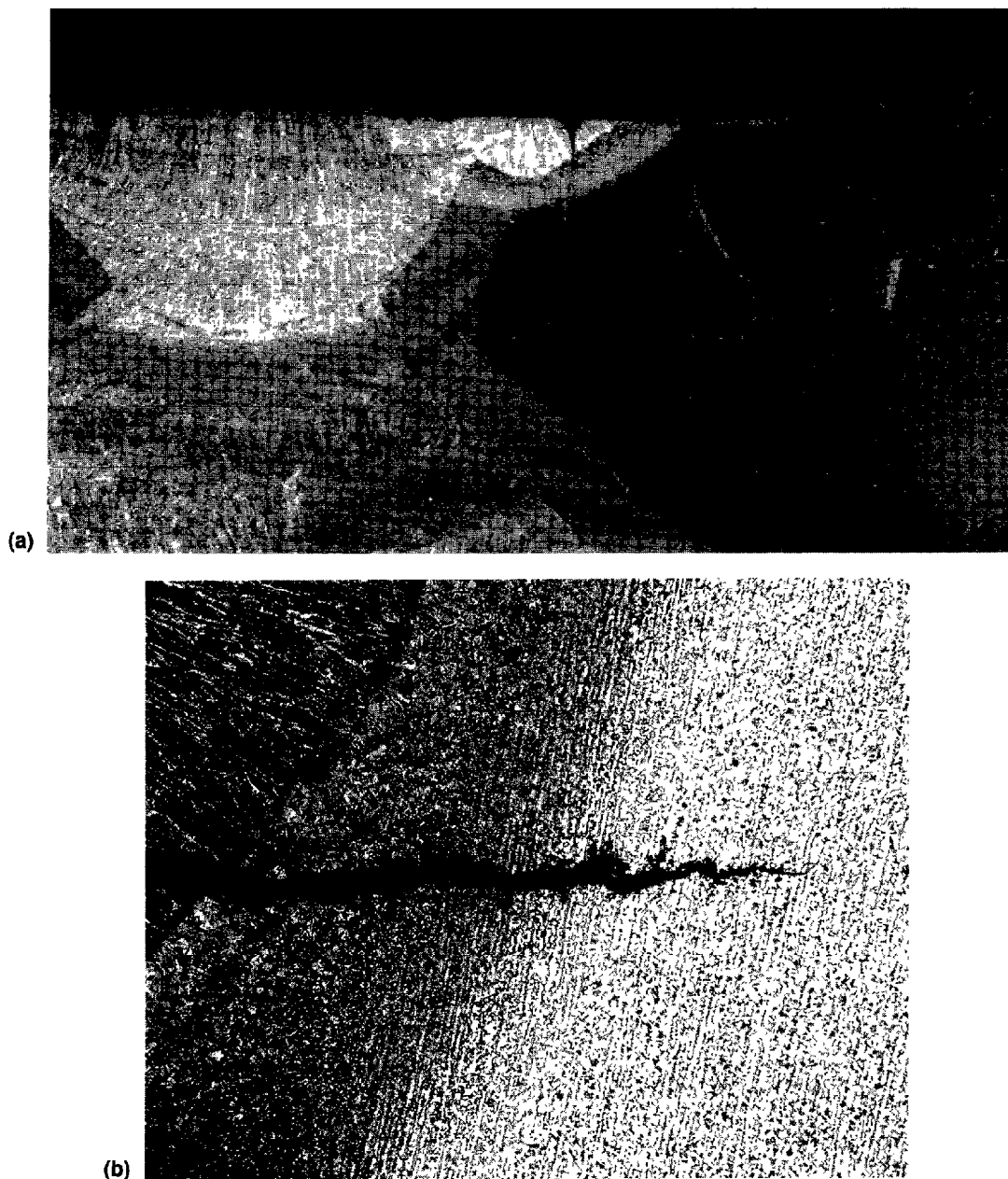


Fig. 4-7—SSC in advanced steel. Crack propagates past extent of HAZ: (a) 35 mm photograph (PN 3343-10); (b) micrograph, magnification 50 $\times$ (PN 3343-15)

In several cases, AUT shear wave examination conducted on the O.D. of the test window was able to identify indications in the plate which were associated with internal SOHIC with a through-wall orientation. In some cases, these crack indications were not identified by subsequent WFMPPT on the I.D. surface in the same location. Fig. 4-13 shows the O.D. view of the WFMPPT conducted on one of the windows prior to sectioning (See Appendix V). It shows the locations of many surface crack indications due to (1) HIC near the I.D. surface, (2) SSC associated with low heat input welds, and (3) SOHIC in the HAZ area of the base metal.

The results of the WFMPPT, however, did not reflect

the extent of the through-wall oriented cracking from SOHIC in the longitudinal seam weld, particularly on the side(s) of the weld bordered by the ultra-low sulfur steels. By comparison, AUT P-scan was able to detect the presence of through-wall oriented cracking in the longitudinal weld HAZ extending nearly the entire length of this weld. As shown in Appendix V, the metallographic sectioning confirmed the presence of these cracks.

One limiting aspect of the AUT P-scan was its inability to quantitatively indicate the extent of through-wall crack propagation. The extent of through-wall cracking adjacent to the longitudinal seam weld, observed under severe hydrogen charging

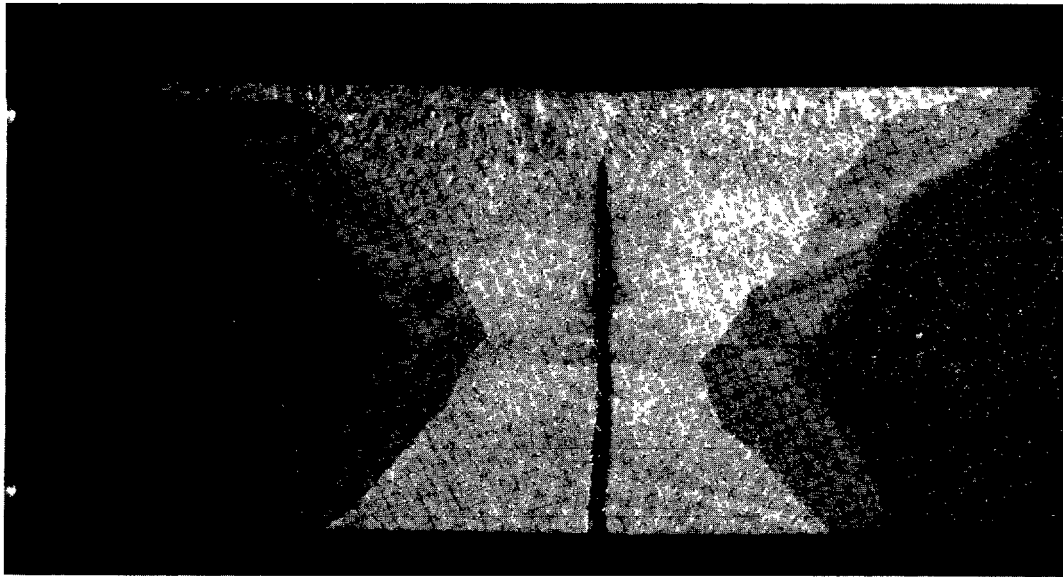


Fig. 4-8—Micrograph of SSC just adjacent to through-wall crack in conventional steel (PN 3343-3)

