

HYDROGEN INDUCED CRACKING BEHAVIOR OF HSLA API X65 CARBON STEEL

BY

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MASTER OF SCIENCE

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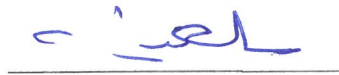
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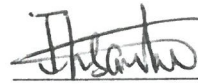


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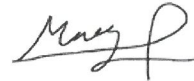
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|Dedication to my parents, my brothers, my sisters and my family |

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LIST OF ABBREVIATIONS

API:	American Petroleum Institute
HIC:	Hydrogen Induced Cracking
SOHIC:	Stress Oriented Hydrogen Induced Cracking
SSC:	Sulfide Stress Cracking
MAOP:	Maximum Allowable Operating Pressure
MAWP:	Maximum Allowable Working Pressure

|

ABSTRACT

Full Name : [Eissa S. Al-Zahrani]
Thesis Title : [Hydrogen Induced Cracking Behavior Of HSLA API X65 Carbon Steel]
Major Field : [Materials Science and Engineering]
Date of Degree : [December and 2023]

Hydrogen Induced Cracking (HIC) is one of the predominant damage mechanisms that affect the integrity of pipelines that transport oil and gas products. The current industrial practice is to utilize HIC resistance materials as a first line of defense. However, HIC resistance materials are not fully immune of Hydrogen Induced Cracking. Accordingly, National Association of Corrosion Engineers (NACE) has established a test method (NACE Test Method 0284) to evaluate the HIC susceptibility. This test method is performed on the raw materials which are plates or coils and as a pipeline. In some circumstances, the pipe fails the HIC testing whereas the plates or coils pass the HIC testing. This research focuses on investigating the HIC behavior of HIC resistant LSAW API X65 pipeline steel samples. The research was conducted on two main parts which include NACE Test Method 0284 in as welded and heat treated conditions and Electrochemical Hydrogen Charging, which shall demonstrate the effect of hydrogen charging on the mechanical properties of the piping materials. Then, the grain size and inclusion contents were measured to investigate the impact of heat treatment and raw material processing. The fracture surfaces were evaluated using Scanning Electron Microscopy and Energy Dispersive X-Ray Analysis. The HIC testing results indicate that the heat treated specimens passed the HIC testing while the non-heat treated HIC samples encountered failures in HIC testing. It can be highlighted that the failure of HIC testing is always encountered in the welded area which referenced here as zero degree. The HIC

failure encountered in base metal adjacent to the weld and Heat affected zones areas due to the presence of inclusions in the metal and micro-residual stresses developed during pipe forming and welding processes. All failures in HIC were attributed to globular inclusions resulted from Ca treatment conducted during casting process of steel plates. There was no presence of elongated manganese sulfide inclusions which are known as hydrogen trap site and HIC crack initiation. The research revealed that Ca treatment for thermos-mechanical plates with ultra-low sulfur content is responsible for development of globular inclusion, mainly CaO, resulting in HIC failure subsequent to pipe manufacturing processes. The electrochemical hydrogen charging experiment on tensile specimens demonstrated that the sample exposure to hydrogen embrittlement that altered the mechanical properties. Both electrochemical hydrogen charging experiment and NACE TM 0284 illustrated identical tensile testing results.

ملخص الرسالة

الاسم الكامل: عيسى صالح عيدان الزهراني

عنوان الرسالة: سلوك التكسير المستحث بالهيدروجين للفولاذ الكربوني عالي القوة ومنخفض السبانك API X65

التخصص: هندسة المواد

تاريخ الدرجة العلمية: ديسمبر 2023

التكسير الناجم عن الهيدروجين هو أحد آليات الضرر السائدة التي تؤثر على سلامة خطوط الأنابيب التي تنقل منتجات النفط والغاز. الممارسة الصناعية الحالية هي استخدام مواد مقاومة هيكل كخط دفاع أول. ومع ذلك ، مواد المقاومة هيكل ليست محصنة تماما من الهيدروجين الناجم عن تكسير. وبناء على ذلك ، أنشأت الرابطة الوطنية لمهندسي التآكل (نيس) طريقة اختبار (طريقة اختبار نيس 0284) لتقييم حساسية هيكل. يتم تنفيذ طريقة الاختبار هذه على المواد الخام التي هي لوحات أو لفائف وخط أنابيب. في بعض الحالات ، فشل الأنابيب اختبار هيكل في حين أن لوحات أو لفائف تمرير اختبار هيكل. ويركز هذا البحث على التحقيق في سلوك هيكل من هيكل مقاومة لساو أبي 65 عينات أنابيب الصلب. تم إجراء البحث على جزأين رئيسيين يشملان طريقة اختبار نيس 0284 في الظروف الملحومة والمعالجة بالحرارة وشحن الهيدروجين الكهروكيميائي ، والذي يجب أن يوضح تأثير شحن الهيدروجين على الخواص الميكانيكية لمواد الأنابيب. بعد ذلك ، تم قياس حجم الحبوب ومحتويات التضمين للتحقيق في تأثير المعالجة الحرارية ومعالجة المواد الخام. تم تقييم أسطح الكسر باستخدام الفحص المجهر الإلكتروني وتحليل الأشعة السينية المشتتة للطاقة. تشير نتائج اختبار هيكل إلى أن العينات المعالجة حراريا اجتازت اختبار هيكل بينما واجهت عينات هيكل غير المعالجة حراريا إخفاقات في اختبار هيكل. ويمكن تسليط الضوء على أن فشل اختبار هيكل واجه دائما في المنطقة الملحومة التي المشار إليها هنا كما درجة الصفر. فشل هيكل واجه في المعادن الأساسية المجاورة للحام والحرارة المناطق المتضررة المناطق بسبب وجود شوائب في المعادن والضغوط المتبقية الصغيرة وضعت خلال تشكيل الأنابيب وعمليات اللحام. ونسبت جميع حالات الفشل في هيكل إلى شوائب كروية أسفرت عن شكل العلاج كما أجريت أثناء عملية الصب من ألواح الصلب. لم يكن

هناك وجود لشوائب كبريتيد المنغنيز الممدودة والتي تعرف باسم موقع مصيدة الهيدروجين وبدء الكراك. وكشف البحث أن العلاج كا لوحات الترمس الميكانيكية مع محتوى الكبريت منخفضة جدا هي المسؤولة عن تطوير إدراج كروي ، أساسا تساو ، مما أدى إلى فشل هيكل اللاحقة لعمليات تصنيع الأنابيب. أظهرت تجربة شحن الهيدروجين الكهروكيميائية على عينات الشد أن تعرض العينة لتقصف الهيدروجين الذي غير الخواص الميكانيكية. كل من الكهروكيميائية الهيدروجين شحن التجربة و نيس تم 0284 يتضح نتائج اختبار الشد متطابقة.

CHAPTER 1

INTRODUCTION

1.1 Background

Cracking mechanisms associated with oil and gas containing wet H₂S are the most frequently encountered failures in carbon steel pipelines [1–4]. In principle, sour crude and gas corrode the internal surface of the metallic components and produce hydrogen atoms that penetrate the steel surface and become intercepted in either reversible (grain boundaries and dislocations) or irreversible (nonmetallic inclusions and precipitate/interfaces) trap sites, Figure 1. Accordingly, the hydrogen atoms, once trapped, generally try to create hydrogen molecules (H₂), which develop huge internal pressure and consequently bulging of the steel surface, referred to as hydrogen blister formation in steels [5].

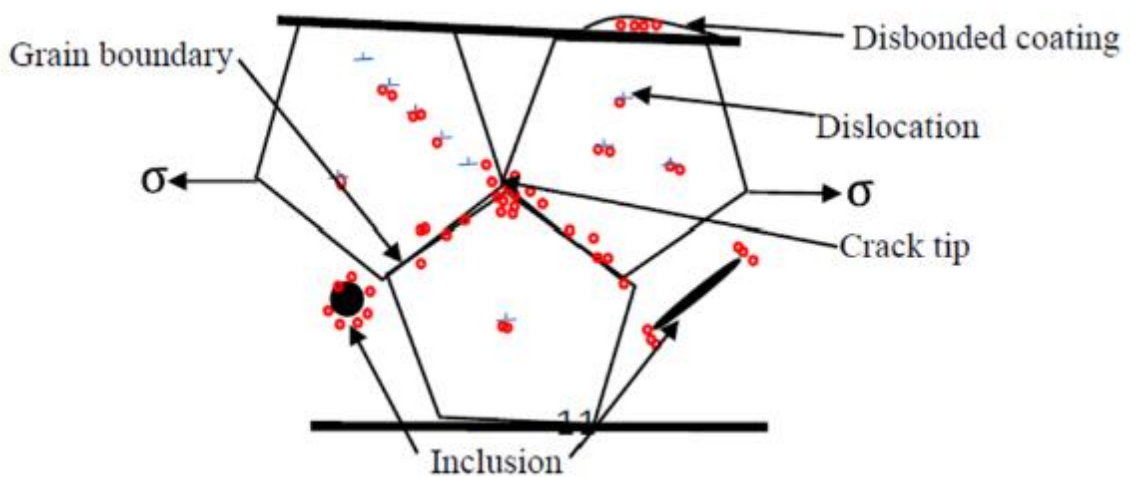


Figure 1: Schematic shows typical hydrogen trapping sites in the steel matrix [5].

The sour environment is defined as follows:

1. Free water with more than 50 ppm H_2S , or
2. Free water with pH less than 4 and presence of H_2S , or
3. Free water with pH more than 7.6 and 20 ppm dissolved hydrogen cyanide and presence of H_2S , or
4. In the gas phase, the partial pressure of H_2S exceeds 0.0003 Mpa (0.05 psia).

The metallic components, including pipelines and pressure vessels, operate in sour service generally experience so-called wet H_2S damage, which is classified into four main forms, i.e., sulfide stress cracking (SSC), stress-oriented hydrogen-induced cracking (SOHIC), hydrogen blistering, and hydrogen-induced cracking (HIC). HIC can be found at different locations, such as internal and outer surfaces, in the center of the steel thickness, and near weld areas. This type of cracking is encountered in a place that is parallel to the rolling direction, resulting in stepwise cracking that does not require the presence of external as well as residual stresses, Figure 2 [6].

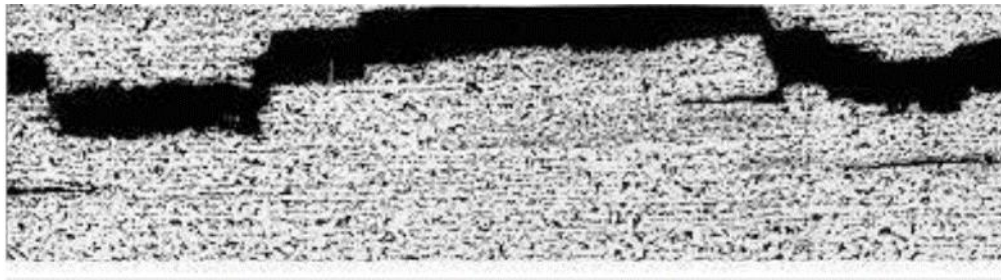
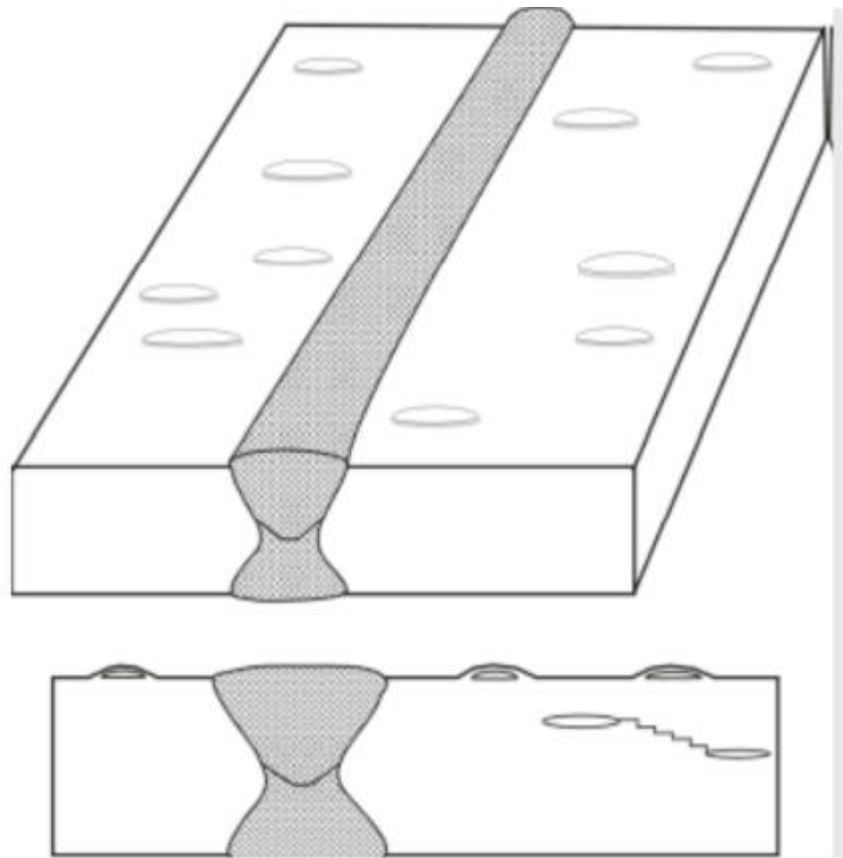


Figure 2: Typical morphology of Hydrogen Blister and HIC [6].

SOHIC is a special form of wet H₂S damage, which mainly occurs close to the weld seam because the residual stresses from welding and forming are consistently high in this area. SOHIC failure can develop in both HIC-susceptible and HIC-resistant steels, and it is observed like staggered small cracks where it is indicated approximately perpendicular to the principal stress, either residual or applied. This results in a array just like a ladder-like

crack. It always links pre-existing HIC cracks and follows the stress direction, Figure 3. The weld fusion line is the highest stress location where hydrogen atoms accumulate in this region even though there is no nonmetallic inclusion or clusters. Ladder-like cracks of HIC can be observed in this location and might lead to through-thickness cracking. These cracks of SOHIC are always located relatively perpendicular to the applied or residual tensile stress axis [6–10].

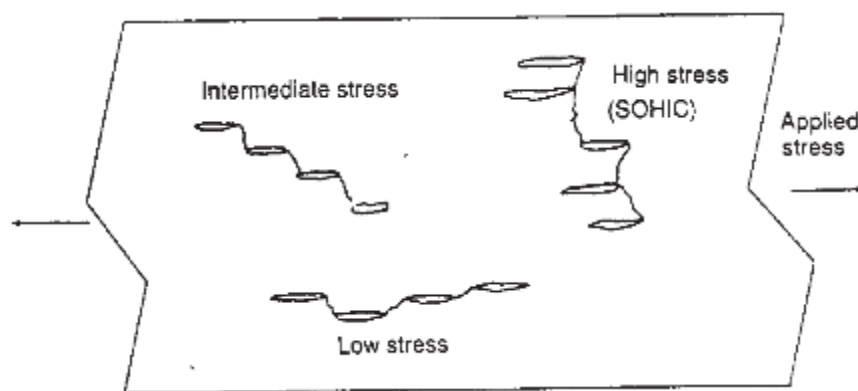


Figure 3: Typical morphology of SOHIC cracks [11].

SSC is distinct from HIC failure and is mainly encountered in pipes exposed to water and H₂S. The main condition for SSC to occur is to have tensile stress and a high hardness value (more than 250 HV). The crack can be observed on the surface of a localized high hardness zone, i.e., hard spots, such as inclusions, cold-worked regions, weld metal, and heat-affected zones, respectively. The occurrence of SSC can be controlled by reducing the microscopic stresses and optimizing the carbon equivalent by implementing suitable post-weld thermal treatment (PWHT) [6].

The NACE-developed test methods NACE TM 0284 (Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking) and NACE TM 0177 (Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H₂S Environments) are used to determine whether the materials of

construction for piping and pressure vessels are suitable for wet H₂S applications [5]. NACE TM 0284 is only conducted on carbon steel materials. This test method has no established passing/rejection criteria; however, the values are stipulated by oil and gas producers. Duplex stainless-steel materials and other Corrosion Resistance Alloys (CRAs) are not inherently susceptible to HIC or SOHIC cracking mechanisms due to the absence of hydrogen trap sites. On the other hand, NACE TM 0177 is conducted on all materials of construction because SSC depends mainly on the susceptibility of the hard microstructure, including duplex or CRA materials. However, NACE MR0175/ISO15156 has the maximum allowable hardness values for all materials. Therefore, SSC is not deemed necessary.

1.2 Research Objectives

Part of the evaluation process of any sour service environment (i.e., crude or natural gas) is to conduct HIC testing on the raw material (i.e., plates/coils) and as a manufactured pipe. This stringent process is applied to ensure the pipelines meet the requirement for sour service. However, many failures were discovered during the manufacturing process of lines even though the raw material is HIC resistant and has passed HIC testing. This study's primary goal is to investigate the HIC behavior of HIC resistant LSAW API X65 pipeline steel samples. The specific objectives are as follows:

1. To investigating HIC behavior in according to NACE TM 0284 as well as electrochemical charging.
2. To identify the damage mechanism encountered in the as manufactured pipe steel samples.

3. To understand the role of pipes manufacturing process (LSAW) in developing HIC.
4. To investigate the effect of heat treatment on HIC resistance of LSAW steel samples.

There are various consequences from discovering the HIC failure after pipes are manufactured. These include the following:

1. Project schedule delays as the HIC test failures are discovered after pipe manufacturing, which may lead to the replacement of large quantities and costly carbon steel plates or coils.
2. If the through-thickness cracks are not discovered during HIC testing, potential failures during operation could occur, leading to catastrophic sequels ranging from loss of lives due to exposure to fatal H₂S gas and damage to oil and gas equipment.
3. A negative cost impact will be incurred due to retesting, scrapping, cutting, and segregating the failed pipes. In addition, project schedule delays will be encountered due to the slow release of the tested tubes.

1.3 High Strength Low Alloy (HSLA) Steels

For the building of transportation pipes, High Strength Low Alloy (HSLA) steels are frequently used. These steels were created to outperform standard carbon steel in terms of mechanical qualities and to offer superior resistance to air corrosion [12–18]. These HSLA Steels are made to achieve higher strength, greater than 275 MPa, instead of obtaining a special chemical composition.

However, the chemical composition of these steels could be different for each specific thickness to meet mechanical requirements [14]. They generally contain 0.05 to 0.2 wt.% carbon and other additional elements such as Nb, Ti, and V in an amount less than 0.1 wt.% and Mn and Mo in an amount exceeding 0.1 wt.%. The HSLA Steels are classified as per American Petroleum Institute (API 5L) into different grades depending on the strength of the material (e.g., X42, X46, X52, X56, X60, and X65 ...etc.). The letter X represents steel grade and digit numbers refer to the minimum specified yield strength of the material in Ksi unit [16].