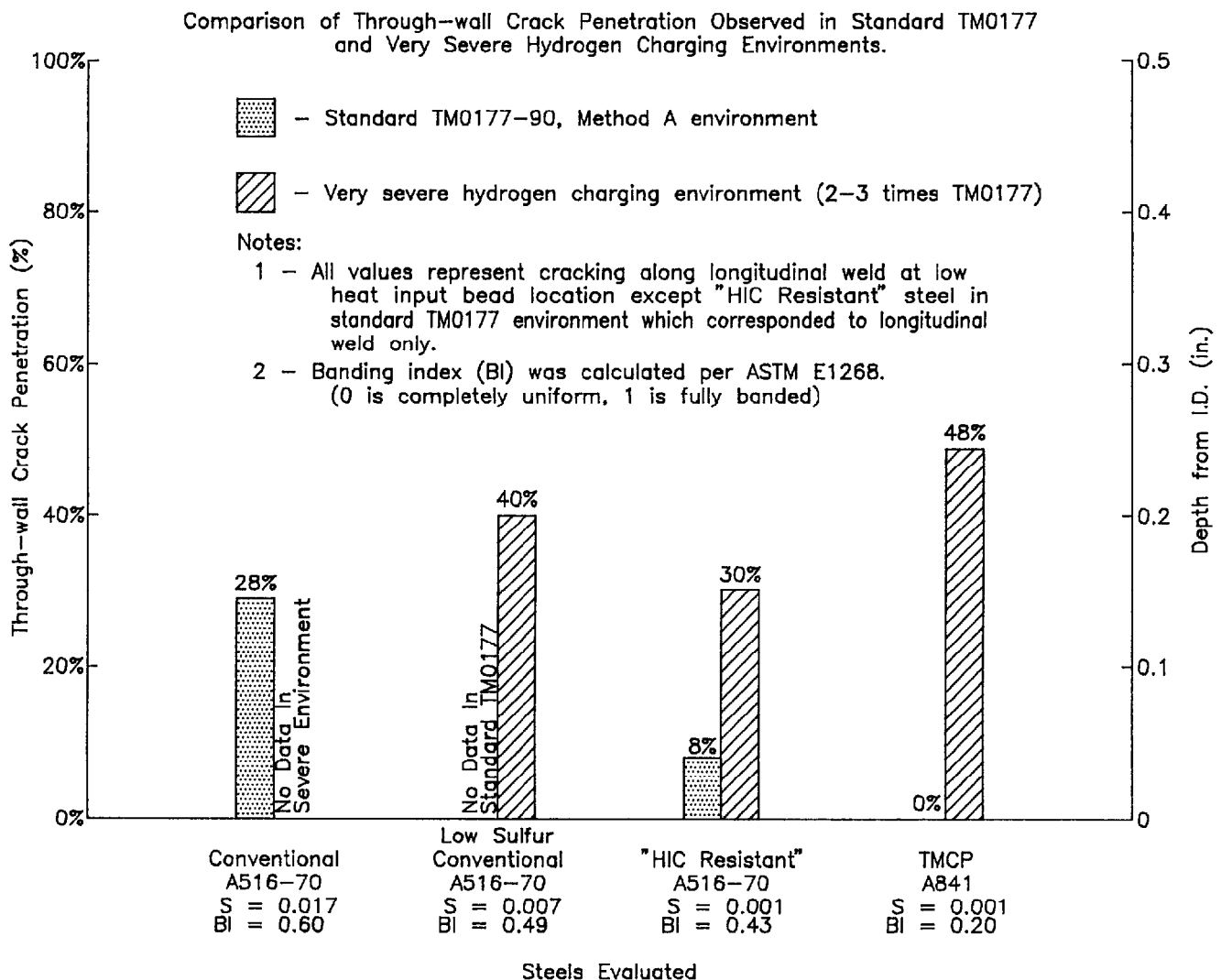


Fig. 4-9—Comparison of through-wall crack penetration observed in standard NACE TM0177 and very severe hydrogen charging environments

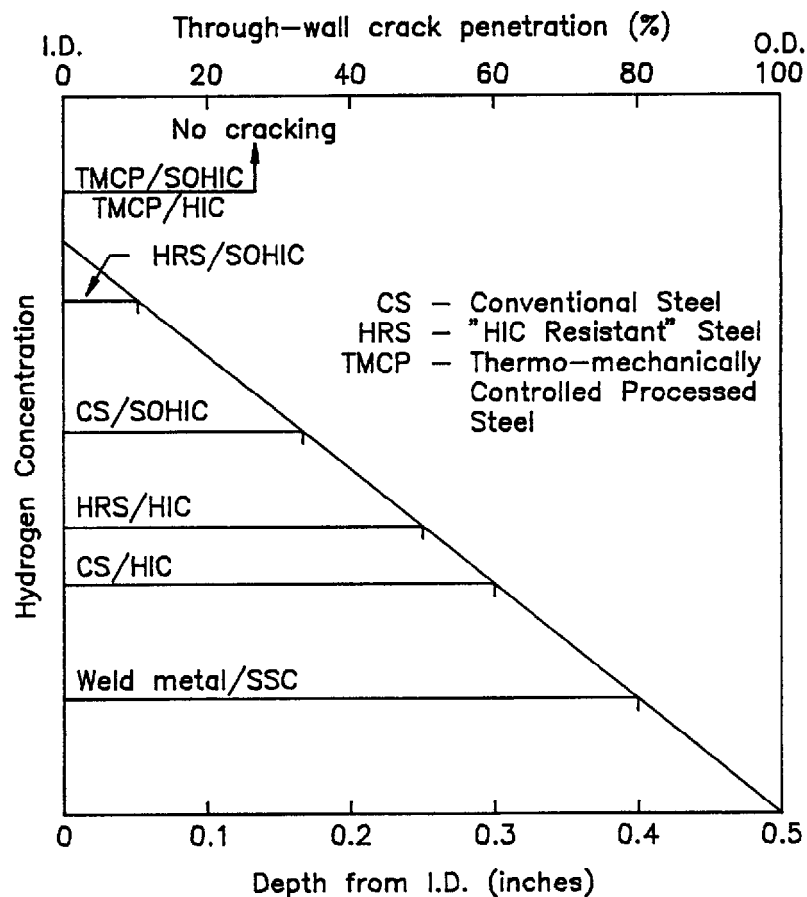


Fig. 4-10—Critical hydrogen concentrations required for cracking in the steels evaluated