The HSLA Steels are categorized into six (6) different groups, which are:

1. Weathering Steels: The alloying elements for these steels are copper and phosphorus. They are intentionally added in order to improve the corrosion resistance from atmospheric weather.
2. Microalloyed Ferrite Pearlite Steels: These steels come with carbides or carbide forming elements like niobium, vanadium, and titanium in a small amount (less than 0.1 wt.%). The transformation temperature control, grain refinement, and precipitation strengthening are enhanced as a result of adding these carbide forming elements.
3. As-Rolled Pearlite Steels: This group contains carbon-manganese steels with additional alloying elements that improve strength, toughness, formability, and weldability.
4. Acicular Ferrite (Low-Carbon Bainite) Steels: the carbon content of this group is lower than 0.05 wt.%, and the yield strength is higher than 690 MPa. This allows

this group to have excellent weldability, formability features, and toughness properties.

5. Dual-Phase Steels: The main microstructure for this group is a martensitic inserted in a ferritic matrix. This microstructure provides excellent ductility and high tensile strength properties.
6. Inclusion-Shaped Controlled Steels: The main alloying elements for this group are calcium, zirconium, titanium, or rare-earth elements. This gives this group excellent ductility and through-thickness toughness properties. The addition of these elements strives to modify the shape of sulfide inclusion from elongated to globular/spherical shape [14].

Thermomechanical Controlled Process (TMCP) is the available option to achieve the desired properties of HSLA steels by using ferrite grain refinement and precipitation of micro-alloy carbonitride particles [14]. The TMCP process involves thermomechanical rolling and accelerating cooling to obtain higher strength and toughness steel plates, and this is achieved by refining the transformed microstructure. Initially, ferrite nucleation sites are increased during cooling by refining the grains and deforming the austenite. After then, the structure is carefully polished by accelerating cooling through a series of controlled rolling phases. A significant driving force for transformation should be present at a lower transformation temperature, which can be reached by chilling sufficiently to restrict atom diffusion [16]. Sometimes, some steelmakers tend to first hot-roll the plates and soak them for a certain period of time and then submit them on an inline quenching and tempering process. The microstructure of steel after applying quenching and tempering process is bainite-martensite. This microstructure has appeared with no

accelerated cooling application. The tempering process is expected to soften the martensite and improve its ductility and toughness [15].

A new process of TMCP, called unconventional TMCP, was introduced in 1998. Obtaining high strength and high toughness (with less alloying elements) is the primary goal of this method. The microstructure of the steels produced by unconventional TMCP is composed of a finely distributed martensite-austenite component (MA) as the second phase and a bainitic matrix as the first phase. An advanced accelerated cooling is also applied to achieve highest cooling rate. The system also includes online heat treatment (HOP) induction heating equipment. A schematic TMCP diagram for "conventional" and "unconventional" production processes is shown in Figure 4-A. It can be seen that the conventional TMCP involves controlled rolling followed by accelerated cooling and, finally, air cooled. On the other hand, fast heating occurs following accelerated cooling in the unconventional TMCP, which is then succeeded by air cooling. Figure 4-B shows the alterations in microstructure brought about by the HOP process[15].

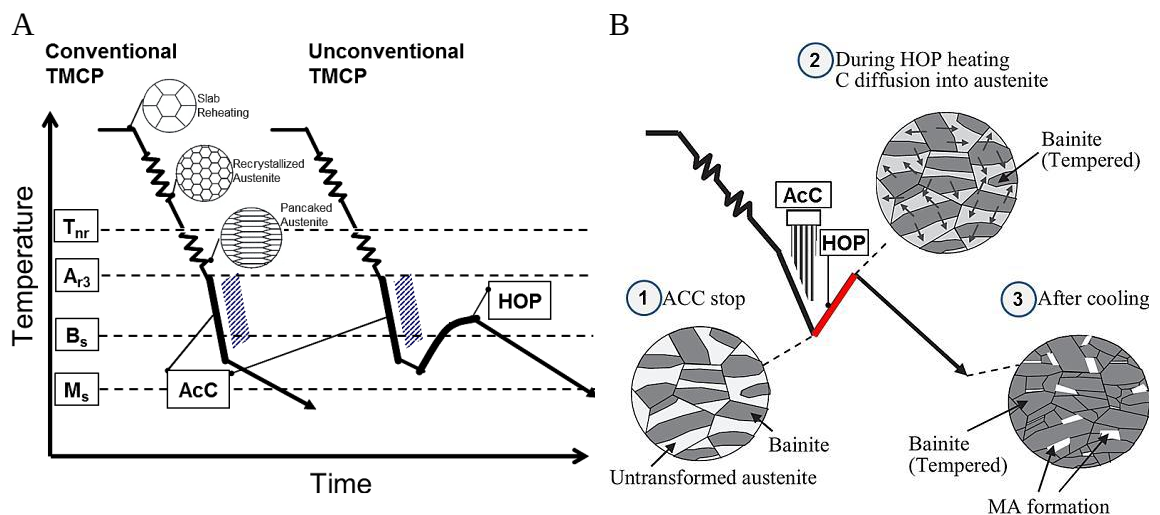


Figure 4: Schematic of Conventional and Unconventional of TMCP process (A), Microstructural changes introduced by applying HOP process [15].

Hydrogen Cracking (HIC) is generally mitigated by using HIC resistance materials as a common line of defense. However, these HIC resistance materials are not fully immune

from HIC failure due to several factors, such as different microstructures, process environment, alloying elements...etc. Hence, it is worth highlighting the manufacturing process of HIC plates/coils and the pipeline manufacturing process.

1.4 HIC Resistance Steel Plate Manufacturing Processes

The manufacturing process of HIC resistance plates/coils is different than normal steels. Figure 5 shows the typical manufacturing route for HIC resistance steels. The furnaces used for producing HIC resistance steels are Kambara Reactor (KR), Blast Oxygen Furnace, Ruhrstahl Heraeus (RH) Vacuum Degassing, and Ladle Furnace. Then, the molten steels shall be shaped in a continuous casting machine followed by rolling and accelerated cooling.

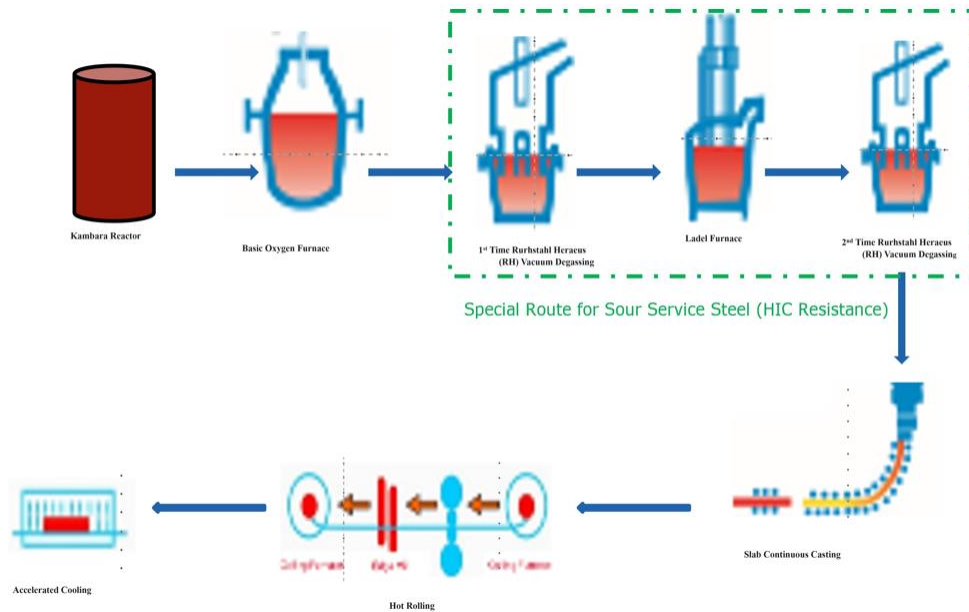


Figure 5: Typical manufacturing route for HIC resistance plates/coils

The following subsections shall discuss the major steps in producing HIC resistance plates.

1.4.1 Kambara Reactor (KR) Pretreatment

The main objective of the KR pretreatment process is to desulfurize the molten metal before sending it to the Blast Oxygen Furnace. The pretreatment process is carried out by stirring the molten iron ore and charging the reactor with flux that contains mainly Mg powder and CaO to achieve a sulfur level below 0.005%. This process shall be completed at a temperature as high as 1180 °C.

1.4.2 Basic Oxygen Furnace

The hot metal from KR shall be weighed in order to determine the amount of scrape, flux, oxygen, and addition of a small amount of iron ore if needed (Figure 6. Gases are injected by oxygen and nitrogen from top to bottom, respectively).

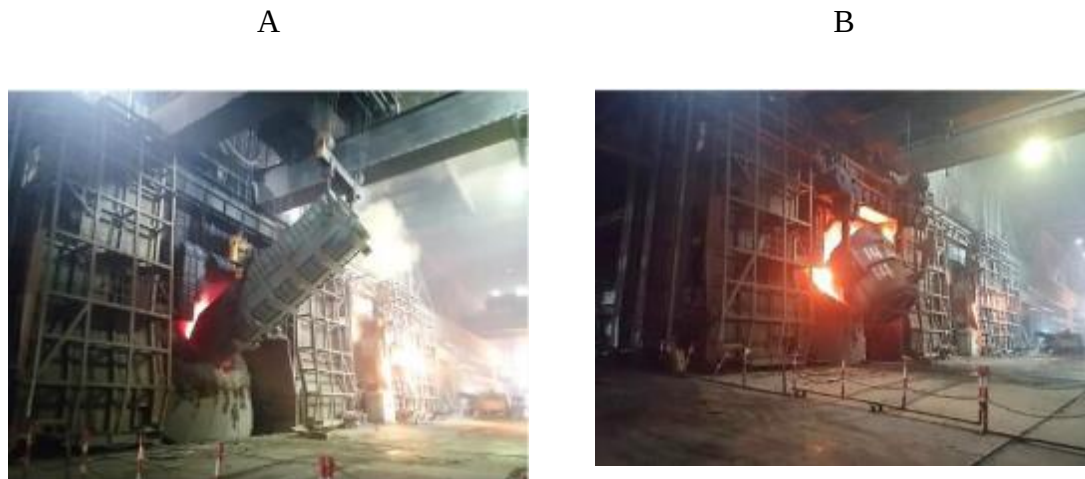


Figure 6: Feeding Basic Oxygen Furnace (A) with Scrape and (B) Molten Metal.

Thereafter, slag formation agents such as burnt lime and dolomite, together with some fluorspars, are dropped in the furnace. In the BOF, the main controlling parameters are ensuring Phosphorus (P) content is below 0.015%, Carbon (C) content is below 0.05%, the temperature of molten metals is in the range of 1320-1420 °C and tapping

temperature is in the field of 1700 °C. Then, the molten metals shall be sent to 1st time RH Vacuum Degassing.

1.4.3 1st Time Ruhrstahl Heraeus (RH) Vacuum Degassing

The Ruhrstahl Heraeus (RH) Vacuum Degassing Furnace is a special furnace compared to the traditional steel-making process. The main purpose of this furnace is to further decarburize molten steel to a level of no more than 0.01% after BOF. This decarburization process provides excellent smelting conditions for subsequent steelmaking processes and a control basis for stable and controlled carbon content. The vacuum degassing furnace operates in 2 bar or less negative pressure, and the molten steel is circulated at a rate of 100 tons/min. The holding time for vacuum-degassing furnaces is more than 10 min.

1.4.4 Refining (Ladel Furnace)

The molten metal is directed to Ladel Furnace after 1st Time RH Vacuum Degassing, where the refining process takes place. Inert gas is injected from the bottom of the furnace to create stirring action and thorough blending to reduce the inclusion content of molten steel. Scrape, charge carbon, and flux are sometimes added in the Ladel Furnace. The Ladel Furnace is heated via three (3) AC electrodes, Figure 7, each with its own phase. The resistive heat produced when an arc strikes air provides the melting energy. In this process, the target carbon content is controlled by combining it with the consumption of electrodes.

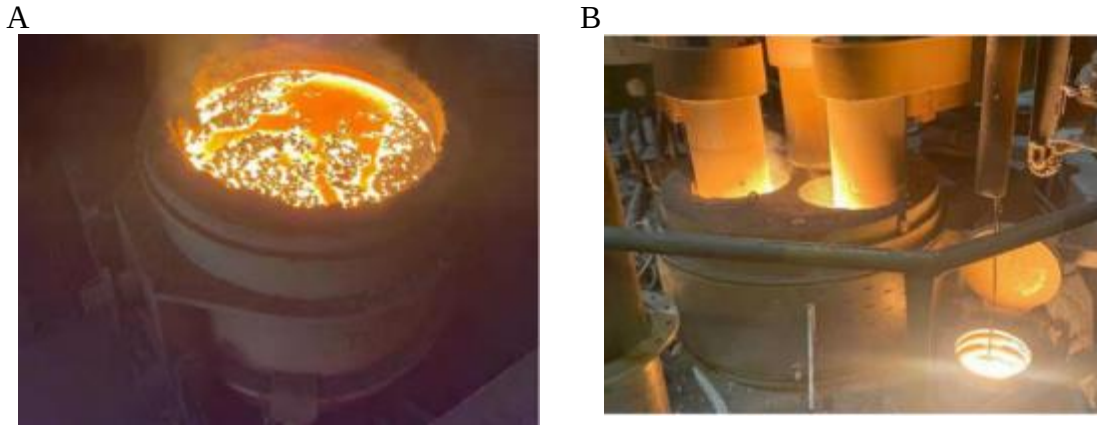


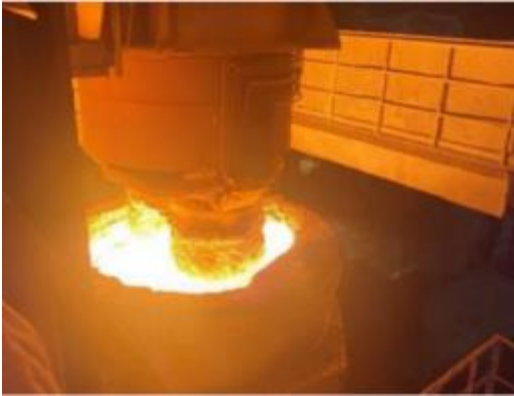
Figure 7: Ladel Furnace (A) after heating, (B) Electrodes for heating Ladel Furnace.

To gather these undesirable components and offer a basic material for CO evolution to foam, a slag high in lime is utilized. Through its ability to stop the unshielded arc from radiating directly onto the furnace roof, this foamy slag contributes to increased energy efficiency during heating. Up until tap time, the foaming motion keeps the slag constantly flushed from the EAF. After reaching the proper temperature and steel chemistry (1620–1650 °C), the steel is tapped through a spout, placed in a ladle, and placed into a ladle furnace for further RH treatment.

1.4.5 2nd Time Ruhrstahl Heraeus (RH) Vacuum Degassing

The main objective of the 2nd vacuum degassing furnace is to minimize the content of dissolved gases, particularly hydrogen and oxygen. The vacuuming system is designed to expose molten steel to a vacuum and create turbulence to free the dissolved gases, Figure 8 to ensure greater soundness of final products.

A



B



Figure 8: (A) Rurhstahl Heraeus (RH) Furnace, (B) Temperature Measuring During RH

The vacuum degassing furnace is operating at negative pressure (2 bar) and a circulation flow rate of 100 tons/min. Ca-Si line is injected into molten steel at a rate of 200m/min to facilitate the formation of inclusion. In the second vacuum degassing, soft stirring is carried out to enhance the floating of inclusion and enhance dehydrogenation of moulded steel to reach below 1.5 ppm.

1.4.6 Slab Continuous Casting

Figure 9 shows typical process for continuous casting of molten steels.

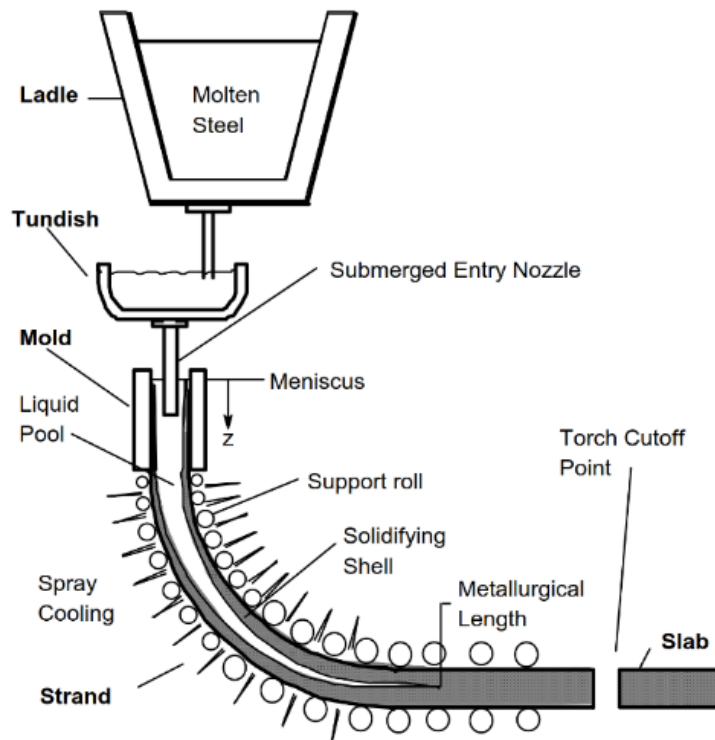


Figure 9: Schematic of steel continuous casting.

Molten steel travels from a ladle via a tundish and into the mold during continuous casting. Both the ceramic nozzles between the plates and the slag cover covering each vessel shield the molten metal from the air. Once within the mold, the molten steel solidifies into a shell as it freezes up against the water-cooled copper mold walls. In order to maintain a constant state throughout the operation, the drive wheels lower in the machine continually remove the body from the mould at a pace, or "casting speed," that corresponds with the flow of incoming metal. The hardening steel shell serves as a container to hold the residual liquid underneath the mould exit. Rolls prevent the steel from expanding too much as a result of the thermostatic pressure. Until the molten core solidifies, the surface temperature of the strand is maintained by spraying water and air mist to cool it down in between rolls. The strand can be torch-cut into slabs once the center has solidified to its fullest extent (at the "metallurgical length").

1.4.7 Slab Reheating & Descaling

After the slabs are cut in the continuous casting shop, the slabs are reheated again in a reheating furnace that is operated with mixed fuel gases. The reheating temperature is in the range of 1150 – 1260 °C.

The descaling is carried out to surface scales by high-pressure water at a level of a minimum of 20 MPa, Figure 10.



Figure 10: Surface Descaling

1.4.8 Hot Rolling

Hot rolling is carried out by a single stand four-high mill with max. Rolling force of 8,000 tons, Figure 11. Rough rolling is conducted at around 1050°C with multiple passes to achieve a thickness reduction of 60%. After the temperature of slabs was cooled to around 850°C, finish rolling was carried out subsequently to achieve final thickness.



Figure 11: Hot Rolling

1.4.9 Accelerated Cooling

After the finished rolling phase, plates are moved to the Accelerated Cooling Device and then cooled to a proper temperature (600°C) with an appropriate cooling rate (20°C), Figure 12.



Figure 12: Accelerated Cooling

The main parameters that need to be controlled in Accelerated Cooling are start and end cooling temperatures and cooling rate.

1.5 Pipes Manufacturing Processes

There are many technologies available for processing and manufacturing pipelines due to the limitations of these technologies. These manufacturing processes are classified into two main categories, which are seamless and welded pipes. The seamless pipes are produced without interaction with the welding process. However, the welded pipes are categorized based on the welding process implemented, which includes longitudinal submerged arc welding (LSAW), spiral welding, helical welding and electric resistance welding. This research will focus on the explanation of the manufacturing processes of longitudinal submerged arc welding (LSAW) pipes. These pipes are usually manufactured via three main methods, which are JCOE, UOE, and Three Roll Bending techniques. These techniques are designed to apply cold forming on the plate to shape the pipe [19,20].

1.5.1 LSAW-JCOE Technique

This technique uses the forming press as the main component for shaping the pipes. JCOE technique has recently dominated the pipe mill market compared to other approaches (UOE and Three Roll Bending) because it offers high product quality, great ductility, and low investment cost. It is also widely utilized in the production of substantial wall thickness small diameter pipes [19]. The JCOE process can produce lines up to 18.3 m in length and with a maximum wall thickness of 45 mm, with a diameter range of 457 to 1422 mm (18 to 56 inches) [20]. In this process, the initial step is to crimp and mill the edge of the plate prior to introducing it to the forming process. Then, the plate is moved to a bending position by manipulators to develop open-seam pipes using a die that matches the pipe dimensions. Two primary manipulators bend the plate's initial side into a half-round (the J shape) during

the forming press stage. After that, the containers will move to the opposite side of the plate and across the second manipulator to form an O and a C shape.. This is to produce the open seam pipes. Thereafter, welding shall be carried out to form the final shape of the lines and followed by a cold mechanical expander to relieve residual stresses developed during welding [38]. Figure 13 shows a schematic diagram of the JCOE Technique.

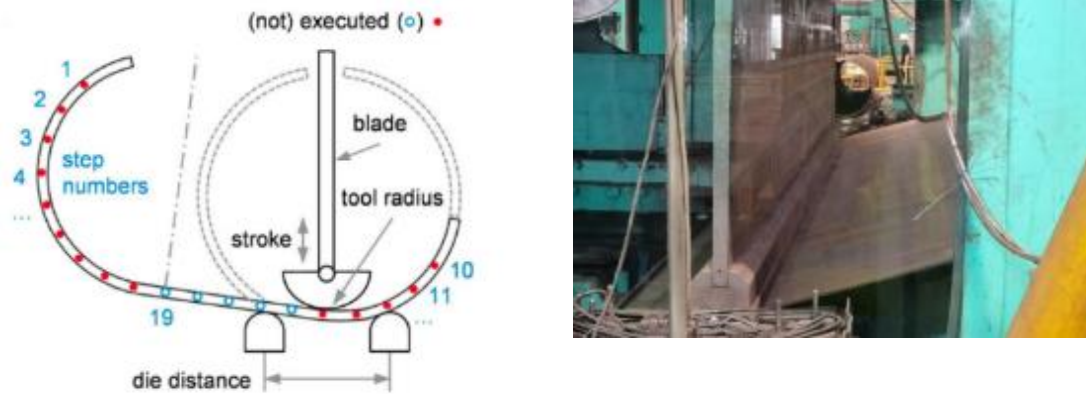


Figure 13: JCOE Forming Technique [20].

1.5.2 LSAW-UOE Technique

This technique is one of the cold-forming processes that deform the plates to shape pipes. This technique uses the same process for bending and crimping the edges of plates prior to forming. The UOE refers to actual mechanical steps to create the lines. U stands for shaping the container to generate a U-shape, and O indicates the second stage where the plates will be included in an O-shape. Thereafter, a longitudinal welding seam is introduced to create the final shape of pipes. Eventually, the E represents the cold

mechanical expansion where the absolute condition of the lines is obtained [21,22].

Figure 14 shows a schematic diagram for the UOE process.

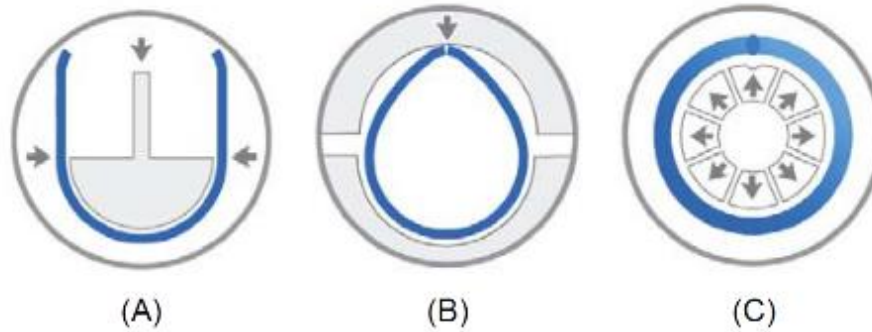


Figure 14: UOE Forming Process. (A) represents U-shape, (B) represents O shape and welding and (C) represents mechanical expansion [22].

1.5.3 LSAW-Three Roll Bending Technique

This technique is totally different from JCOE and UOE, as the edges of the plates shall remain flat without bending and crimping. Whereas the crimping process of advantages will take place after the pipes are formed using a cutter bending machine [19]. The loading site, roll bending stage, and unloading stage are the three crucial steps in this operation. In principle, the three-roll bending operates by placing three identical rollers in space and putting the pipe in between them. Then, the two lower rollers rotate with each other in the same manner via an actuating motor to move the line under the friction of these rollers. Thereafter, the upper roller will start moving as a reaction to the clash of pipe movement. The push plate permits the pipe to proceed in order to complete the line's direction. Every individual pipe segment is subjected to three positive and three reverse bending areas when the line is positioned on the rollers. Additionally, each roller is equipped with a taper at the end of the roller. To make sure the pipe is smoothly stung between the three rollers under the pressure of the push plate, the loading end taper is

utilized. However, the taper of the unloading lot is utilized to safeguard the tube when unloading after receiving several reciprocating bends [23]. Figure 15 illustrate a schematic diagram for three roll-bending technique to form lines.

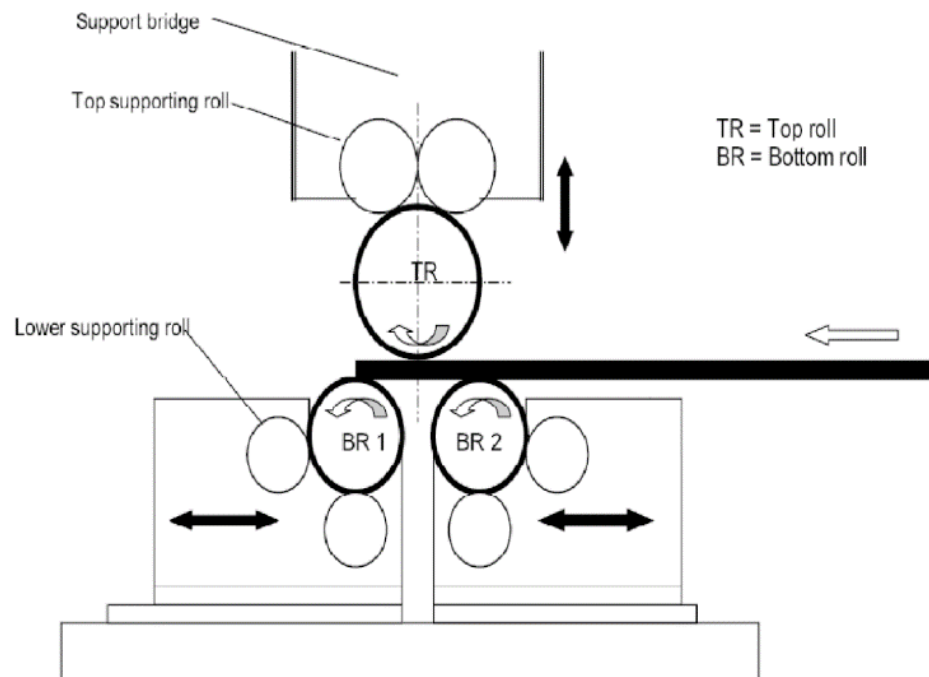


Figure 15: Schematic of three roll bending process for forming pipes [19].

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Both the HIC and Hydrogen embrittlement (HE) are broad descriptions of damage mechanisms encountered when metals are exposed to a hydrogen environment. It is a type of metallurgical damage that results in reducing the ductility and fracture toughness of metals, as defined in API-571 [6]. In 2016, NACE issued a report titled "International Measures of Prevention, application, and Economics of Corrosion Technology Study," which highlighted the 2.5 trillion USD global cost of corrosion [24]. Unfortunately, the cost associated with hydrogen-induced cracking (HIC) damage is not presented in the report, which means that the cost of corrosion resulting from HIC damage cannot be measured that easily as compared to other decline-related failures [24]. The need for gas and oil has skyrocketed due to the world's growing need for fossil fuel energy [9,24]. In order to fulfil these demands, energy companies tend to process even H₂S containing crude and gas. H₂S containing sour oil and gas has a higher tendency to corrode metallic components, including piping, pipelines, and pressure vessels, during normal operation. In principle, hydrogen atoms penetrate and diffuse into the steel surface and become trapped in either reversible (grain boundaries and dislocations) or irreversible (nonmetallic inclusions and precipitate/interfaces) trap sites, Figure 1. Accordingly, the hydrogen atoms, once trapped, generally try to create hydrogen molecules (H₂), which develop huge internal pressure and consequently bulging of the steel surface, referred to as hydrogen blister formation in steels. When these metallic components are used in sour service, they typically suffer from "wet H₂S damage," which can be divided into four primary categories as illustrated Figure 16: hydrogen

blistering, hydrogen-induced cracking (HIC), stress-oriented hydrogen-induced cracking (SOHIC), and sulfide stress cracking (SSC)

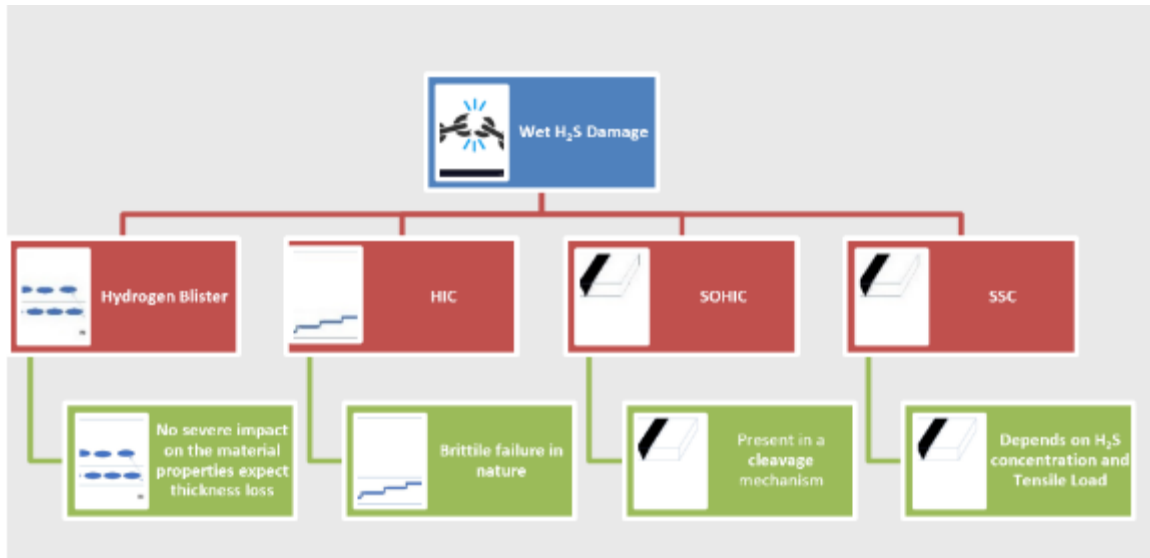


Figure 16: Types of Hydrogen Embrittlement or Wet H₂S damages.

HIC can be found at different locations, such as internal surfaces, outer surfaces, in the middle of the steel thickness, and near weld areas. The extension of this cracking parallel to the rolling direction results in stepwise cracking, and that does not require the application of external as well as residual stresses, Figure 17 [25].

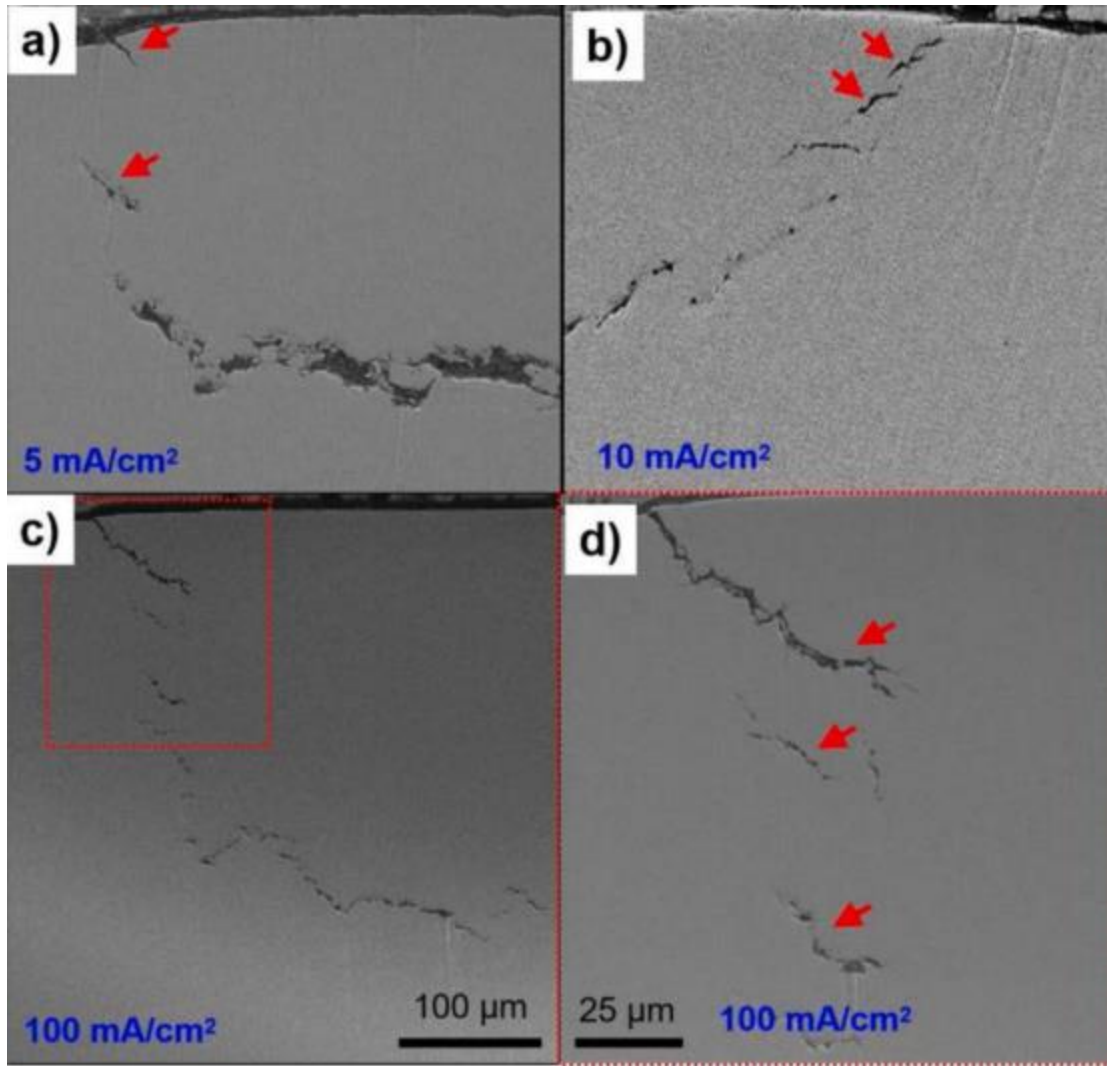


Figure 17: Classical HIC or stepwise cracking after hydrogen charging at different charging current densities: (a) 5 mA/cm², (b) 10 mA/cm² and (c) 100 mA/cm², with the enlarged image for the region marked by red line in (c) shown in (d) [25].

Pressouyer and colleagues have brought attention to the part MnS inclusions play in HIC cracking. The distribution, size, and form of these inclusions are important factors in determining how susceptible certain materials are to HIC.. In general, large, elongated, and homogeneously distributed inclusions are the most susceptible to causing HIC failures. The metallurgical features, such as the microstructure of the surrounding area of the inclusions, are also critical factors determining HIC susceptibility, as HIC cracks can propagate more easily on hard surfaces. Additionally, the density of inclusions is also

considered important, and it is reported that an increase in inclusion density accelerates the HIC crack propagation [26].

The highly stressed areas around the weld seam in non-cold-expanded spiral welded pipes are a result of the welding and edge creation processes. It is important to note that the industry does not now have access to the technology needed to enlarge the spiral pipe. Since spiral pipes are far more expensive than cold-expanded or heat-treated longitudinal tubes, heat treatment is not a financially viable alternative for relieving residual tensions in the pipes. Furthermore, the heat treatment procedure will cause spiral pipes' mechanical qualities, straightness, and roundness to decrease [7].

Because of the significant residual stresses from welding in the vicinity of the weld seam, SOHIC, a unique kind of HIC, is more common there. SOHIC failure manifests as staggered, tiny fractures that are roughly perpendicular to the major stress (applied or residual) axis in both HIC-resistant and HIC-susceptible steels. As a result, pre-existing HIC cracks are linked by a ladder-like crack array. Even in the absence of nonmetallic inclusions or clusters, hydrogen atoms can concentrate in high-stress zones close to the weld fusion line. In certain areas, stacked arrays of HIC may form, which might cause a stepwise cracking that occurs quickly through thickness (SWC). The tensile stress axis is roughly perpendicular to the through-thickness fractures in SOHIC. Gregory et al. investigated the SOHIC of an amine absorber column in a refinery and reported the SOHIC crack formation in the base of HIC-resistant steel owing to the presence of huge applied and residual stresses due to the repair of the vessel's shell [9]. Another type of failure is "SSC failure," which is distinct from HIC failure and is mainly encountered in pipes exposed to water and H_2S in the presence of tensile stress and a high hardness value

(exceeding 250 HV). The crack can initiate on the surface in a highly localized high hardness zone, i.e., hard spots such as inclusions, cold-worked regions, weld metal, and heat-affected zones, respectively. The occurrence of SSC can be controlled by reducing the microscopic stresses and carbon equivalent by applying appropriate post-weld heat treatment (PWHT), as per API-571 (2020).

API 5L carbon steel is one of the most commonly used steel grades in the oil and gas industry for various applications such as transmission and cross-country pipelines, so this review will focus in detail on the issue of HIC in these carbon steel grades. It is important to note that Saudi Arabia operates more than 21,000 km of oil and gas pipeline network under different environmental conditions and may encounter HIC during its service life. Hence, the importance of understanding HIC damage mechanisms and their prevention is extremely important in preventing catastrophic failure [27].

2.2 HIC Testing and Inspection Methods

Evaluating the HIC resistance of steel plates for a wet H₂S service environment is an important step in order to prevent future catastrophic failures. There are various methods to determine HIC resistivity and hydrogen diffusion in steels. They can be divided into three categories, i.e., metallurgical processes, electrochemical techniques, and nondestructive testing (NDT) tools, as described below in detail.

2.2.1 Metallurgical Microscopical Tools

NACE TM0284 "Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen Induced Cracking" is one of the mainly used industrial standards for selecting HIC-resistant steels, Figure 18.