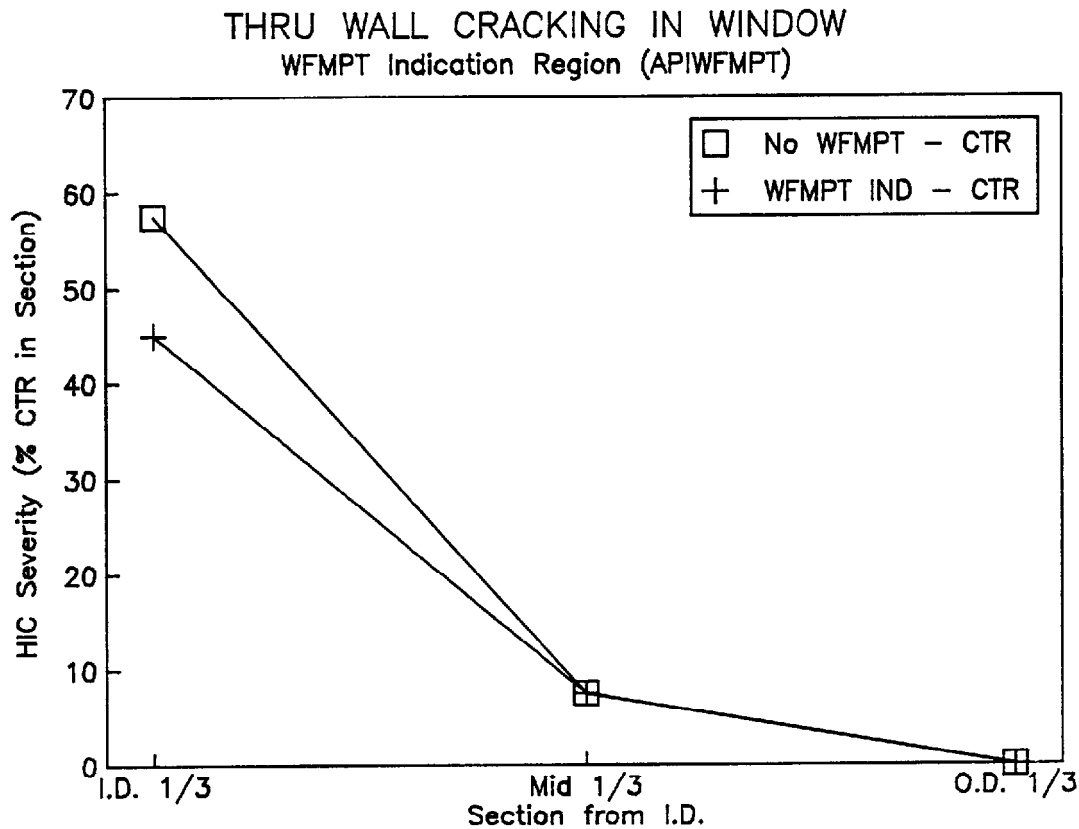
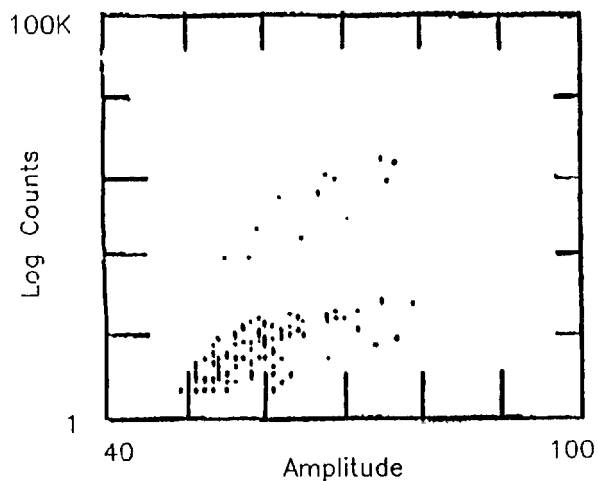
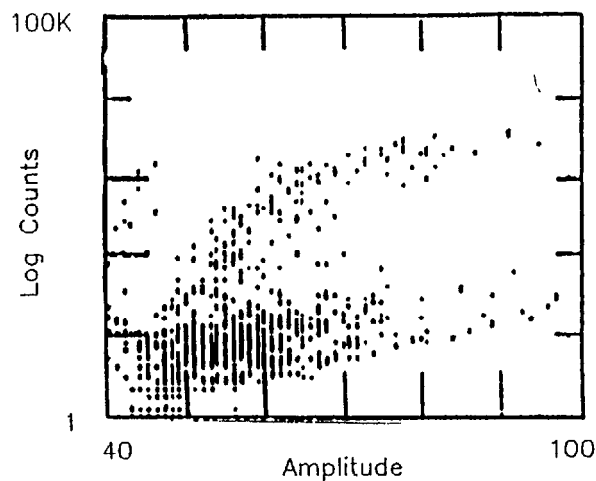


Fig. 4-11—Sectioning results based on positive WFMP regions and regions of no WFMP indications