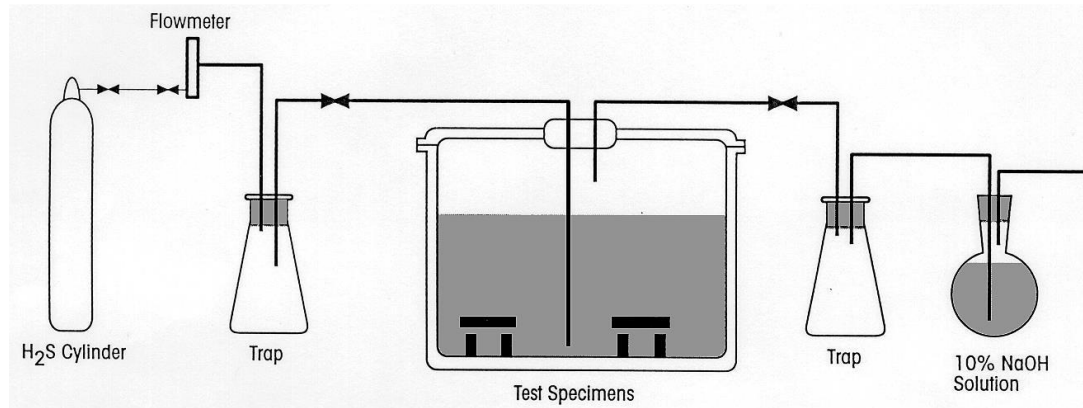


Figure 18: Schematic Diagram of Typical Test Assembly [NACE TM 0284]

This method evaluates the HIC resistance of unstressed steel plates in any one of three different service environments, Table 1, in the presence of H_2S

Table 1: Types of solutions for NACE TM 0284

S.N	Designation	Composition
1	Solution A	An acidified brine solution contains sodium chloride (NaCl) and acetic acid (CH_3COOH) dissolved in distilled or deionized water saturated with H_2S at ambient temperature and pressure.
2	Solution B	Synthetic seawater solution saturated with H_2S at ambient temperature and pressure.
3	Solution C	The buffered solution contains sodium chloride (NaCl) and sodium acetate (CH_3COONa) dissolved in distilled or deionized water saturated with a gas containing mixtures of H_2S and CO_2 at ambient temperature and pressure.

After completing the test, the sample should be subjected to NDT evaluation, such as ultrasonic testing and metallography examination [5]. This is followed by crack length ratio (CLR), crack sensitivity ratio (CSR), and crack thickness ratio (CTR) calculation as below.

$$CLR = \frac{\sum a}{W} \times 100\% \quad (2.1)$$

$$CSR = \frac{\sum (a \times b)}{(W \times T)} \times 100\% \quad (2.2)$$

$$CTR = \frac{\sum b}{T} \times 100\% \quad (2.3)$$

where W is the section width, T is the test specimen thickness, an is the crack length, and b is the crack thickness. It should be highlighted that there are no acceptance and rejection criteria for this test. However, oil and gas companies have developed their own acceptance ranges.

The typical CLR, CSR, and CTR values are 10%, 2%, and 3%, respectively.

NACE TM 0284 was carried out on electrical resistance welded API X52 pipeline steel by Khalili et al. For 96 hours, they submerged the samples in a distilled water solution containing 0.5 weight percent acetic acid and 5 weight percent salt chloride. The nitrogen was purge for one hour at a rate of 100 mL/min in order to deaerate the solution. The solution was then used to clear the H₂S gas. At room temperature, the initial and final pH values were 2.7 and 3.4, respectively.. Finally, the samples were sectioned and examined under the microscope to determine the CLR (12%), CSR (0.5%) and CTR (2.5%). Figure 19 shows SEM and EDX results of the developed cracks where Si-rich and sulfide inclusions were the main cause of the crack initiation.

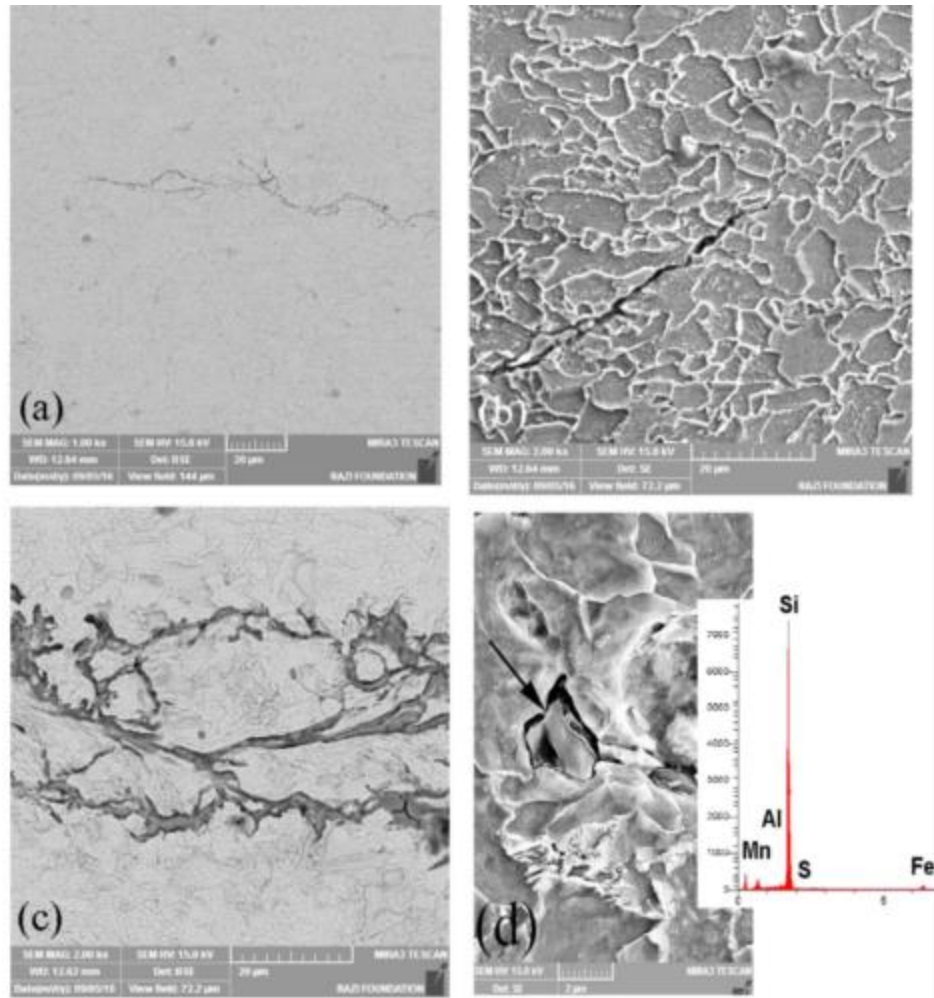


Figure 19: SEM and EDX results of HIC failure encountered after conducting NACE TM 0284 [28].

2.2.2 Electrochemical Technique

Electrochemical methods are also quite popular and can be used to measure hydrogen permeation in steels. ISO 17081, "method of measurement of hydrogen permeation and determination of hydrogen uptake and transport in metals by an electrochemical technique," is used extensively in the industry. Table 2 summarizes different electrochemical testing methods [5].

Table 2: Summary of Electrochemical Tests for HIC evaluation

S.No.	Testing Technique	Brief Description	Expected Outcome
1	Hydrogen microprint (silver decoration) technique	- It can slow the local hydrogen distribution of atomic hydrogen. - Silver bromide (AgBr) loses its silver ion (Ag⁺) due to the detected hydrogen. As a result, metallic silver (Ag⁰) fragments are produced, which serve as hydrogen indicator sites. After that, unreduced AgBr crystals are cleaned with a solution containing 1.4 mol/l sodium nitrate (NaNO₂) and 0.6 mol/l sodium thiosulfate (Na₂S₂O₃). At that point, metallic silver (Ag⁰) is the only element visible on the steel surface, indicating the location of hydrogen present at the imperfection.	Detects hydrogen at microscopic level
2	Thermal desorption spectrometry	Before being heated at a regulated pace in a tube furnace, the specimen is electrochemically charged with hydrogen or submerged in hydrogen gas inside of a container. The hydrogen emitted is then measured and quantified using a gas chromatograph that is mounted on the stove.	The expected outcome is to show the average values corresponding to the hydrogen desorption rate.
3	Devanathan–Stachurski (DS) method	- Double cells apparatus. The first cell works as a cathode cell that is typically considered the source of hydrogen charging, and the next cell, called an anodic cell, acts as hydrogen desorption. - The susceptibility to hydrogen damage is obtained from the hydrogen diffusion coefficient.	The outcome will show the hydrogen diffusion pattern, trap density, and relate the hydrogen flux with time.

Jack et al. conducted a hydrogen permeation experiment using the modified DevanathanStachurski electrochemical cell, Figure 20, to investigate the HIC behaviour of API X65 by ISO 17081 [4]. The samples (20mm X 20mm X 2mm) were cut from different steel plates, and tests were conducted in a solution mixture of "0.1 M H₂SO₄ + 3 g/L NH₃SCN+ 0.1 M sodium hydroxide (NaOH)" in charging and oxidation cells

respectively. In the oxidation cell, argon was utilized for deaeration, saturated calomel electrode (SCE) with 3 M potassium chloride (KCl) as a reference electrode, and graphite rod served as the counter electrode in both cells. A G750 Gamry potentiostat provided a steady voltage of 250 mV for the oxidation cell, while an Instek DC power supply provided a five mA charging current. Equations like these were used to compute the parameters, including permeability coefficient ($J_{\infty}L$), effective diffusivity (D_{eff}), apparent solubility (C_{app}), and total number of hydrogen-trapping sites (N_T):

$$J_{\infty}L = \frac{I_{\infty}L}{FA} \quad (2.4)$$

$$D_{eff} = \frac{L^2}{6 t_L} \quad (2.5)$$

$$C_{app} = \frac{J_{\infty}L}{D_{eff}} \quad (2.6)$$

$$N_T = \frac{C_{app}}{3} \left(\frac{D_L}{D_{eff}} - 1 \right) \quad (2.7)$$

Where F is Faraday's constant = 96,500 cm mol⁻¹, L (cm) is the steel membrane thickness, A (cm²) is the specimen area that was charged and oxidized, and DL (lattice diffusion coefficient) = 1.28 X 10⁻⁴ cm² s⁻¹.

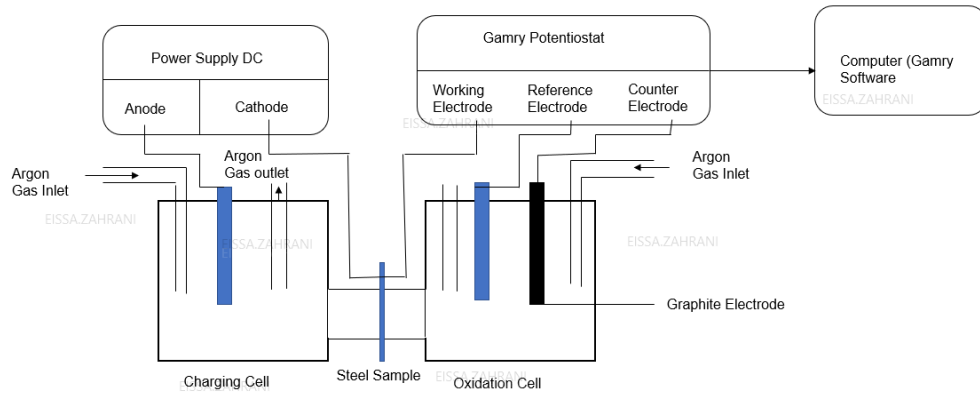
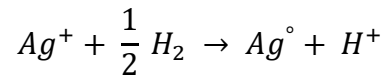


Figure 20: Typical arrangement of Hydrogen permeation testing in accordance with modified Devanathane-Stachurski electrochemical cell [4].

Thermal Desorption Spectrometer (TDS) is used by cathodically connecting the steel samples with atomic hydrogen. The system shall analyze the hydrogen atoms leaving the specimens as soon as the temperature increases. As a result, the system determines the

hydrogen trapped in each single lattice defect of the steel sample, like lattice hydrogen, dislocation hydrogen, grain boundary hydrogen, and hydrogen trapped in sediment [29]. Furthermore, the hydrogen microprint technique is incorporated with other electrochemical techniques to detect hydrogen microscopic levels in steel [5]. Jack et al. investigated the HIC behavior of API 5L X65 pipeline steel using the hydrogen microprint method. To expose the grain boundary, they used 2% vital to etch one surface of the sample. Next, they applied silver bromide (AgBr) emulsion—a mixture of 5 g of AgBr powder and 10 mL of 1.4 M sodium nitrite (NaNO₂)—on that same etched surface. The specimen was then charged in the hydrogen permeation testing setup's charging cell. According to the following equation, the hydrogen will thus seep through the steel membrane and react with the emulsion on the other side of the steel, reducing the silver on the emulsion side.



The sample was then submerged in a solution containing 1.4M sodium nitrite (NaNO₂) and 0.6M sodium thiosulfate (Na₂S₂O₃) for one minute in order to remove the unreacted emulsion and leave reduced silver as a white deposit on the steel surface. Finally, the specimen was subjected to a SEM examination in order to track the hydrogen's route through the steel sample., as shown in Figure 21 [4].

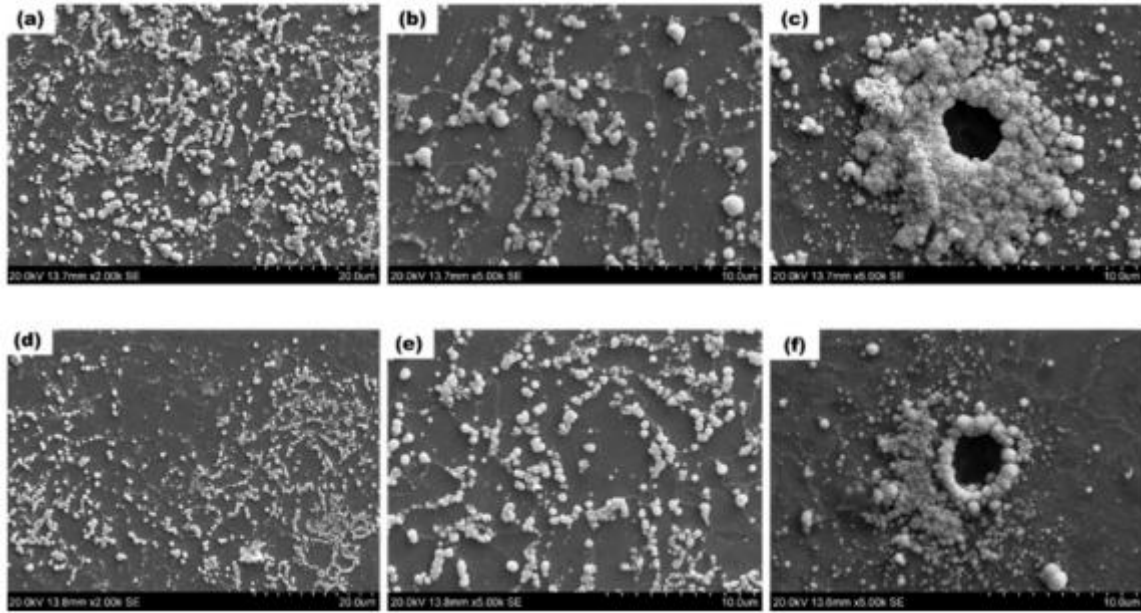


Figure 21: Hydrogen microprint technique, where SEM image showing reduced silver as white deposits on steel surface of API 5L X65 steel [4]

2.2.3 Inspection of HIC

Inspection and monitoring of HIC damage is a crucial step in preventing catastrophic failures in the oil and gas industries operating in sour service environments. Moore Jr. presented several actual case studies on HIC and hydrogen blister damage related to bad services for the oil and gas industries. It was found that seamless pipelines are not prone to HIC damage compared to welded pipes [30].

The available Non-Destructive Testing (NDT) tool for HIC damage evaluation is Ultrasonic Testing (UT). The UT consists of several types, including manual Ultrasonic Testing, Flight Diffraction (TOFD), Phased Array Ultrasonics Testing, and Automated Combined UT. These tools are generally used for evaluating large components, not at the laboratory scale. However, they can be used in the laboratory as required in NACE TM 0284 prior to conducting metallography tests. This facilitates the identification of crack locations and measures the crack length and width ratios. Firstly, the manual UT is

recommended to survey the suspected components due to its low cost. It can provide information on subsurface discontinuity and highlight any presence of subsurface defects. The manual UT does not detect HIC damages accurately. Hence, more advanced techniques should be used along with manual UT in order to evaluate potential HIC damage properly.

The common industrial practice in inspecting HIC damage is to screen the metallic components that are operating in the sour service regime. Once the equipment is identified, the suspected location where wet H₂S damage may be encountered should be examined by manual ultrasonic testing. If the resulting findings are suspected of indicating HIC damage, the suspected site should be analyzed using an advanced nondestructive testing (NDT) technique (phased array NDT) to measure the crack length and width values. The suspected metallic components should be assessed using the American Petroleum Institute (API-579) Fitness For Service (FFS). Accordingly, the new operating pressure should be adjusted based on the findings from the FFS assessment [31–34]. Fig. 8 shows the main steps that are considered while assessing pressure vessels and metallic piping components.

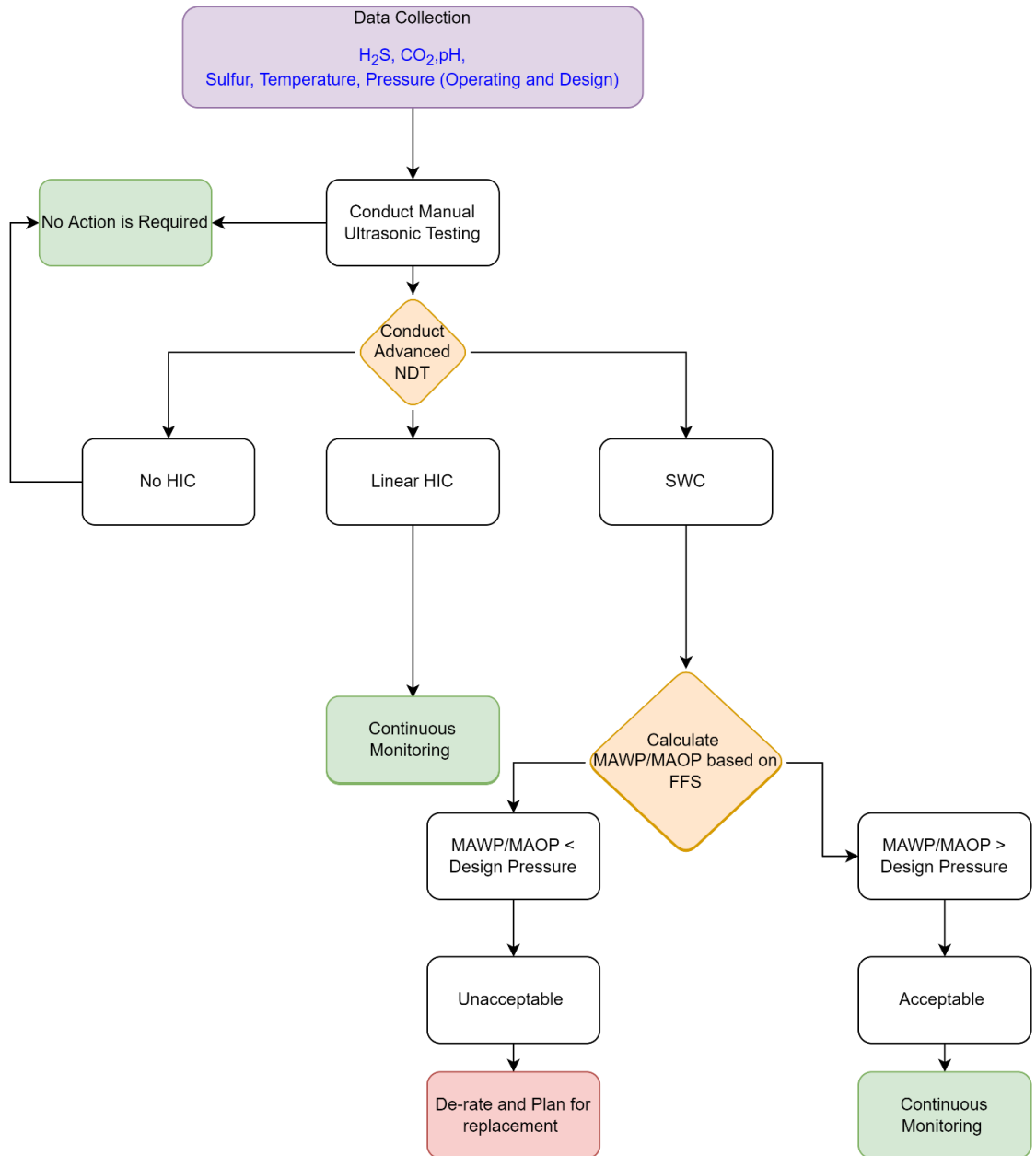


Figure 22: Flowchart of common industrial practice for assessing HIC.

Yasuda et al. developed an in-situ 3D analysis of hydrogen-induced cracking behaviour using the phased array ultrasonic testing technique. They investigated the HIC initiation and propagation during actual testing of HIC. The array was immersed in the test solution, and results were obtained every 30 minutes during the test. Additionally, the hydrogen diffusion behaviour was studied by Finite Element Analysis (FEA). They found

that HIC susceptibility was affected by hydrogen diffusion and corresponded well with the steady value of hydrogen permeability. It was concluded that HIC initiation takes a longer time, and the migration of crack initiation sites into the inside of the specimen is lower at lower H_2S partial pressure, where the steady-state hydrogen permeation is low [34].

2.3 Materials and Environment Interaction

HIC damage is mostly encountered in carbon steel, stainless steel, and corrosion-resistant alloys (CRA). There is plenty of published data discussing the hydrogen-related embrittlement issue in carbon steel, as they are commonly used in many different applications. Carbon steels, due to their lower cost as compared to stainless steels (SSs) and corrosion-resistant alloys (CRAs), are an obvious material selection choice for the end users, as even SSs and CRAs are susceptible to HIC problems. This shows the significance of this problem and that it can occur even in high-grade materials, so a proper understanding of the contributing factors is very crucial. There are many different material environment interaction factors that directly and indirectly affect the HIC, as shown in Figure 23. In the following sections, the effect of microstructure, microtexture, heat treatment, alloying elements, inclusions, precipitates and environmental factors will be discussed in detail.

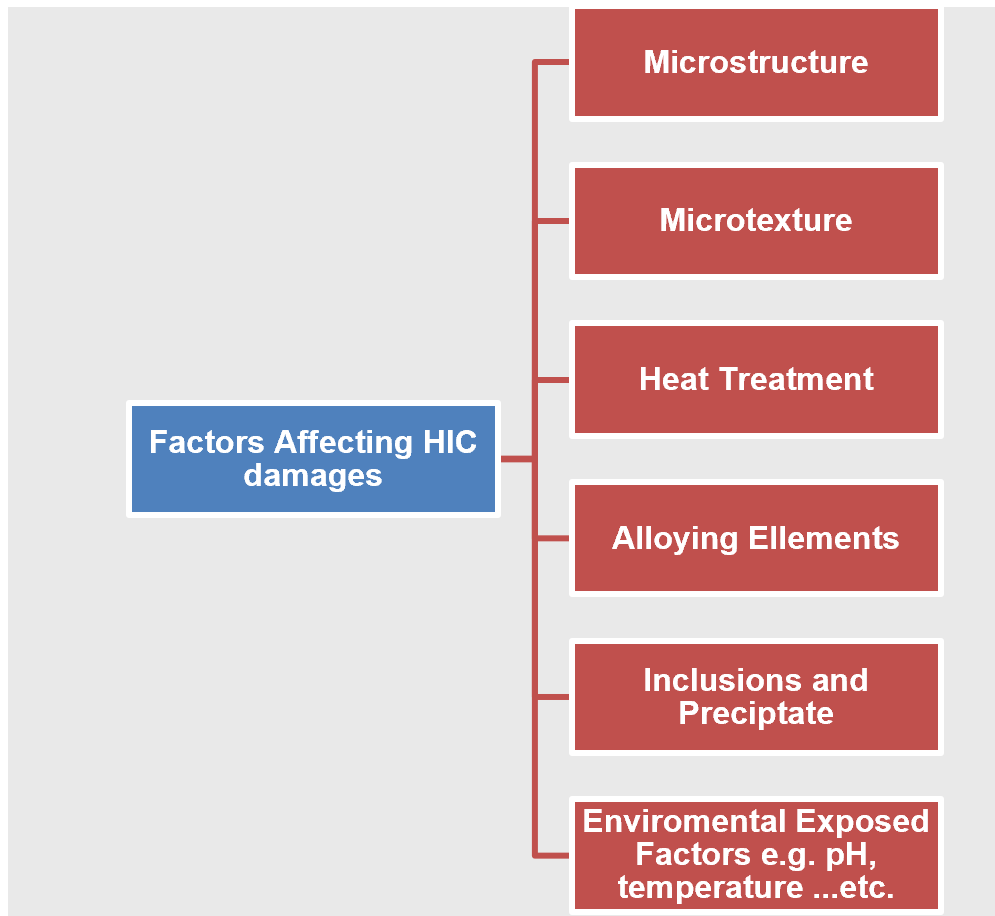


Figure 23: Factors affecting HIC damages on carbon steel materials.

2.3.1 Effect of Microstructure on HIC

The majority of carbon steel API 5L grades are manufactured via the thermomechanical controlled processing (TMCP) technique, Figure 24. The TMCP steels contain phases such as ferrite, pearlite, polygonal ferrite, quasi-polygonal ferrites, Widmanstätten ferrite, acicular ferrite, bainitic ferrite, bainite, martensitic microstructures, etc.

Figure 25 shows the typical microstructure of ferrite-pearlite microstructure, ferritebainite microstructure, bainitic microstructure, and uniform bainitic microstructure [5]. The factors such as "deoxidation practice, steel composition, and processing history" play a key role in HIC susceptibility. These factors affect the morphology of nonmetallic inclusions,

and that will subsequently affect the hydrogen trapping sites and so the HIC of these steels [35]. The impact of microstructure on HIC susceptibility of X80 carbon steel pipe was assessed by H. B. Xue and Y. F. Cheng. The microstructure of the pipe was composed of a polygonal ferrite and bainitic ferrite matrix, with martensite/austenite (M/A) elements dispersed along the grain boundaries., as shown in Figure 26. The reported grain size was in the range of 3.9 μm , and various spherical or elongated shape inclusions were observed, which were found to be Si-enriched, Al oxide-enriched, and–Mg–Ca–O mixture, respectively, as shown in Figure 27. There were no commonly reported MnS inclusions present as sulfur content was very low (0.01 wt.%) in this steel grade. The observed inclusions (Si-enriched and aluminium oxide) were reported to act as crack initiation sites for X80 carbon steel pipe and other high carbon steel materials. It is interpreted that under such conditions crack is generally initiated and propagated without the presence of external stress [36].

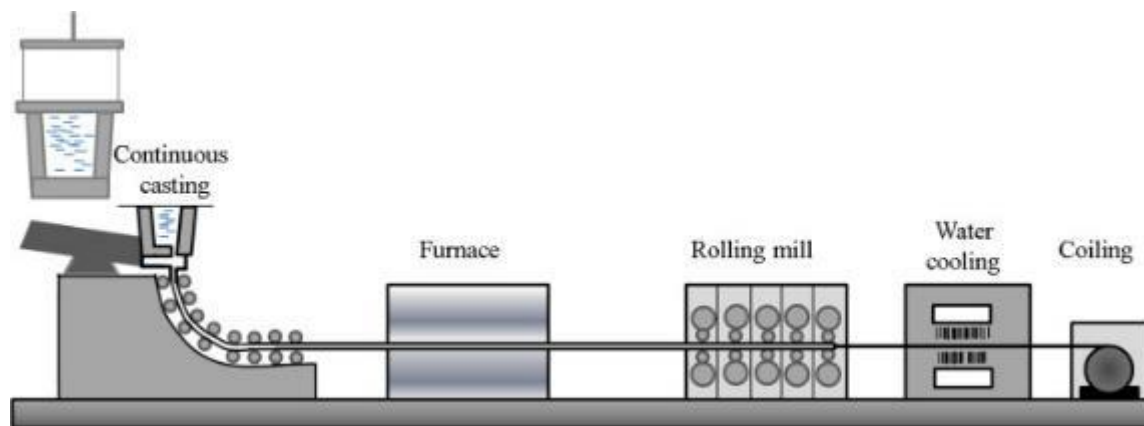


Figure 24: Typical Manufacturing Process of TMCP steels [37].

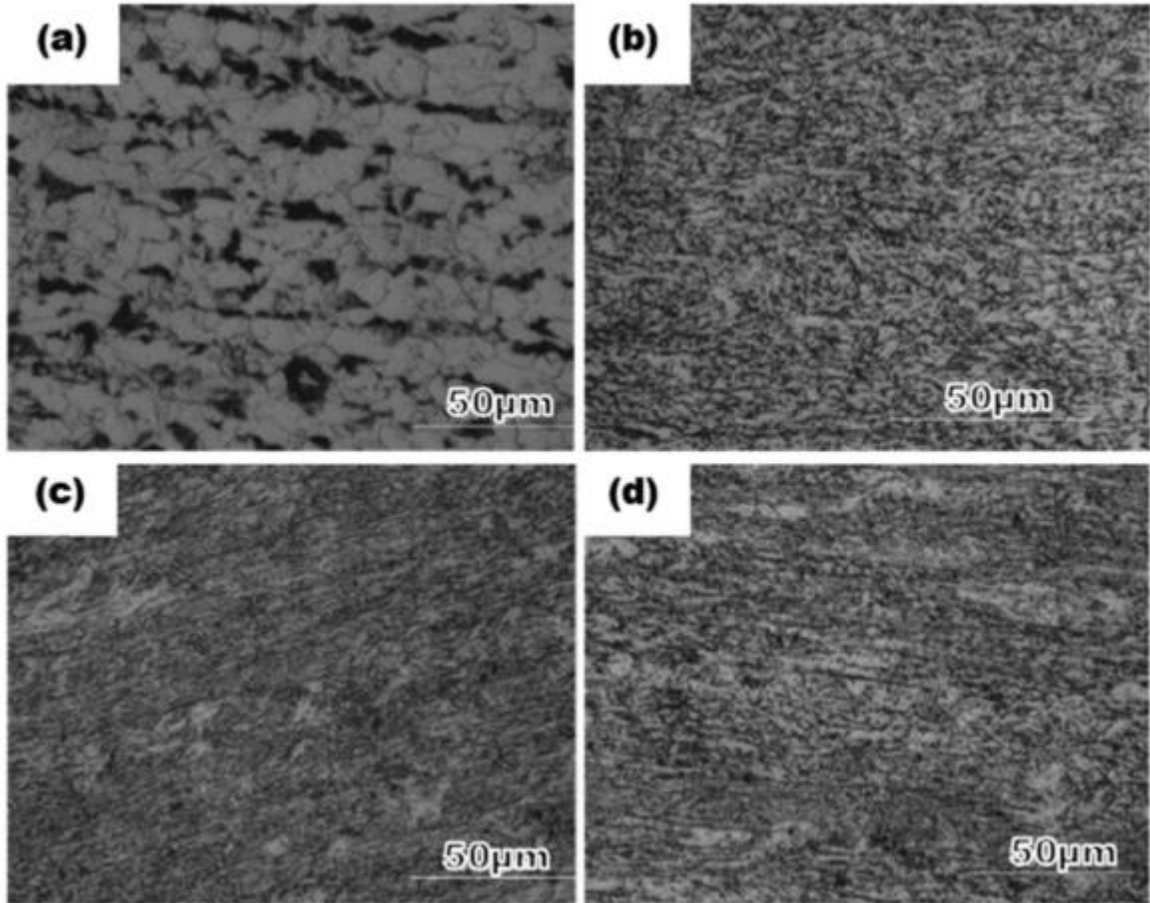


Figure 25: Typical microstructures of ferrite-pearlite (a), ferrite-bainite (b), bainitic (c) and uniform bainitic (d)

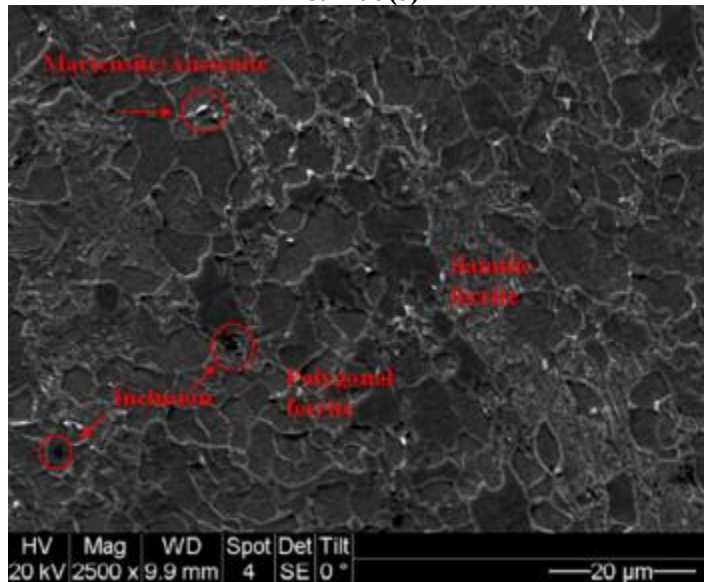


Figure 26: SEM microstructure photo of X80 pipe, Martensite / Austenite phase are shown on the top, bainitic ferrite phase is shown on the right side and polygonal ferrite is shown in the bottom [36].

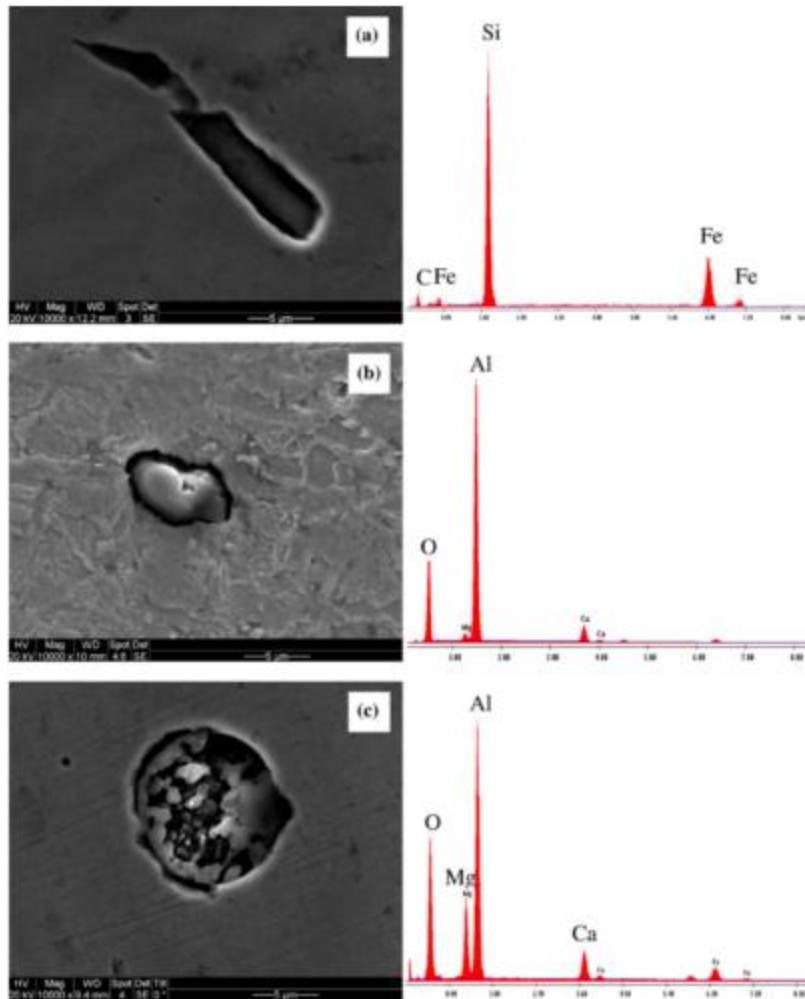


Figure 27: Morphology and composition of different inclusions observed on X80 pipe: a) Si-enriched, b) Al oxide-enriched, and c) Al-Mg-Ca-O mixture [36].

Mohtadi et al. measured the metallurgical, mechanical, and hydrogen permeation

characteristics of the X60 and X70 steels to thoroughly examine the impact of microstructure on the HIC behavior of these materials. In the X60 and X70, respectively, polygonal and acicular ferrite were found. In both steels, bainite and a little area of martensite were also detected.. The published literature shows that the HIC susceptibility of X60 and X70 is mainly influenced by the formation of MnS inclusions. However, the authors reported here that MnS inclusions were not present in X60 steel. Based on HIC results conducted on these two steels, it was reported that X70 is more susceptible to HIC than X60 due to the absence of MnS inclusions in the former [38].

Masoumi et al. investigated how different thermomechanical processes, as well as microstructure, precipitations, and crystallographic orientation, affected the HIC susceptibility of X70 pipe. A pearlite/ferrite banded microstructure has been described, and HIC was seen with some deviation towards the normal direction and parallel to the rolling direction. Based on their data, they concluded that higher HIC susceptibility and fracture propagation are caused by the ferrite/pearlite phase[39].

A detailed discussion of the many elements influencing HIC in oil and gas pipeline steels was provided by M. A. Mohtadi-Bonab et al. Using distinct glass jars for each sample, they performed an electrochemical hydrogen charging experiment using API 5L Grade X70 steel. They submerged the sample in two liters of 0.2 M sulfuric acid and three grams per liter of ammonium thiocyanate. For the experiment, a current density of 20 mA/cm² is employed. For eight hours, the specimens were charged with hydrogen. They stated that ferrite, the weakest and softest component of carbon steel, is thought to be resistant to HIC cracks. However, additional components like carbon and manganese must be added to carbon steel in order to enhance its strength, which lowers its HIC resistance [2].

Goutam et al. conducted a review on HIC damage in pipelines and pressure vessels intended for sour service and reported that HIC occurs on banded ferrite pearlite microstructure. HIC resistance is increased by a low sulfur thermomechanically controlled process (TMCP) because it reduces ferrite/pearlite banding in steels. They discovered that quenched and tempered bainite/martensite microstructure is more resistant to HIC damage than ferrite and pearlite microstructure, and that carbide enrichment may be thought of as a beginning site for HIC.. Conversely, lower bainite

microstructure has better HIC-resistant properties than quenched and tempered steels. In fact, all the metallic components operating in a sour service environment are requested to be satisfied and tempered [40].