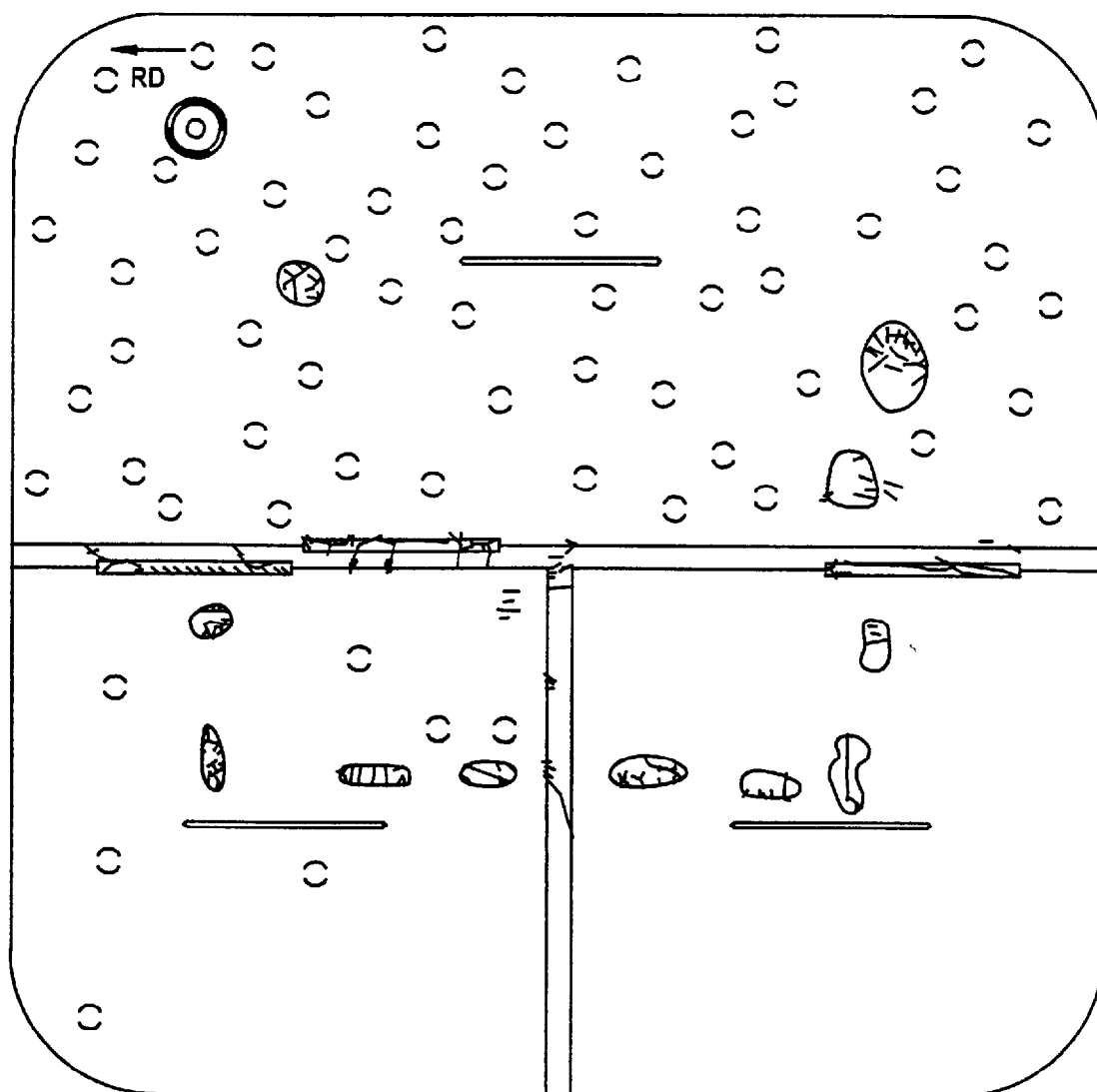
Stage 2 AE Results



Stage 8 AE Results

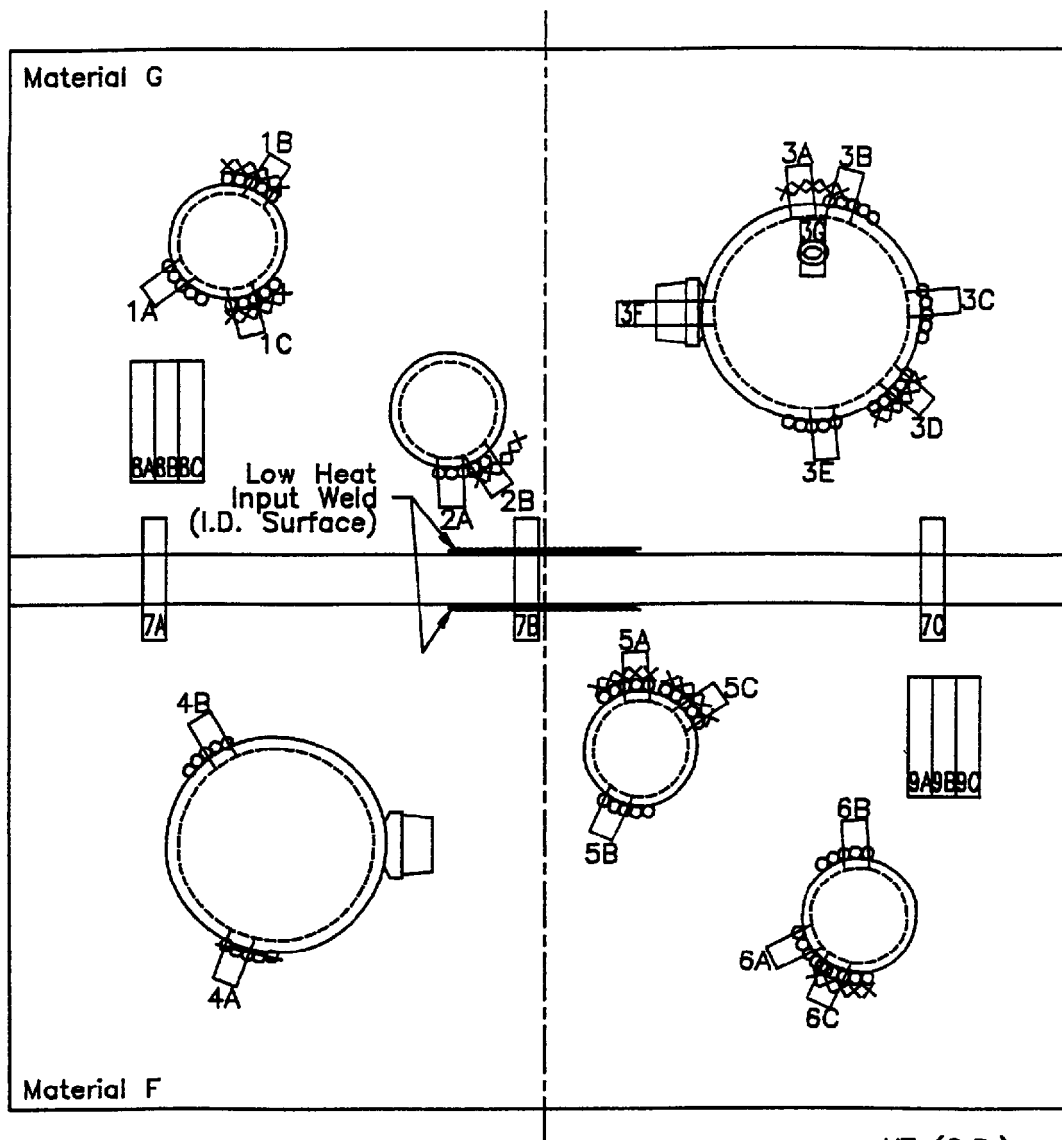
Note: High level of AE response after cleaning and solution renewal. Upper band AE response was characteristic of through-wall cracking.

Fig. 4-12—AE counts versus amplitude data taken during stages 2 and 8 of the environmental staging/effect of cleaning evaluation



O.D. view for comparison to AUT data. All WFMT cracking is on I.D. surface.

Fig. 4-13—O.D. view of the WFMT conducted on the I.D. of the environmental staging/effect of cleaning evaluation



Material F: A841

CLI Number: 3250

Chemistry: C=0.09, P=0.003, S=0.001

Material Condition: TMCP

Mechanical Properties: Yield=67.0ksi, UTS=79.0ksi, % elongation=25.0

Material G: A516 Grade 70

CLI Number: 2280

Chemistry: C=0.22, P=0.027, S=0.020

Material Condition: Normalized

Mechanical Properties: Yield=52.0ksi, UTS=76.9ksi, % elongation=42.0

○ UT (O.D.)

× WFMPPT (I.D.)

Fig. 4-14—Crack locations based on WFMPPT and manual UT on the evaluation of nozzle attachments/PWHT



Fig. 4-15—AUT T-scan data taken at the conclusion of the simulation of thick plate behavior evaluation. For full-color version, see insert following p. 14

THRU WALL CRACKING IN WINDOW Long vs. Girth Welds (APIPOST3)

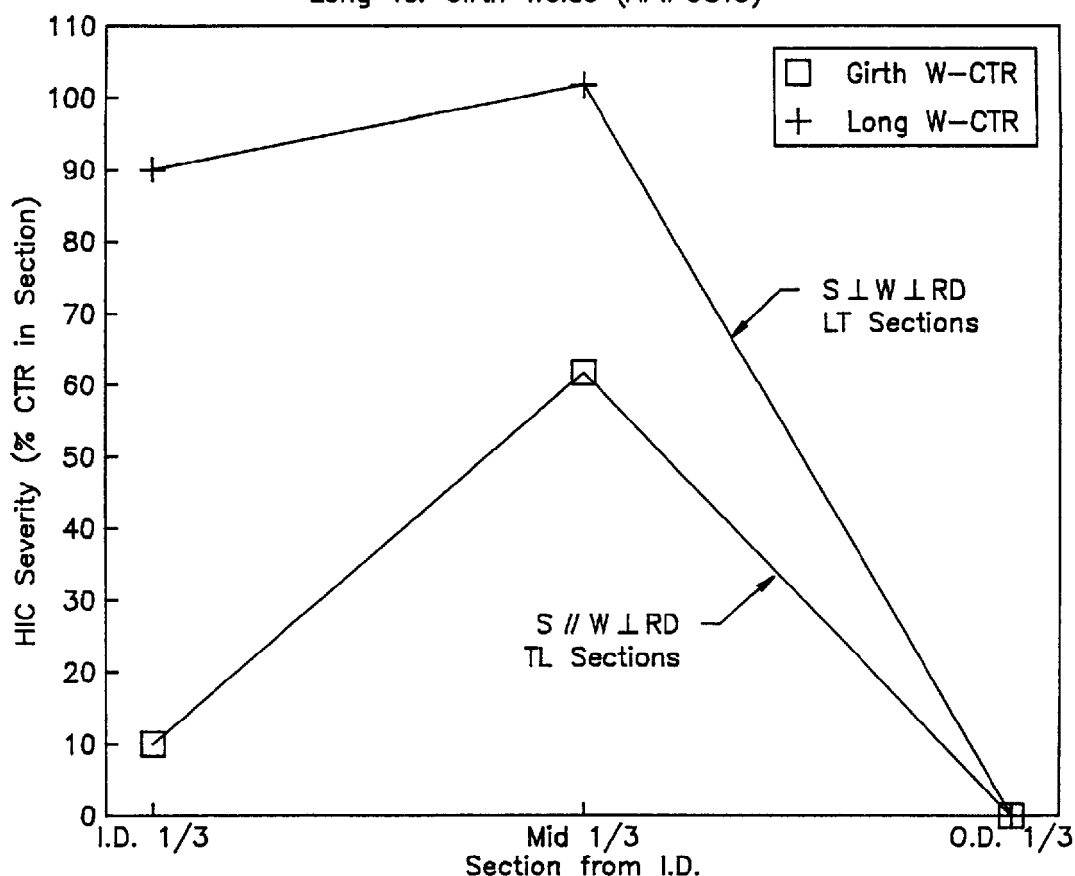


Fig. 4-16—Increased severity of SOHIC as characterized by increased CTR values associated with LT sections