Three distinct high-strength pipeline steels with microstructures of granular bainite & lath bainite (GB + LB), granular bainite & acicular ferrite (GB + AF), and quasi-polygonal ferrite (QF) were the subjects of Jing et al.'s investigation. The findings of the research demonstrated that, in contrast to steel with granular bainite & lath bainite (GB + LB), which shown low resistance to HIC damage, steel with granular bainite & acicular ferrite (GB + AF) and quasi-polygonal ferrite (QF) displayed the maximum HIC resistance. It is believed that the bainite phase has a higher strength and is more prone to HIC damage compared to the ferrite phase [41]. Xinfeng et al. investigated the HIC damage on pure iron and found that HIC cracks are propagated in an intergranular manner due to hydrogen pressure. The interior HIC's shape revealed a flat table with a 6:1 length-to-width ratio with damages related to one another in the form of a bridge pattern. The microstructure investigation demonstrated that the internal HIC started at the grain boundaries, where the combined effects of stress concentration and dislocation pile-up led to an accumulation of hydrogen concentration at the crack tip along the grain boundaries [31,42]. It is noteworthy to emphasize that the HIC resistance of pipeline steels is influenced by the crystallographic texture, grain orientation, and grain boundary. Venegas et al. investigated the role of microtexture in HIC interaction and coalescence on pipeline steels and found that the mixed mode stress state and isotropic linear-elastic fracture mechanics responded differently, which in turn affected the HIC damage and accelerated the growth of cracks in the steels under study [31,43]. According to various authors [43–46], the thermochemically controlled process is thought to be among the

best ways currently available to improve the mesotexture with a high proportion of low-angle grain boundaries and the dominated $\{1\ 1\ 1\}$ ND texture fiber in the crystallographic texture, which increases the susceptibility of pipeline steels to HIC damage. The impact of microstructure on the HIC susceptibility of TMCP carbon steels is summarized in Figure 28. .

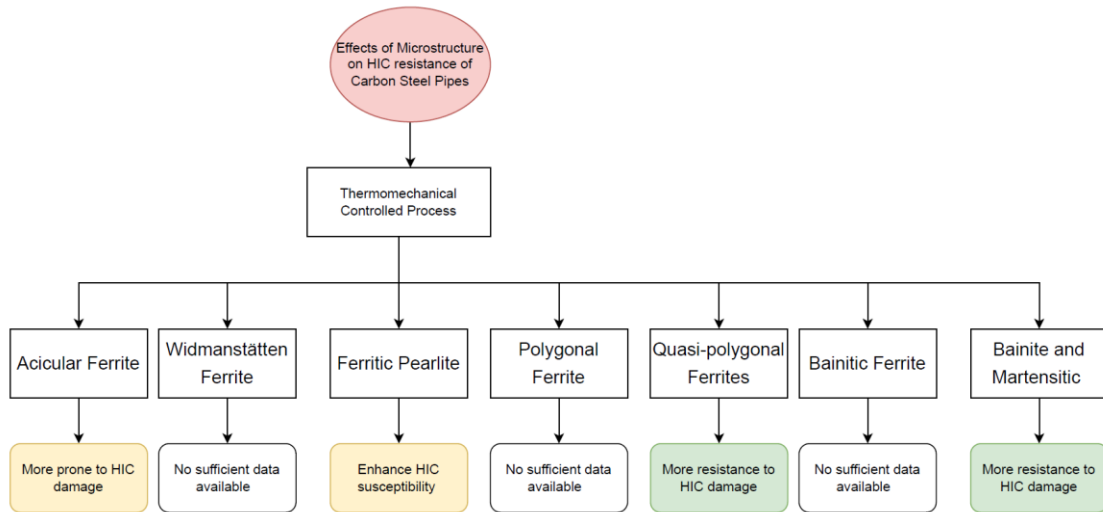


Figure 28: Effect of Microstructure on HIC resistance of TMCP carbon steel pipes

In short, it can be said that microstructure in terms of phases available, phase combinations, inclusions etc. plays a critical role in HIC susceptibility of steels. It is believed that with the availability of cleaner steels being produced using advanced steel making and processing routes, HIC susceptibility can be decreased, as it helps in preventing crack initiation sites. The ferritic phase is considered more HIC resistant than hard phases such as pearlite, as ferrite can absorb more stresses and arrest crack propagation. It is well documented that ferrite/pearlite banded microstructure is prone to HIC, as the crack can initiate at the ferrite/pearlite interface.

The current industrial practice is to use carbon steel grade X70 and below in severe sour environments. By using high strength pipes, such as X80 and higher, the tube's wall thickness and, consequently, its capital cost, may be decreased. HIC failure was not caused

by polygonal ferrite or bainitic ferrite matrix including martensite/austenite elements dispersed along the grain boundary, according to B. Xue and Y.F. Cheng's HIC studies on X80 steel. Though MnS inclusions contribute significantly to HIC, however even in the absence of such supplied inclusions, HIC cracks can initiate at Si and aluminum enriched oxide inclusions [36]. A further investigation needs to take place in order to eliminate the effect of microstructure on the HIC susceptibility of X80 pipelines and ensure the HIC crack will initiate from the microstructure.

2.3.2 Effect of Heat Treatment on HIC

Heat-treatment processes such as quenching and tempering are some of the best solutions to enhance HIC-resistance of materials. Quenching and tempering can be used to optimize the microstructure of steel plates encountered from casting and pearlite banding. On the other hand, the effect of nonmetallic inclusions cannot be resolved by heat-treatment processes. It has been discovered that tempering acicular ferritic steels for 30 minutes at 650 °C decomposes the martensite into cementite and pearlite, hence reducing the sensitivity to HIC damage. Han et al. used an electrochemical permeation approach to examine the impact of heat treatment at 620 °C on acicular ferrite steels. Based on their results, they have concluded that heat treatment at 620 °C for 1 hour on specifically acicular ferrite steels or TMCP pipes tends to increase the hydrogen diffusible content and decrease the HIC susceptibility. It is believed that iron carbide is formed in the grain boundary and acts as HIC crack initiation sites. These iron carbides precipitate on the grain boundary at heat treatment temperature, which subsequently reduces the HIC resistant property of the steel [47]. Kim et al. (2012) carried out HIC testing in accordance with NACE TM 0284 and Sulfide Stress Cracking Testing in accordance with NACE TM 0177

and ASTM G39, respectively, and studied the HIC resistance of ASTM A516 carbon steel for pressure vessel plates at different cooling rates (normalizing heat treatment). It was discovered that the HIC resistance rose in the following order: oil-cooled (OC), air-cooled (AC), and water-quenched (WQ) specimens. The WQ exhibited a bainitic microstructure, AC material showed ferrite/banded pearlite and a fine grain size of ferrite with scattered pearlite appeared in OC material, along with some hard segregated phases such as bainite and M/A constituents. It was reported that WQ resulted in increasing HIC susceptibility as a hard phase, i.e., bainite was formed [48].

Khalili et al. investigated the HIC susceptibility of electric resistant welded X52 pipe at different heat-treatment cycles as shown in Table 3 below.

Table 3: Heat Treatment Cycles used for investigating HIC susceptibility of electric resistant welded X52 pipe

Number of Cycles	Heat Treatment Condition
1 st	1000 °C for 20 seconds using induction heating process and water spray to 25°C
2 nd	1000°C for 20 min. and air cooled to 25°C
3 rd	1000°C for 20 seconds and the outer side was water quenched and tempered at 480°C for 20 seconds and air cooling to 25°C
4 th	1000°C and two sides were water quenched and tempered at 480°C for 20 seconds and air cooling at 25°C
5 th	1000°C for 20 seconds and the outer side water was quenched and heated again up to 1000°C for 20 seconds and the outer side water was quenched and tempered at 480°C for 20 seconds and air cooling for 25 °C

They have reported an improvement in ductility and impact toughness of the welded steel after the heat treatment. In fact, the second heat treatment cycle was found to be the best heat treatment process for improving the HIC resistance. It was recommended that postweld heat treatment be carried out to enhance the mechanical properties and the HIC resistance by two cycles of quenching followed by tempering [28]. Ramirez et al. investigated the effect of three different cooling rates after austenitization on API X80 pipe

in order to simulate the effect of welding. They have reported that the samples with inclusions and segregation zones showed low resistance to HIC. By contrast, the model with acicular ferrite and granular bainite exhibited high resistance to HIC [49].

For two hours, API X65 pipe was subjected to austenitizing heat treatment at 850 °C, 950 °C, 1050 °C, and 1150 °C by Mousavi et al. to investigate the impact of phase equilibria and microstructure on corrosion and HIC damage. Under a microscope, ferrite pearlite with islands of martensite/austenite elements was visible, while steels austenitized at higher temperatures had a lower percentage of pearlite.. They concluded that an increase in HIC damage with an increase in the quantity of pearlite. Based on the above discussion, it can be supposed that heat treatment can positively enhance the HIC resistance of carbon steel pipes but cannot permanently stop the HIC failure in the presence of inclusions or other hydrogen trapping sites. Quenching and tempering are the most suitable heat treatment processes for enhancing HIC susceptibility. Tempering at 500 – 600 °C is found beneficial to enhance HIC resistance for normal carbon steel materials. However, tempering acicular ferrite steels at 620°C for one hour is not recommended due to the formation of iron carbide at the grain boundary and this carbide acts as HIC crack initiation.

Furthermore, austenitization heat treatment is also found to be beneficial to increase HIC resistance [50]. Figure 29 summarizes the effects of heat treatment and its impact on HIC resistance.

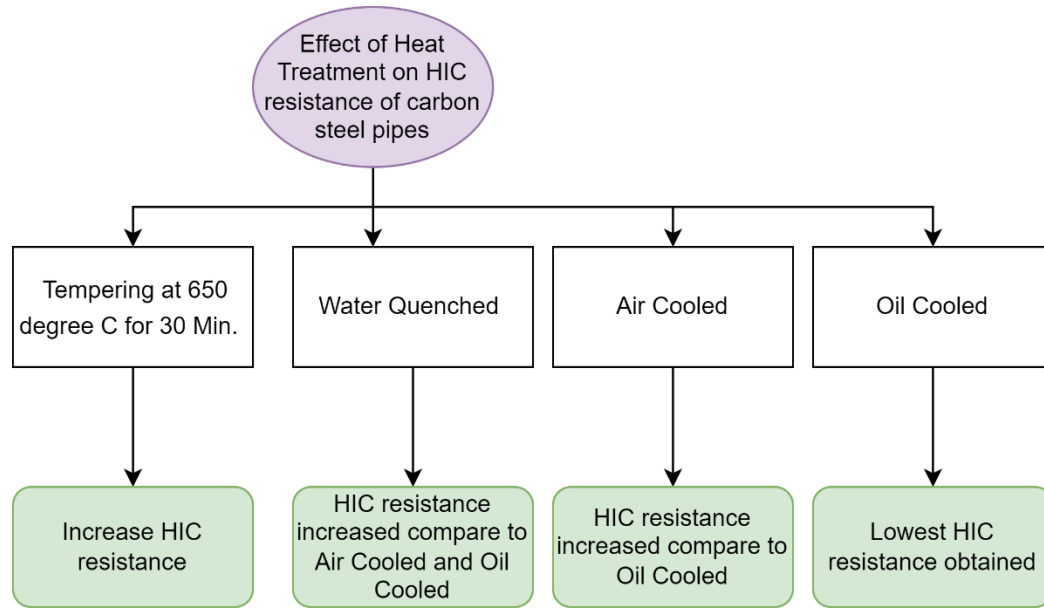


Figure 29: Effects of Heat Treatment on HIC resistance of Carbon Steel Pipes.

2.3.3 Alloying Elements

According to research by Liou et al., niobium (Nb), copper (Cu), chromium (Cr), molybdenum (Mo), and titanium (Ti) additions are important for both mechanical characteristics and HIC resistance in high-strength low-alloy steel (HSLA).. In addition, niobium (Nb) is added for precipitation hardening and grain refinement, and titanium (Ti) also provides grain refinement to the steel. Copper (Cu), chromium (Cr) and molybdenum are added to improve corrosion- resistance properties [51]. Table 4 highlights the importance of adding different alloying elements to the steel.

Table 4: Effects of alloying elements on HIC damage

S.No.	Alloying Element	Range	Remarks	References
1	Niobium	0 to 0.0073 wt.%	- Adversely impacts HIC resistance due to its ability to form MnS inclusions. - However, nanosized NbC precipitates were found to increase the HIC resistance because it works as an irreversible trap for hydrogen atoms.	[5,51,52]

S.No.	Alloying Element	Range	Remarks	References
2	Copper	0.29 wt.%	Copper increased the HIC resistance due to the formation of a black surface film known as Mackinawite (FeS_{1-x}) that works as a barrier preventing hydrogen atoms from diffusing into the steel.	[40,51]
3	Chromium	0.3 wt.%	- Increased Cr content to 0.3 wt.% will result in increasing the HIC resistance of steel. - It is discovered that Cr raises the A3 temperature, which lowers the Bs and Ms temperatures and postpones the ferrite and pearlite transition. - The microstructure often shifts from pearlite, ferrite, and trace quantities of bainite to a ferrite and bainite structure upon the addition of Cr.	[40,51]
4	Titanium	---	The addition of titanium will form rhombic TiN inclusions that are close to the effect of MnS inclusions, indirectly affecting the HIC resistance.	[40,51]
5	Carbon and manganese	Mn (1.8% wt.)	- Carbon and manganese attempt to segregate in solidifying steel, which results in forming a hard bainitic or martensitic microstructure. - The modified pipeline steel X80 with low manganese content (1.8%) performed better and reduced HIC susceptibility compared to commercial X80 products with Mn contents in the range of 1.00 to 1.20 % wt.	[40,52–54]
6	Vanadium	---	HIC damage resistance is increased by developing vanadium carbide precipitates.	[55]

The impact of micro alloying elements on HIC resistance was reviewed by Enyinnaya et al. According to reports, control over grain development during recrystallization and the austenite-ferrite transition results in grain refinement in micro alloyed steel. In pipeline steels, for example, Nb, Ti, and V are added as alloying elements to define microstructural grain boundaries and inhibit grain development. Additionally, adding these elements would result in eliminating C, N, S and P from the solid solution during hot rolling process. For

example, Titanium content in the reliable solution is decreased due to the presence of phosphorous by forming unwanted phosphides, which will result in lower amounts of carbide precipitation and thus will reduce the HIC resistance in high strength steels. According to reports, the elements Mo, V, Nb, Cr, and Ti—also referred to as nitride and carbide forming elements—tend to reduce the diffusion of hydrogen in the steel lattice because they form precipitates that help trap hydrogen and improve the mechanical properties of steel by strengthening it solidly. On the other hand, the addition of excessive amounts of Ni on pipeline steels can impact the resistance of cracking in a hydrogen sulfide environment due to the formation of martensite microstructure. Adding Cu as an alloying element for pipeline steels that operate in mild H₂S service environments has been found to lower hydrogen diffusion and corrosion rates. It is claimed that the addition of Cu on pipeline steel grade X120 increases the mechanical properties particularly strength due to the formation of Curich precipitates [5].

Based on the above discussion it can be concluded that alloying elements, mainly Mn, Mo, Cr, Ni, Nb, V, Ti, Cu etc., enhance the strength, toughness and hardness of steels and increase HIC resistance. However, an excess of these elements could react with other elements in the steel matrix and produce unwanted phases or inclusions which could act as an HIC crack initiation site.

2.3.4 Inclusions and Precipitates

As Figure 30 illustrates, inclusions have a significant impact on the HIC resistance of steel components used in sour service environments. The elongated MnS inclusions are reduced as a result of low calcium and sulfur concentrations. Mn is added to steels as a solid

solution-strengthening agent to stop the development of FeS particles, according to Mimoun Elboujdaini and Malcolm G. Hay. However, sulfur can combine with Mn to form MnS (type II and a soft inclusion), and it is considered the most unwanted inclusion in steels. MnS inclusions look like a pancake shape, which is elongated in the direction of working. On the other hand, Type I MnS inclusions solidify before the rest of molten steel and have a globular shape. Hence, Type I MnS inclusions are not that dangerous as compared to Type II inclusions. It is reported that sulfur content below 10 ppm and a calcium-sulfur ratio higher than 2 controls the shape of inclusions and decreases the susceptibility to HIC damage accordingly[35,56]. Moon et al. studied the impact of calcium addition and the Ca/S ratio on the HIC resistance of carbon steel pipes. They reported that CaS inclusions were more HIC-resistant than MnS inclusions. The reported reason was the shape difference between these two types of inclusions, spherical CaS vs elongated MnS. The spherical shape of CaS inclusions acted as a trapping site for hydrogen, and absorbed hydrogen is found distributed around the CaS inclusions. On the other hand, elongated MnS inclusions tend to concentrate the hydrogen atoms on the edges of the inclusions, which causes a drastic increase in HIC susceptibility. The Ca/S ratio expected to prevent HIC damage and produce spherical-shaped CaS inclusions should be higher than 1.25 [57].

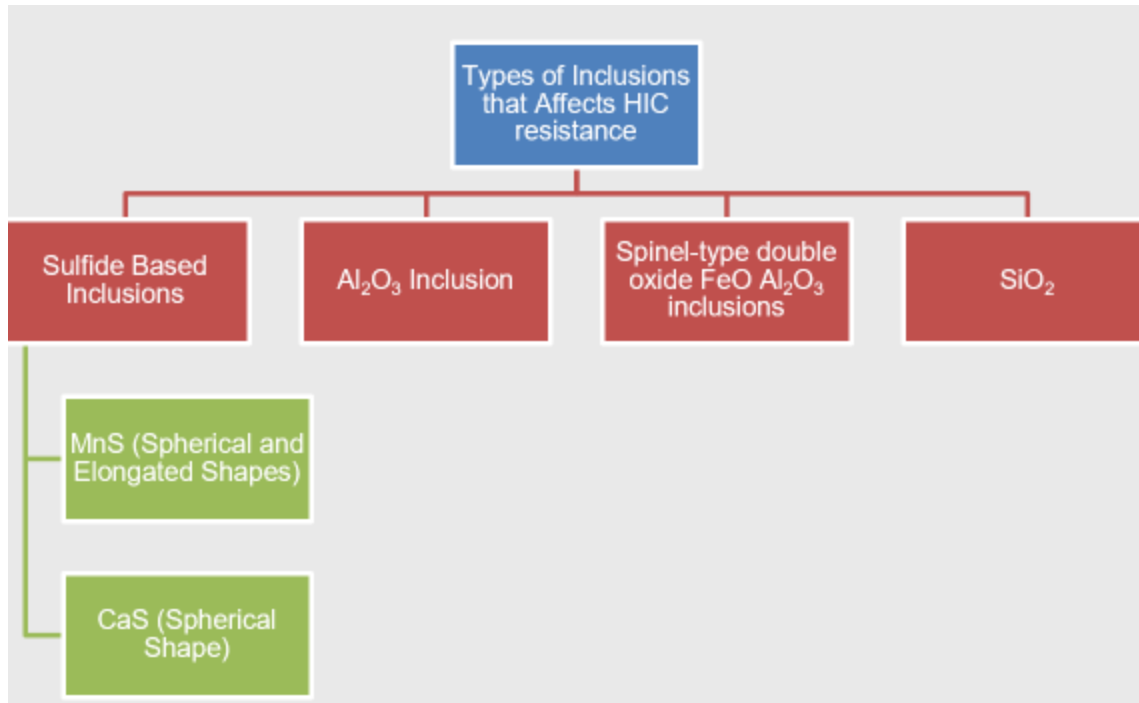


Figure 30: Types of inclusions that affects HIC resistance of steel

FF. Huang et al. studied the effect of microstructure and inclusion on X120 steel pipe. The microstructure of X120 steel contains lath bainite and granular bainite. The X120 steel was observed to have many inclusions consisting of Al_2O_3 or MnS and spinel-type double oxide $\text{FeO Al}_2\text{O}_3$ inclusions. These inclusions have a direct impact on the resistance to HIC damage [4,57]. It was concluded that the amount of inclusions found in X120 was responsible for decreasing its HIC resistance. MnS is a well-known inclusion that adversely impacts the steel from HIC damage. Other oxide inclusions did not show an adverse impact on the HIC resistance. However, several studies show that oxide inclusions like aluminum oxide initiated HIC damage [4,40]. Jack et al. looked at the X65 pipe's HIC behavior. It was discovered that the inclusions with a globular form affected the steel's mechanical qualities, including its strength and ductility. Si-enriched oxides and carbides were the inclusions found in the X65 pipe, and they contributed to the start of the HIC damage [58].