conditions, was 50% of the wall thickness in the “HIC Resistant” steel and 30% of the wall thickness in the low sulfur conventional steel. These cracks gave similar cracking indications with the amplitude based AUT (P-scan) system. The AUT P-scan system is able to detect and determine the extent of through-wall cracking using automated time of flight diffraction (TOFD) techniques. However, these techniques become limited when applied to thin shells such as those used in this research program.

Manual UT relative arrival time techniques proved to be reliable for crack sizing in the simple plate specimens used in this program. Manual UT techniques were used to determine both the lateral and through-wall extent of crack propagation in the hard weld study. Metallographic sectioning showed good agreement with the results of manual UT sizing results (See Appendix II). Additionally, the depth of HIC in the plate, as determined by manual UT, was also found to agree very closely with the value found by metallographic sectioning.

The only cases where manual UT crack sizing was not found to be accurate were in determinations made in the attachment areas of the nozzles. In this case, many crack indications were found in these areas using manual UT (See Fig. 4-14). However, when these areas were metallographically sectioned, no

cracks were identified which corresponded to the indicated crack sites (See Appendix VI for both manual UT report and metallographic crack measurement data).

4.3.4. AUT longitudinal wave (T-scan) techniques were able to locate regions of internal planar HIC cracking. However, extensive HIC which produced overlapping cracks led to oversizing of the HIC indications based on AUT T-scan alone. Additionally, metallographic sectioning alone could produce non-conservative results due to chance sectioning. The best combination for quantitative HIC sizing was found to be destructive sectioning based on AUT T-scan measurements.

The results of comparative AUT (T-scan) and metallographic sectioning are shown in Fig. 4-15 and in Appendix IV, respectively. The AUT data indicated distinct areas of HIC within the plate specimen and an indication of the HIC depth within the plate. However, when comparing the crack sizes from the AUT scan and the metallographic sections from similar sections, it was found that the overlapping areas of HIC made the HIC regions appear larger on the AUT scan than was found with metallography.

Similarly, due to the isolated nature of HIC in some areas in the plate, it appeared possible to grossly under-estimate the degree of HIC if the metallo-

graphic sections were made by chance sectioning through regions of low HIC density. The best assessment of HIC severity was found when AUT was used as the basis for selecting sites for metallographic sections. In this manner, appropriate sites could be selected for sectioning. In these cases, the crack measurements made from the AUT scans were in close agreement with the crack measurements made on the metallographic sections.

4.3.5. Metallographic evaluations in conventional steels conducted on LT sections resulted in more severe SOHIC indications than for similar evaluations made on TL sections.

Fig. 4-16 shows increased severity of SOHIC as characterized by increased CTR values associated with LT sections. This effect appears to be related to the orientation of the weld, maximum tensile stress and rolling direction of the steel. When the maximum tensile stress direction is perpendicular to the welding direction and the rolling direction, CTR values tend to be maximized. The cracking was limited to the inner $\frac{2}{3}$ region (i.e., I.D. $\frac{1}{3}$ and middle $\frac{1}{3}$ regions) of the plate thickness. The values of CTR in this region are consistently greater than those found for welds where the welding direction is perpendicular to the rolling direction and parallel to the direction of maximum tensile stress as was the case for the girth weld in the window specimen.

4.3.6. Acoustic Emission (AE) monitoring showed separate and identifiable acoustic responses for through-wall (i.e., SOHIC and SSC) and in-plane cracking (i.e., HIC). On-line AE measurements were able to detect the onset of HIC and SOHIC and could be used to provide an indication of the activity of each type of cracking.

As described in Section 4.3.2, AE monitoring was able to provide a direct indication of the extent of active cracking resulting from planar HIC cracking and through-wall SSC and SOHIC. Fig. 4-12 showed the increase in SOHIC activity, demonstrated by an increased amount of upper band activity observed in the AE correlation plot of log counts versus amplitude. By comparison, planar HIC cracking was found to be associated with the lower band on this AE correlation plot.

Additional evidence of the upper/lower band relationship is shown in Fig. 4-17. This figure illustrates the variation of internal test pressure, peak AE amplitude and average amplitude plotted as a function of test duration. The peak amplitude appeared to vary with the internal pressure in the vessel. Without internal pressure, cracking in the test window was found to be primarily of a planar nature resulting from HIC. With the application of the pressure stress in the test window a significant increase in CTR and through-wall oriented crack growth was observed (See Fig. 4-18). Since the peak amplitude was typically associated with the upper band on the AE correlation plot, it was suggested that the upper AE band was most likely related to the formation of through-thickness oriented cracks (i.e., SSC and SOHIC).

The average AE amplitude appears to be relatively constant throughout the test period in Fig. 4-17. The AUT data from this same test showed that the increase in HIC cracking also increased in a relatively constant manner during this period. Therefore, since the average amplitude is most heavily dependent on the lower AE band of the correlation plot, it was suggested that the lower band was most indicative of

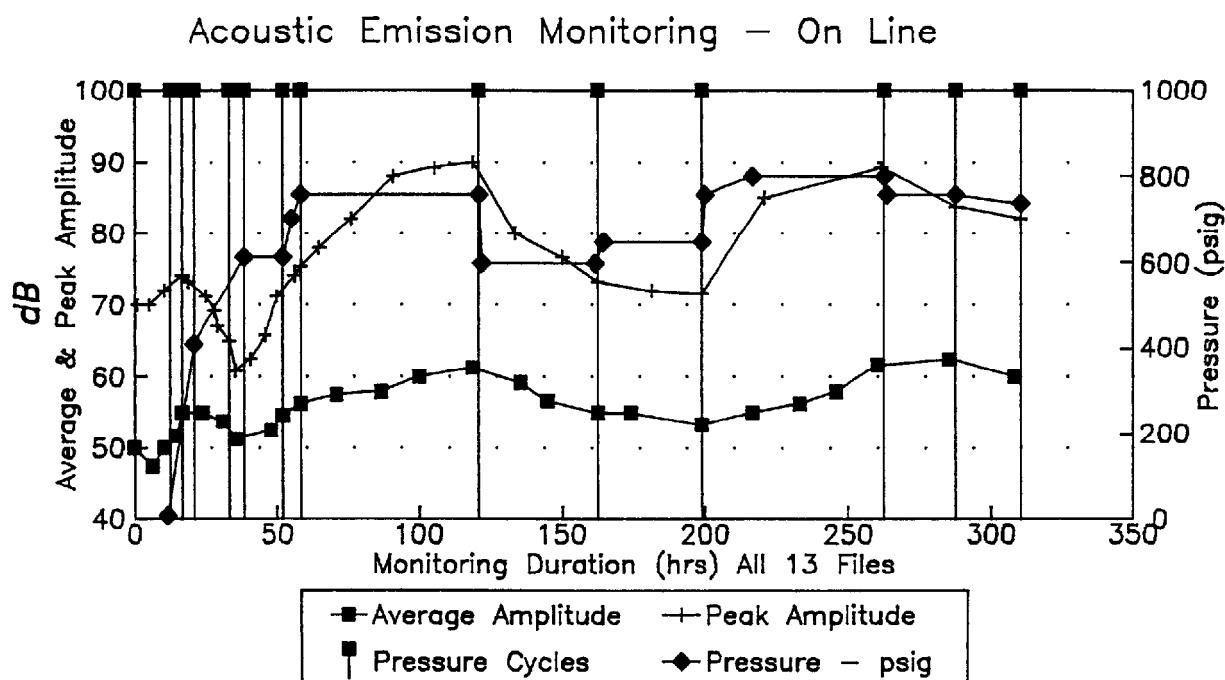


Fig. 4-17—Variation of internal test pressure, peak AE amplitude and average amplitude

HIC / SOHIC of Window with Exposure Time

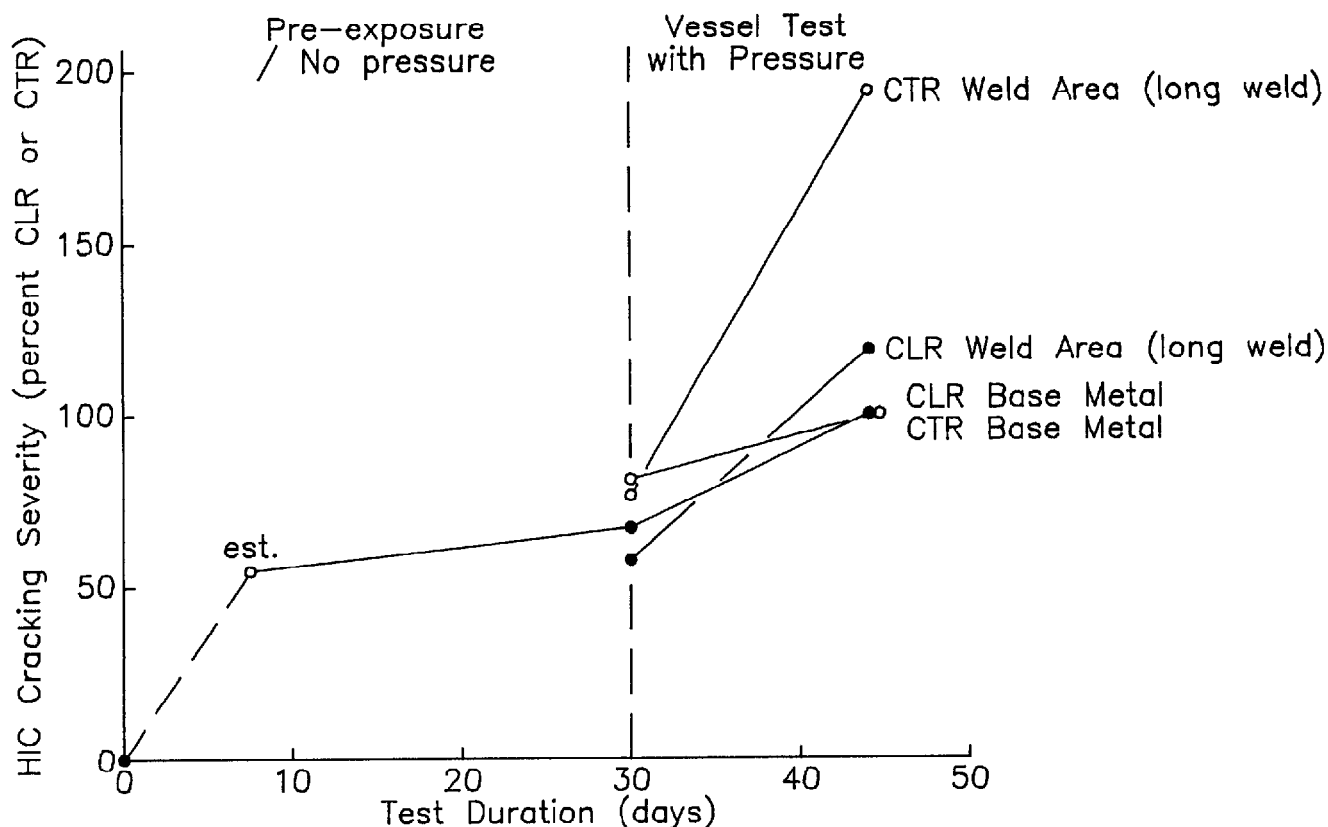


Fig. 4-18—Increase in CTR values after subjecting material to a pressure stress in the test vessel

the planar HIC cracking taking place in the test window. Observations made on subsequent test windows such as those shown in Fig. 4-18 appeared to confirm these early findings.

4.3.7. Based on AE monitoring and metallographic sectioning, the rate of HIC was not dependent on changes in the internal pressure of the test vessel. By comparison, SOHIC was found to be directly related to the internal pressure (i.e., increased SOHIC with increasing pressure), presence of low heat input welds, and severity of hydrogen charging.

Figs. 4-12 and 4-18 indicate that HIC in susceptible steels can occur under conditions with no stress and increase only slightly for conditions of increased operating pressure up to stress levels beyond normal operating limits for such equipment. However, severity of SOHIC depends on the operating stress levels. The results of AE monitoring suggest that SOHIC increases with operating pressure. Operating situations which increased tendencies for SOHIC were: (1) high operating pressures, (2) presence of non-stress relieved, low heat input welds (with high hardness and local residual tensile stress), (3) lack of PWHT, and (4) hydrogen charging severity of the environment. The results shown in Appendix V and VI support these findings.

4.4 Vessel Design and Integrity

4.4.1. The residual strengths of tensile specimens taken from the conventional steel plate containing substantial HIC damage were 95 to >100% of the 70 ksi specified minimum tensile strength for ASTM A516-70. Measured elongations, however, ranged from 40–85% of specified minimum values. Specimens removed across the welds exhibited greater degradation in mechanical strength than base metal specimens. Maximum degradation was found to correlate with the regions of maximum SOHIC extension. These areas were in the HAZ of the welds which were perpendicular to the hoop stress in the vessel.