In conclusion, inclusion and precipitates are some of the major factors affecting HIC damage. Their presence in the steel matrix is not avoidable but can be controlled. In addition to the chemical composition of the inclusion, it is found that the size and shape are important to determine HIC resistance because these inclusions could link together with adjacent inclusions and result in through-thickness cracking. The most dangerous inclusion reported in the literature is the elongated MnS, where the HIC crack could catastrophically propagate and result in through-thickness cracking of steel. However, several inclusions like oxides-based and Si-rich oxides adversely impact the HIC susceptibility inside the steel matrix. Therefore, it is important to quantify and control the inclusion level in the steel matrix to enhance the HIC resistance.

2.3.5 Effect of Environmental Exposed Factors

Elboujdaini et al. reviewed the environmental factors that increase the susceptibility to HIC damage, which include pH, concentration of chloride ions, temperature and formation of a protective scale. It is clear from the Pourbaix diagram (potential vs pH) of the Fe-H₂O system Figure 31 that Fe is stable with the construction of a protective oxide layer, and hydrogen diffusion is not severe when the pH value is in the high range of 7 or more, and increases when exposed to low pH values [59].

pressure. In general, this type of material is usually used at high temperatures exceeding 100 °C and high hydrogen partial pressure. At this high temperature, HIC is not a real concern, and the most probable damage mechanism encountered at high temperatures and high hydrogen partial pressure is a high-temperature hydrogen attack [25].

2.4 Characteristics of HIC and Modelling

A mathematical model was created by Gonzalez et al. to forecast the rate of HIC fracture propagation in steel plates subjected to sour service. This model's basic idea is to quantify the extent of circular embedded fractures, which builds up hydrogen pressure and raises the stress intensity factor over the plane strain fracture toughness of the hydrogen-dissolved steel. As soon as fracture extension happens, the volume of the crack cavity increases and the internal pressure decreases, causing crack arrest. Then, the process will repeat once the crack cavity is filled with hydrogen. This model was verified with an experiment on X52 pipe operating in sour service [62].

A fracture-based model was created by Findley et al. to forecast the occurrence and spread of HIC cracking. They underlined that experimental data on the fracture toughness and subcritical crack propagation behavior of the pipeline microstructure in the presence of hydrogen are necessary for the fracture-based model [63]. Lei Fu et al. have investigated, using fracture mechanics, the development conditions of HIC damage in steel. According to the fracture mechanics hypothesis, the reduction of surface cohesion energy and plastic deformation energy at the interface by hydrogen atoms causes a decrease in fracture toughness. It is thought that during the expansion of fracture instability, the hydrogen in the material is split into two groups: 1) the hydrogen atoms around the crack, and 2) the

hydrogen molecules inside the crack cavity. The driving force behind fracture propagation caused by hydrogen gas pressure and the resistant force created by hydrogen atoms are the HIC damage creation processes [63].

Traidia et al. developed a mechanistic model that could conservatively predict the HIC damage growth rate based on the pre-existing crack growth rate estimation. ProHIC, a prediction software for the linear hydrogen-induced crack propagation rate in sour-service carbon steel pipes, is the name of the created program. According to their findings, the in-plane steel fracture toughness in a hydrogen environment and the hydrogen charge flux from the corrosion wall determine the HIC damage rate [64].

Dong et al. developed a microfracture shear band fractural model of HIC damage, and the mechanism of HIC damage was also studied. Based on the developed model, the HIC damage propagation is divided into three stages.

1. **Stage I:** The crack propagates poorly when $K_I > K_{ISCC}$. Currently, mechanical causes are the main influence; as K_I grows, da/dt increases quickly before gradually slowing down.
2. **Stage II:** Da/dt does not fluctuate with K_I ; that is, once K_I reaches a particular value, da/dt stays constant. For the time being, electrochemical factors are in charge.
3. **Stage III:** K_I keeps rising, and mechanical elements once more take center stage. K_{IC} rises with K_I , and the material breaks fast.

Where da/dt is the crack propagation rate, K_I stress intensity factor, K_{ISCC} is the HIC critical intensity factor, and K_{IC} is the fracture intensity factor [65].

In conclusion, the fracture model during the first stage is fractal intergranular cleavage fracture; during the second stage, it is intergranular and transgranular fracture; and during the third stage, it is both intergranular and transgranular mode.

Table 5 summaries the reviewed models related to HIC damage crack growth calculation and their uses and limitations.

Table 5: Summary of reported Models for HIC

S.No	Author	Basis of Model	Usage	Limitations
1	Gonzalez et al.	mathematical model	- Measure the extension of embedded circular cracks that result from the accumulation of hydrogen pressure. - The model was verified with an actual experiment	- The model does not take into account metallurgical conditions such as inclusion distribution along the crack plane, weak interface, grain boundaries...etc. - The model overestimated the crack growth rate after long-term exposure in a sour environment.
2	Findley et al.	fracture-based model	Predict HIC cracking and its propagation	It requires several experimental data on the fracture toughness and subcritical crack growth behavior of modern pipeline microstructure in the presence of hydrogen
3	Fu and Fang	Fracture Mechanics	It is used to study the formation criteria of HIC damage in steel. - The HIC damage formation mechanisms are both the driving force of crack propagation and the resisting force produced by hydrogen atoms. - The model was verified by actual experiments and finite element simulation.	It does not predict HIC crack grow.

S.No	Author	Basis of Model	Usage	Limitations
4	Traidia et al	Mechanistic Model	- It is used to predict the existing HIC damage growth rate. - The model was verified by actual field data.	It is not suitable to calculate new HIC damage.
5	Dong et al.	Fractural Model	- It is used to study a microfracture shear band fractural model of HIC damage. - The model is also used to study the mechanism of HIC damage.	The model does not account for slags, pores, and other defects in materials. The model assumes the chemical composition of the material is uniform and the material is isotropic on a microscopic scale.

2.5 Avoidance of HIC Failures

One of the best methodologies for preventing HIC damage is to select HIC-resistant materials for metallic components operating in a sour service regime. These types of steels are costly compared to non-HIC-resistant steels and are subjected to HIC testing by NACE TM 0284 prior to being operated in a sour service environment [33].

According to Elboudjaini et al., every steel has a unique H atom concentration critical or threshold value at which HIC damage begins. This threshold value is represented as $(C^H_O)_{TH}$ if the concentration of diffusing H atoms (C^H_O value) is calculated at the charging surface. The following should be taken into account in order to prevent HIC damage in new pipes:

- 1. Lower C^H_O , the surface concentration of diffusing H atoms.**

Lowering the C^H_O value can be achieved by adding corrosion inhibitor and adding alloying elements to the steel.

2. Raise $(C^H_O)_{TH}$, the threshold surface concentration of diffusing H atoms to initiate HIC.

Raising $(C^H_O)_{TH}$ can be achieved by the following

- Eliminating Type II MnS inclusions.
- Reducing the segregation of trace/impurity elements in the steel.
- Increasing the homogeneity of the microstructure.

Eliminating type II MnS inclusions can be carried out in three steps that include limiting the sulfur content to 0.003 wt.% as a maximum value and 0.001 wt.% as a minimum value, specifying the manganese content to be in the range of 1.00 wt.% and 0.80 wt.%, and controlling the MnS inclusion shape [35].

Additionally, the only suitable protection method is the application of a protective coating or cladding to eliminate or prevent hydrogen atoms from penetrating inside the steel surface [66]. However, this coating only lasts for a short time and requires regular maintenance. In addition, it is difficult to coat small-size diameters, which makes the application of coating difficult for small-size pipes.

2.6 Conclusion

To sum up, HIC occurs on steel components that operate in wet H₂S environments. It appears due to cathodic corrosion between the steel and the service environment, which produces hydrogen atoms. The produced hydrogen atoms then become trapped in reversible and irreversible sites and form hydrogen molecules. The hydrogen molecules

create huge pressure inside the steel and tend to initiate cracks. Several factors affect HIC susceptibility, including metallurgical factors and environment-related factors. Inspection and monitoring of the HIC damage can be achieved by applying manual and advanced NDT techniques. Lowering the threshold surface concentration of diffusing H atoms that started HIC and increasing the threshold surface concentration of diffusing H atoms that start HIC are two ways to prevent HIC harm.

CHAPTER 3

EXPERIMENTAL METHODS

This chapter shall discuss the research methodologies to investigate the root cause(s) of failing HIC testing as manufactured piping and passing the test as a raw material; NACE TM 0284 is considered a major testing to be conducted. The research shall be carried out in various steps, including the following subsections: Testing Materials and Sample Details, Screening Testing, Heat Treatment, HIC Testing, Electrochemical Hydrogen Charging, Grain Size and Inclusion Content Measurements, and Scanning Electron Microscopy (SEM) & Energy Dispersive X-Ray Analysis (EDX). Figure 32 shows the testing plan for the thesis in details including location of test and quantity.

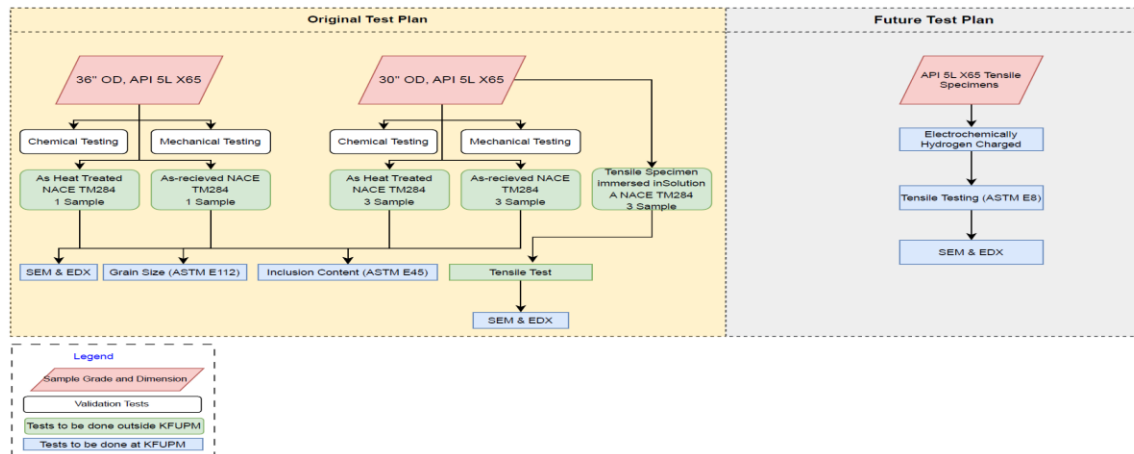


Figure 32: Thesis Testing Plan

3.1 Testing Materials and Samples Details

The targeted material is API 5L Grade X65 manufactured by a thermomechanical controlled process. The raw material, which plates or coils passed the HIC testing criteria

at the steel mill and in the third-party laboratory. However, it failed the HIC test after being manufactured as a pipe product. Two pipes with the same manufacturing process and grade (Gr. API 5L X65) were selected to be investigated, but the suppliers were different. Two sets of HIC samples were collected from one pipe, and six (6) sets of HIC samples were collected from the other pipes supplied from different sources. In total, eight (8) groups of HIC samples were examined. The sample preparation followed the requirement of NACE TM 0284, where three pieces were extracted from the pipe ring, as shown in Figure 33.

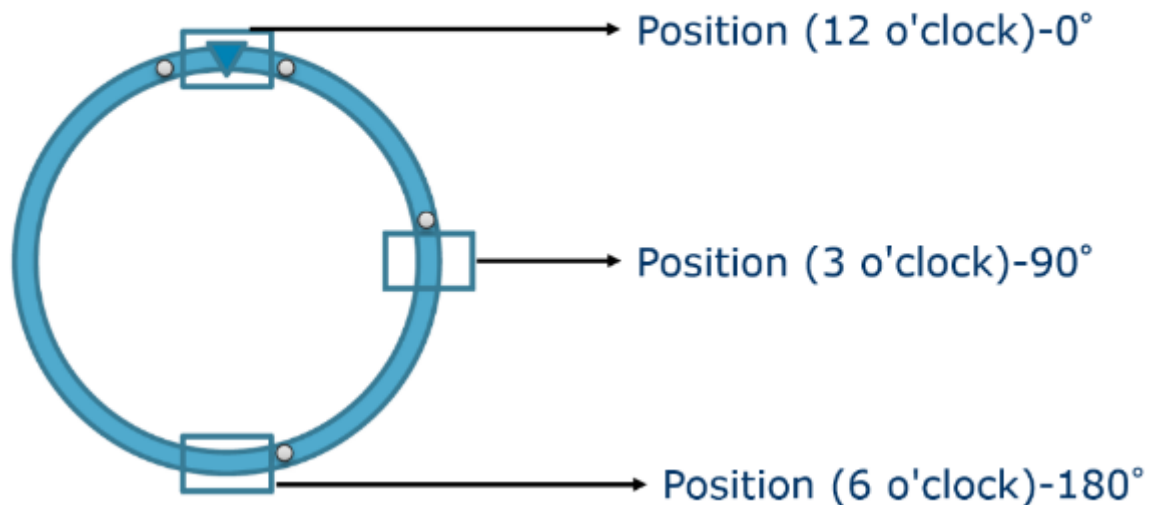


Figure 33: Location of HIC specimens from pipe ring.
The samples shall be prepared in accordance with NACE TM 0284.

Additionally, three tensile specimens were prepared in order to be immersed in the NACE Solution A.

Finally, three tensile specimens were prepared to be electrochemically charged with hydrogen and measure the strength of the materials after charging.

3.2 Screening Testing

The main objective from this testing is to evaluate the quality of the received materials. Therefore, chemical analysis, tensile testing and hardness testing were carried out.

3.3 Heat Treatment

The failed pipes were exposed to a cold expansion technique to remove residual stresses associated with the piping manufacturing process. However, the lines failed HIC testing after relieving residual stress by the hard expansion technique. Therefore, it is proposed to reduce the residual stresses by performing the Post Weld Heat Treatment (PWHT) technique following the general guidelines in ASME B31.3 for heat treatment of process piping. This standard allows the heat treatment process up to 650 °C for carbon steel materials. The heat treatment furnace was manufactured by Carbolite Gero with a maximum temperature of 1300 °C, Figure 34.

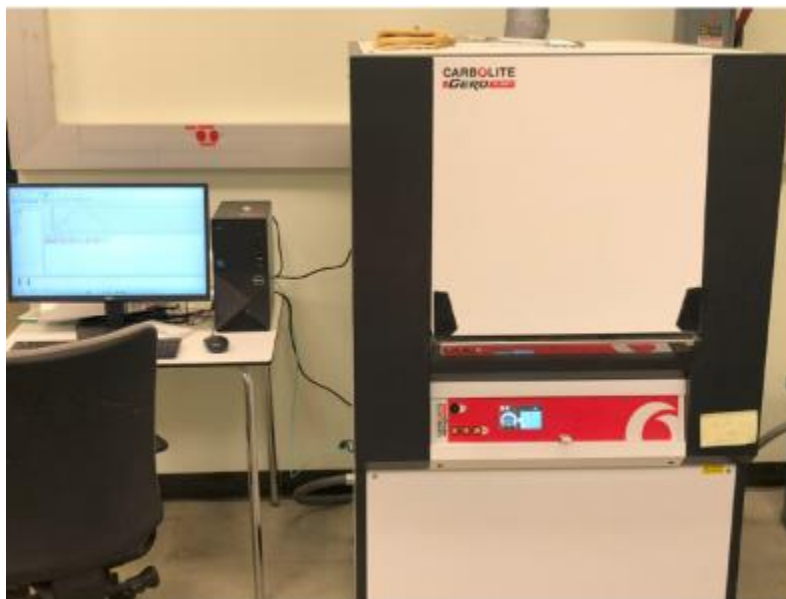


Figure 34: Heat Treatment Furnace

The heat treatment was carried out in accordance with ASME B31.3 for carbon steel material. The soaking time for heat treatment was 1 hour, and the heating rate was 0.33°C per minute. The maximum increase temperature for one hour shall not exceed 200 °C. The soaking temperatures were 650, 600, 550, and 500 °C for samples A, B, C, and D, respectively. Figure 35 shows the heat treatment cycle for these samples.

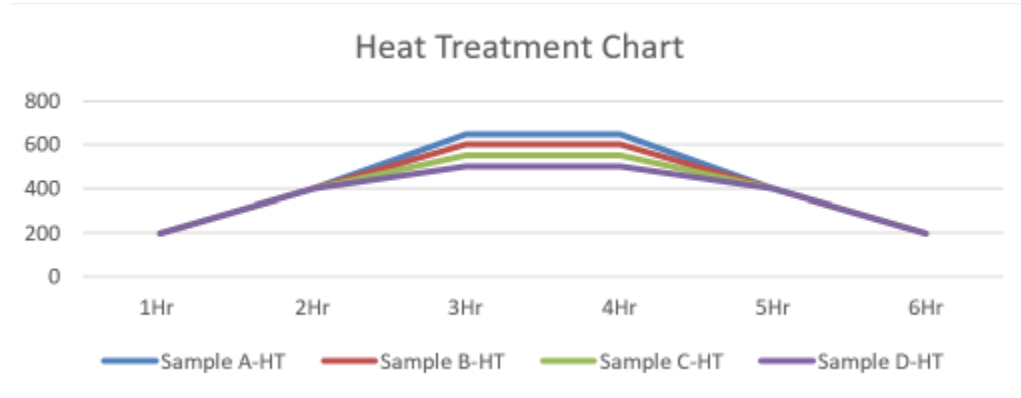


Figure 35: Heat Treatment Cycle for Samples A, B, C and D.

3.4 HIC Testing (NACE TM 0284)

This test requires safety preservations due to the presence of H₂S gas. Hence, NACE TM 0284 was carried out at one of our local piping mill manufacturers, which is Global Pipe Company, located at Jubail Industrial City. HIC testing (NACE TM 0284) shall be carried out on eight (8) samples (4 samples in the received condition and four samples after heat treatment).