Following one-side exposure to NACE TM0177 solution under internal pressure, samples were cut from a test window to allow for determination of the residual mechanical properties of the A516-70 steel plate material. The samples were stored for several weeks at room temperature to allow for loss of diffusible hydrogen from the specimens. Fig. 4-19 shows the actual tensile strengths and elongations for specimens taken in the orientations indicated with the following codes:

1. 516 Specified Minimum Values per ASTM A516-70
2. PRE Pre-Test Values for Actual Plate Material

EFFECT OF HIC ON 516-70 PROPERTIES

Conventional Steel (APIPROP)

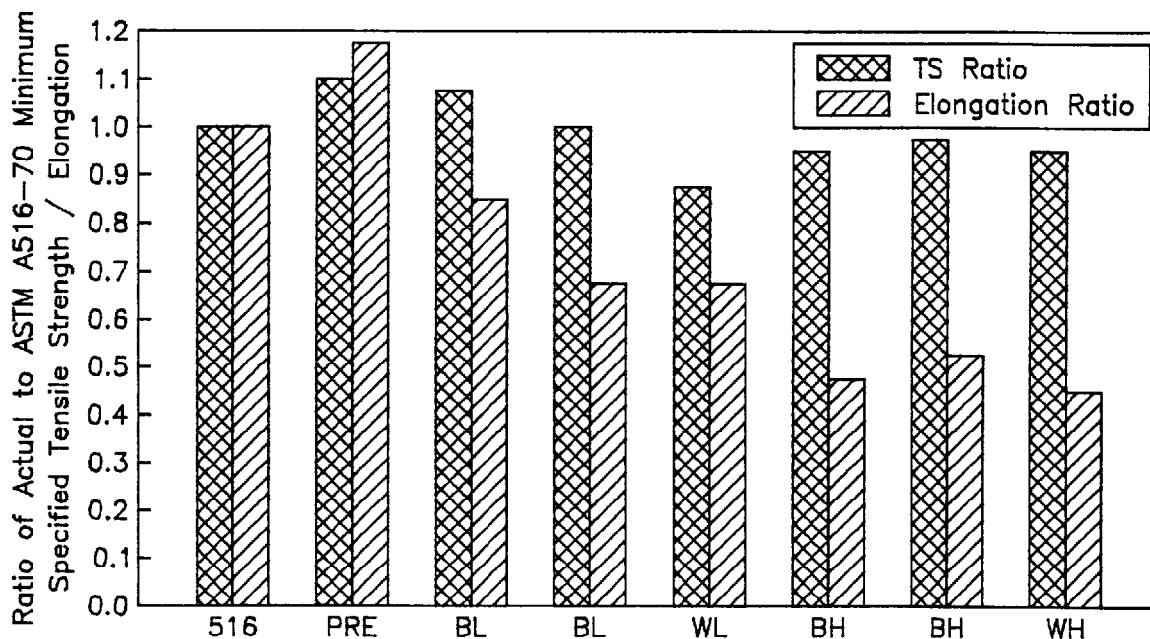


Fig. 4-19—Effect of HIC/SOHIC on mechanical properties of ASTM A516-70

3. BL Base Metal—Longitudinal Direction
4. WL Weldment—Longitudinal Direction
5. BH Base Metal—Hoop Direction
6. WH Weldment—Hoop Direction

As can be seen in this figure, the residual strengths of the specimens tested were all greater than 85% of the 70 ksi minimum specified tensile strength for A516-70. No relationship was observed between the orientation or location from which the test specimen was taken and the residual tensile strength after exposure in the pressure vessel configuration. The elongations of the specimens taken from the exposed material, however, were all less than the minimum specified values for A516-70. Maximum reductions in elongation were observed on tensile specimens oriented in the circumferential direction, particularly those taken across the longitudinal weld. Based on evaluation of the tensile specimens after testing, the severe reduction in elongation was attributed to the presence of SOHIC in the base metal HAZ adjacent to the longitudinal weld.

Base metal Charpy impact specimens were also taken from the base plate in both the longitudinal and transverse directions. Average longitudinal and transverse Charpy impact energy values following exposure were 60 and 34 ft-lb, respectively, at room temperature. The transverse values were essentially the same as those obtained for the A516-70 plate prior to exposure whose average value was 36 ft-lb. It should be cautioned that these values of strength, elongation and Charpy energy are residual values *without* hydrogen; values in the presence of hydrogen could be lower depending on the conditions of exposure and testing.

4.4.2. HIC in the conventional steels tested in NACE TM0177 solution was confined to the $\frac{2}{3}$ thickness (from the I.D.). HIC in the “HIC Resistant” steels tested was limited to the I.D. 20% of the wall thickness except for isolated small regions of centerline cracking. HIC did not occur in A841 base metal in NACE TM0177 tests or in very severe accelerated tests using cathodic charging in NACE TM0177. When HIC reached these depths, it was not possible to obtain further HIC.

Figs. 4-9 and 4-10 indicate typical results with regard to depth of HIC for conventional, “HIC Resistant” A516-70 and A841 steels tested in this program. These data suggest that the critical hydrogen concentration for HIC increases with decreasing sulfur content in the steel and with the application of modern steelmaking and processing techniques. However, if problems such as centerline segregation exist they can limit the overall HIC performance of the steels in wet H_2S service.

Efforts were made to continue HIC beyond the depths indicated above in the experiments conducted in this program using prolonged exposures and external hydrogen barriers and external charging. None of these methods proved to be significant in terms of producing an increase in HIC size or depth of cracking.

4.4.3. SOHIC in the “HIC-Resistant” (A516-70) and ultra-low sulfur advanced steels (A841) tested in the standard NACE TM0177 solution showed lower susceptibility to through-wall cracking by SOHIC than conventional steels based on absolute crack depths. SOHIC was produced to a maximum of

approximately 8% of the wall thickness in these steels.

As previously shown in Fig. 4-9, the results for through-wall SOHIC in the various steels tested in this program indicate that in NACE TM0177 solution, SOHIC was produced in the conventional steel to a depth of approximately 30% of the wall thickness, while the "HIC Resistant" steel exhibited SOHIC to a depth of approximately 10%. No SOHIC was produced in the A841 steel exposed to the NACE TM0177 solution in this study. However, in accelerated charging tests which had two to three times the hydrogen charging levels of NACE TM0177, the TMCP A841 steel did exhibit SOHIC to a depth of approximately 50% of the wall thickness in regions around weldments.

The data presented above and in Sections 4.1.2 and 4.2.6 suggest that decreased levels of sulfur content and decreased microstructural banding reduce the susceptibility to HIC and SOHIC. However, in very severe hydrogen charging environments these same steels, irrespective of sulfur content or degree of banding, may be susceptible to SOHIC. Therefore, to optimize cracking resistance in wet H₂S refinery equipment, it appears that issues related to all wet H₂S cracking mechanisms (i.e., HIC, SOHIC and SSC) must be addressed and the limitations of the specific carbon steels identified. Alternative methods such as the use of stainless steel clad vessels may be needed for very severe environments.

4.4.4. Based on AE monitoring and metallographic sectioning, the rate of HIC was not dependent on changes in the internal pressure of the test vessel. By comparison, SOHIC was found to be directly related to the internal pressure (i.e., increased SOHIC with increasing pressure), the presence of low heat input welds and severity of hydrogen charging.

Figs. 4-12 and 4-18 indicate that HIC in susceptible steels can occur under conditions of no stress, and increase only slightly for conditions of increased operating pressure up to stress levels beyond normal operating limits for such equipment. However, severity of SOHIC depends on the operating stress levels. The results of AE monitoring suggest that SOHIC increases with operating pressure. Operating situations which increased tendencies for SOHIC were: (1) high operating pressures, (2) presence of non-stress relieved, low heat input welds (with high hardness and local residual tensile stress), (3) lack of PWHT, and (4) hydrogen charging severity of the environment. The results shown in Appendix V and VI support these findings.

4.4.5. For conventional and "HIC Resistant" steels, the threshold hydrogen flux for HIC does not vary with sulfur content in the steel or susceptibility to HIC. The main difference between HIC susceptible steels and more resistant steels is the number of HIC initiation sites and the extent of HIC.

The results shown in Figs. 4-1a and 4-1b and Appendix V indicate that conventional and "HIC-

Resistant" A516-70 steels initiated HIC at the same level of hydrogen charging during the sequence of increased hydrogen charging severity. This suggests that differences in the HIC performance of the steels is not related to the increase in resistance of individual sites to HIC initiation in the steels but to (1) reducing the number of HIC initiation sites and (2) the extent of HIC propagation. In the present study, the only material not found to have a HIC threshold hydrogen flux (or concentration) was the A841 steel which was not susceptible to HIC even in the most severe hydrogen charging environment.

4.4.6. PWHT was found to be effective in reducing the SOHIC in the area of the longitudinal seam weld which also contained low heat input welds. However, no effect of PWHT was found for SOHIC of nozzle attachment weldments.

The benefit of PWHT was observed in this study when comparing the results of SOHIC in the longitudinal seam welds containing low heat input welds with and without PWHT in NACE TM0177 solution (See Appendix IV and VI). In this case, SOHIC was observed in non-PWHT longitudinal seam welds containing low heat input welds. SOHIC was located in the HAZ region of the base metal (Appendix IV). Appendix VI shows the results of testing similar longitudinal seam welds containing low heat input welds which received PWHT prior to testing. In this case, no SOHIC was observed to occur in either conventional steel or ultra-low sulfur advanced steel.

As shown in Appendix VI, SOHIC was not observed in the nozzle attachment welds in either conventional (A516-70) or ultra-low sulfur advanced steels (A841). Neither of these attachment welds experienced SOHIC in this investigation in both the PWHT and no PWHT conditions tested in NACE TM0177 solution.

The differences in the behavior of the longitudinal seam welds containing low heat input welds and the nozzle attachment welds highlight the apparent importance of the low heat input welds in the initiation of SOHIC. The combination of the high local hardness and residual stress associated with the low heat input welds provides an excellent initiator or promoter for the initiation of SOHIC. Once these are reduced or eliminated through PWHT, the tendency for the initiation of SOHIC is significantly lessened. These results suggest that if the effect of low heat input welds would be superimposed on the nozzle attachment welds a similar effect of PWHT would be exhibited. The results from this study may indicate why the effect of PWHT on SOHIC has not always been found in wet H₂S refinery experience.

4.4.7. The observations of material behavior found in this study were consistent with the findings of the previously conducted laboratory testing conducted in the MPC Wet H₂S Research Program. The results of the present study validated the wet H₂S cracking test methods developed previously and showed their applicability to refinery wet H₂S environments.

Prior to the initiation of this program, MPC orga-

nized a research program on wet H₂S cracking of steels sponsored by more than twenty major petroleum companies, steel manufacturers and equipment fabricators. The data were collected using standard small scale laboratory tests such as the NACE TM0177 tensile test, the NACE TM0284 HIC test and the double beam test developed during the MPC program.⁸ The data provided valuable fundamental information which has improved both the awareness of the causes of wet H₂S cracking and potential remedies in terms of both new construction and repair of existing equipment. However, there was a desire to validate the findings and conclusions of that program and to explore the complex interrelations of variables that can affect the actual behavior of large scale equipment used in wet H₂S service. The present study, which characterized the full-scale behavior of refinery equipment in wet H₂S service, assisted in bridging this gap. In general, the observations of material behavior found in this study were consistent with the findings of the previous laboratory testing conducted

in the MPC Wet H₂S Research Program. The results of the present study validated the wet H₂S test methods developed previously and showed their applicability to refinery wet H₂S environments.

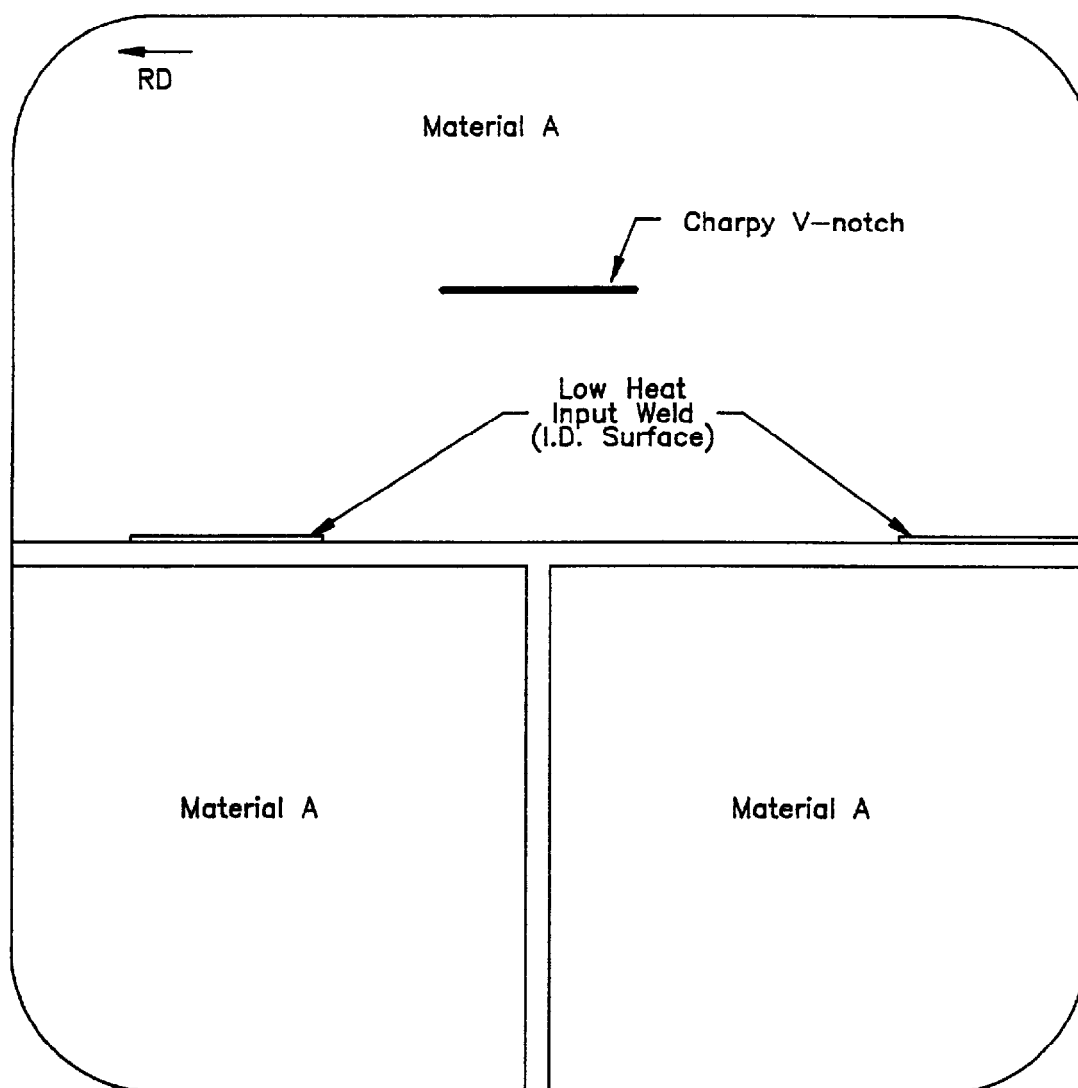
5.0 References

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APPENDIX I

SERVICEABILITY OF HIC DAMAGED STEEL

APPENDIX I - FIGURE 1



Material A: A516 Grade 70
 CLI Number: 2278
 Chemistry: C=0.22, P=0.020, S=0.017
 Material Condition: As Rolled
 Mechanical Properties: Yield=48.2ksi, UTS=78.9ksi, % elongation=25.0

O.D. View

Date: 9/2/93	Project #: L902103KT
Diek #: L500-1	File #: 2209
Scale:	Drawn By: K. Koenig