3.4.1 Sample Preparation

Eight (8) samples were collected from two pipes that were manufactured by different raw materials suppliers. The first and second samples were fabricated by A supplier, and the remaining pieces were supplied by the manufacturer or supplier B. Each HIC sample has

three (3) sets of specimens extracted from zero, 90, and 180 degrees, as shown in Figure 33. Each specimen shall have 100 mm long and 20 mm wide, Figure 36.



Figure 36: Set of HIC specimens

For the welded location, the specimen shall be extracted in accordance with Figure 37 to capture the welded area and heat-affected zone.

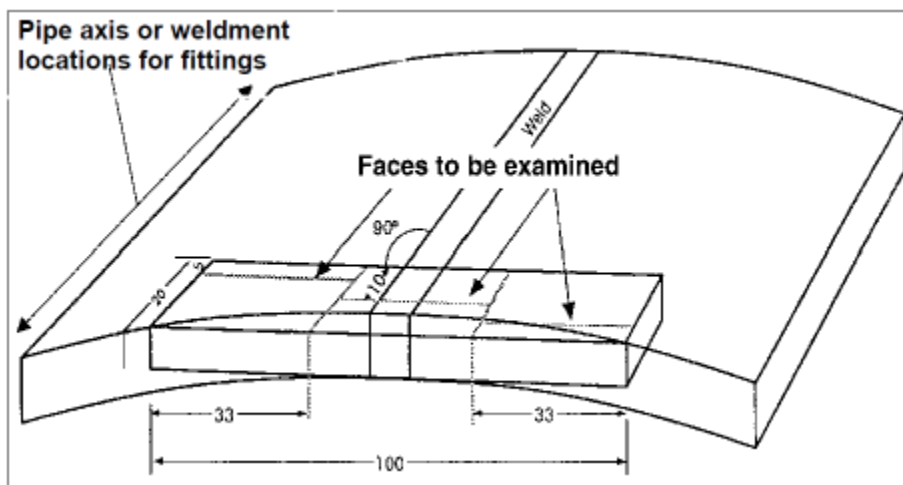


Figure 37: Specimen extracted from Zero degree (welded location).

On the other hand, the specimen from 90 and 180 degrees shall be considered a seamless specimen, as show in Figure 38.

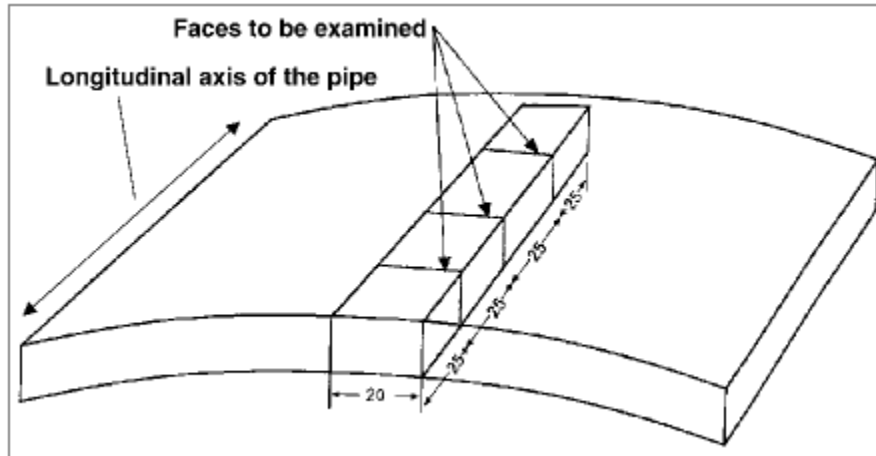


Figure 38: Specimens extracted from 90 and 180 degrees.

Additionally, three tensile specimens, as per ASTM A370, were prepared to be immersed in the NACE solution A to examine the effect after exposure to the NACE solution. The specimens were too small to be placed on a testing vessel, Figure 39.



Figure 39: Tensile Test Specimens prior to immersing in the NACE solution A.

Thereafter, the tensile test specimens shall be subjected to welding after immersion in order to be fitted in the tensile test device, Figure 40.

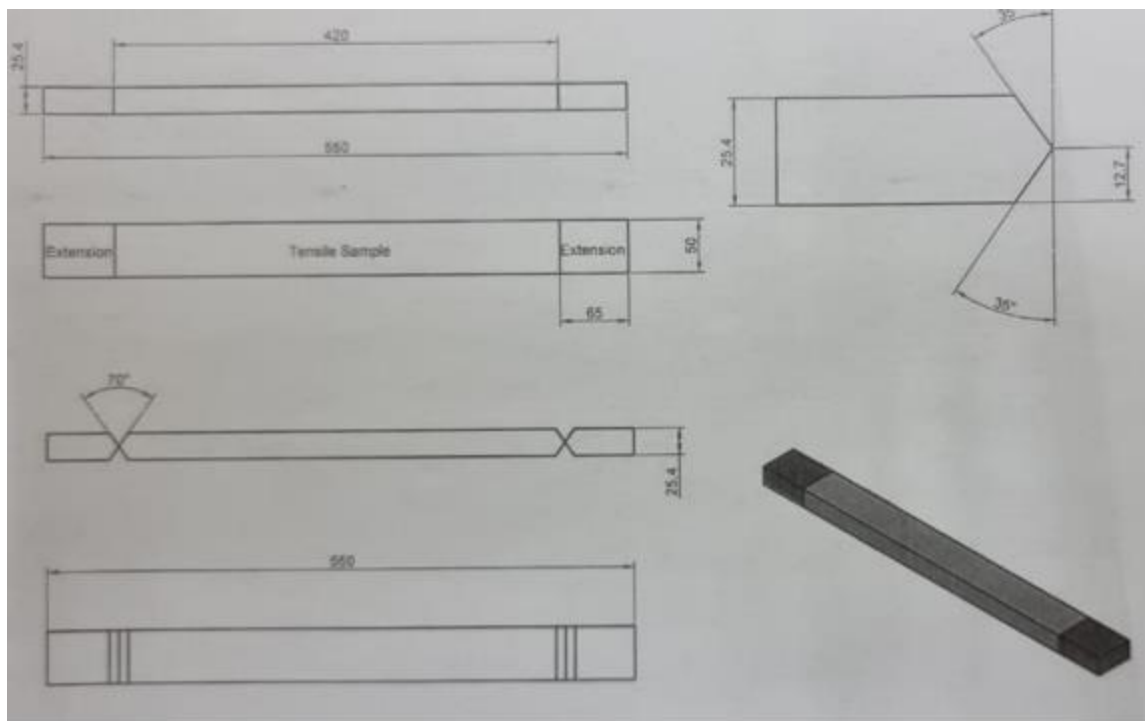


Figure 40: Welding arrangement for tensile testing specimens after immersion test takes place.

3.4.2 Solution Preparation

The test shall be carried out with NACE Test solution A. Solution A shall be an inert gas (nitrogen, argon, or other suitable non-reactive gas) for purging, H₂S gas, NaCl, CH₃COOH, and distilled or deionized water. The solution was prepared in the solution supplier contained in Figure 41. Table 6 below shows the chemical arrangement and H₂S calculation.



Figure 41: Solution supplier container.

Table 6: NACE Solution A preparation

	Solution						pH	Temp.	PPM req.
Titration	A (Iodine 0.1N) ml	HCl ml	C Test Solution ml	Starch ml	B $\text{Na}_2\text{S}_2\text{O}_3$ 0.1N) ml	PPM	4.0 max.	22 ~ 28 °C	Min.
1	25	5	10	2	11	2385.6	3.79	25.2	2300

The PPM, which the actual H₂S content, is calculated in accordance with the equation below.

$$PPM = \frac{(A - B)}{C} * 17040$$

Multiply A and B by 0.1, which is the Normality of Solutions. (3.1)

According to NACE TM 0284, the ration of the volume for the solution to the total surface area of the specimens shall be 3ml per cm². The surface area is calculation based on the equation below.

$$\text{Surface area (cm}^2\text{): } [(Length \times Width \times 2) + (Length \times Thickness \times 2) + (Width \times Thickness \times 2)] / 100 \quad (3.2)$$

The total calculated surface area is 2520.74 cm², and the complete solution volume is 7562.23 ml. Hence, the computed ratio of full solution volume = 3.97 ml/cm².

3.4.3 Apparatus

Figure 18 shows a typical device for HIC testing by NACE TM 0284. However, the standard testing equipment conducted at Global Pipe Company is shown in Figure 42.

The samples were placed with a wide vertical face and separated from the test vessel and another specimen by glass or other non-metallic road with a diameter of 6 mm, Figure 43.

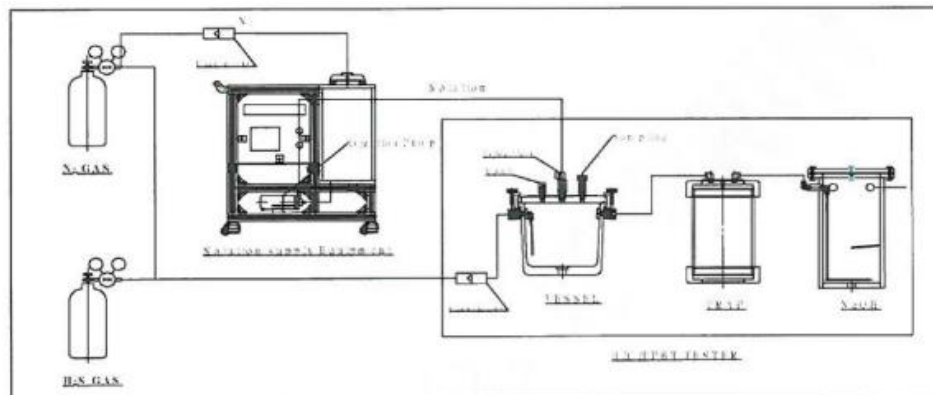


Figure 42: Testing apparatus at Global Pipe Company

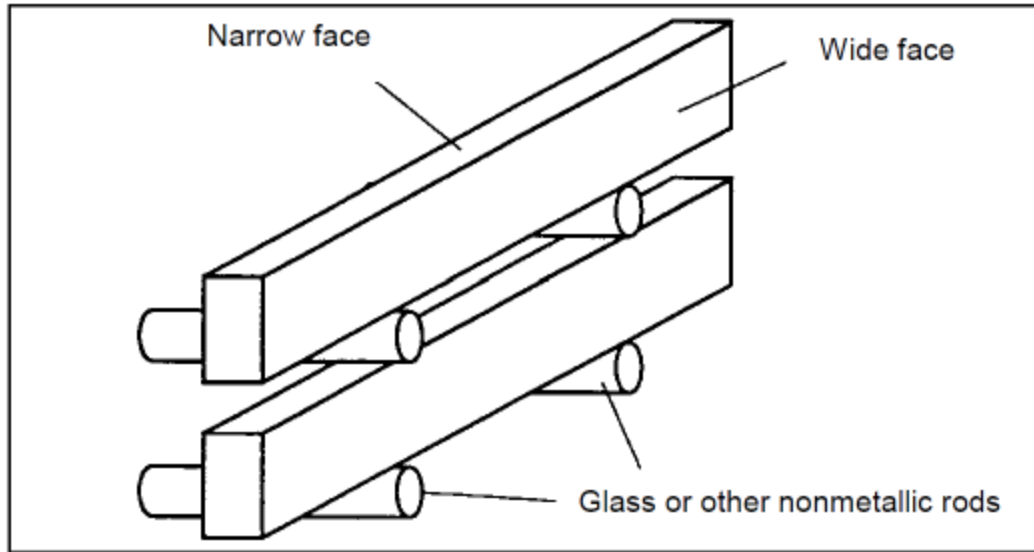


Figure 43: Orientation of specimens inside testing vessels

3.5 Electrochemical Hydrogen Charging

Three (3) tensile testing specimens shall be electrochemically charged in an acidic solution to investigate the impact of hydrogen penetration on the material properties. Then, tensile testing shall be carried out by ASTM E8 and evaluate the fracture surface on the SEM and EDX techniques. The electrochemical hydrogen charging cell consists of an AC power supply, cathode, anode, counter electrode (platinum mesh), and electrolyte (solution), Figure 44. The solution was from 0.2 M sulfuric acid (H_2SO_4) and three g/L ammonium thiocyanate (NH_4SCN). The current density varies between 20, 15, and 10 mA/cm^2 . The charging times were 10, 24 and 36 Hrs. The surface area of the charged portion from tensile specimens was 8.468 cm^2 .

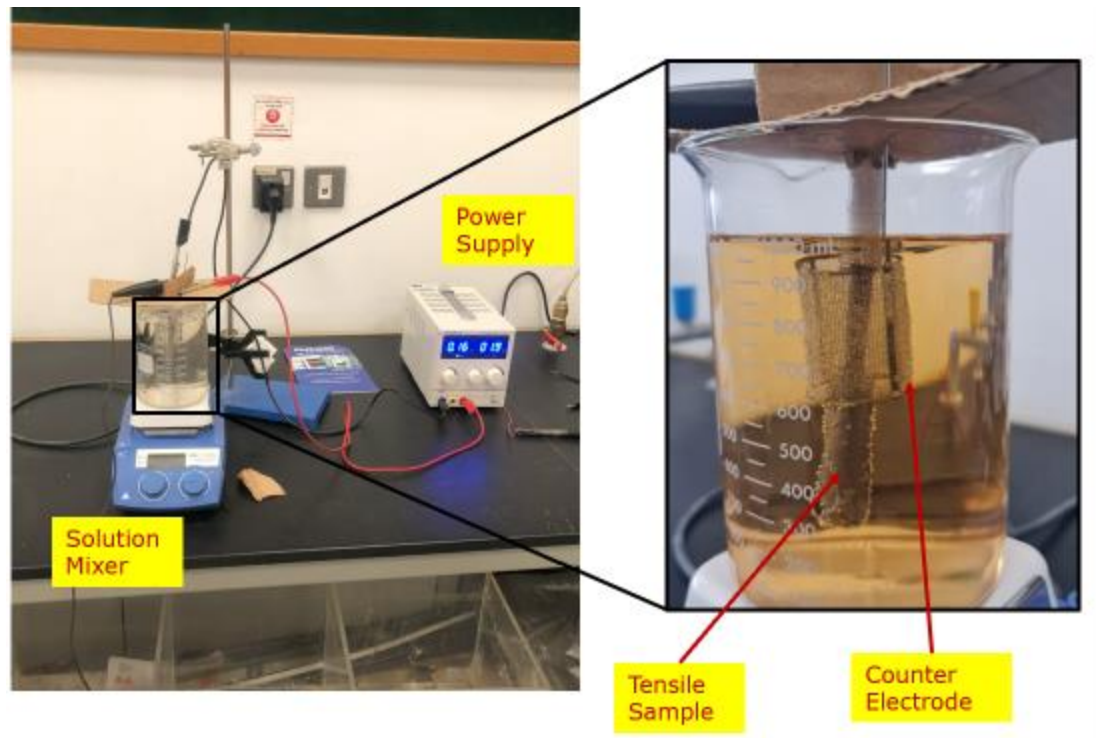


Figure 44: Electrochemical Hydrogen Charging Cell.

3.6 Grain Size and Inclusion Content Measurement

The heat-treated and non-heat-treated specimens shall be subjected to grain size measurement and inclusion content measurement as per ASTM E45 and ASTM E112, respectively, using Olympos Optical Microscope DP74 that is associated with Olympos stream for image analysis, Figure 45.



Figure 45: Olympus Optical Microscope DP74

The Olympus Stream Image Analysis Software is equipped to evaluate the grain size and inclusion content from the photo. The following sections shall elaborate on the main operation function of these tests.

3.6.1 Grain Size Measurement (Planimetric Method)

The specimens were sectioned, mounted, ground, polished, and etched with 2% Nital. Then, install the instance on the microscope and draw a circle of known area (usually 5000 mm² to simplify the calculation). The selected magnification is 100X. However, other magnifications can be chosen subject that. The magnification shall provide 50 grains in the field to be counted within this area. The number of equivalent whole grains is equal to the sum of all grains included in the known area plus one-half the number of grains intersected by the circumference of the site. Thereafter, the number of equivalent whole grains is

multiplied by Jeffries' number to get the number of grains per square millimeter N_A by the equation below. The Jeffries' number is obtained from table 5 of ASTM E112 for each magnification, Figure 46.

$$N_A = F (N_{Inside} + N_{Intersected}/2) \quad (3.3)$$

TABLE 5 Relationship Between Magnification Used and Jeffries' Multiplier, f , for an Area of 5000 mm² (a Circle of 79.8-mm Diameter) ($f = 0.0002 M^2$)

Magnification Used, M	Jeffries' Multiplier, f , to Obtain Grains/mm ²
1	0.0002
10	0.02
25	0.125
50	0.5
75 ^A	1.125
100	2.0
150	4.5
200	8.0
250	12.5
300	18.0
500	50.0
750	112.5
1000	200.0

^A At 75 diameters magnification, Jeffries' multiplier, f , becomes unity if the area used is 5625 mm² (a circle of 84.5-mm diameter).

Figure 46: Photo of table 5 from ASTM E112 Jeffries' Number Multiplier factor

3.6.2 Inclusion Content Measurement (Worst Field Method A)

The specimens should be in a polished condition and obtained with 100X magnification. The inclusions are classified into four types, which are Sulfide (A- Elongated light grey inclusions), Alumina (B- Elongated but consist of globular oxides), Silicate (C- Elongated dark grey inclusions), and Globular Oxide (D- Globular inclusions). These types are categorized into heavy or thin to describe their thickness or number. Then, the four types are separated into Severity levels based on the length or number of particles

present in a 50 mm². Olympus stream image analysis shall scan the targeted area and determine the severity level of the inclusion. Then, the length and number of particles shall be determined from table 1 of ASTM E45, Figure 47.

**TABLE 1 Minimum Values for Severity Level Numbers
(Methods A, D, and E)^{A,B}**

Severity	(mm (in.) at 100x, or count)			
	A	B	C	D ^C
0.5	3.7(0.15)	1.7(0.07)	1.8(0.07)	1
1.0	12.7(0.50)	7.7(0.30)	7.6(0.30)	4
1.5	26.1(1.03)	18.4(0.72)	17.6(0.69)	9
2.0	43.6(1.72)	34.3(1.35)	32.0(1.26)	16
2.5	64.9(2.56)	55.5(2.19)	51.0(2.01)	25
3.0	89.8(3.54)	82.2(3.24)	74.6(2.94)	36
3.5	118.1(4.65)	114.7(4.52)	102.9(4.05)	49
4.0	149.8(5.90)	153.0(6.02)	135.9(5.35)	64
4.5	189.8(7.47)	197.3(7.77)	173.7(6.84)	81
5.0	223.0(8.78)	247.6(9.75)	216.3(8.52)	100

Figure 47: Photo of table 1 from ASTM E45 where length and number of each type of inclusion is determined based on the severity level

3.7 Scanning Electron Microscopy (SEM) & Energy Dispersive X-Ray

Analysis (EDX)

SEM and EDX analyses shall be carried out on all eight (8) samples exposed to the HIC testing environment, Figure 48. The main objective of SEM & EDX is to characterize the cracked area and analyze the affected locations. Additionally, the fracture surface of tensile specimens of immersed samples in solution A and electrochemically charged specimens shall be examined using SEM.



Figure 48: **Field Emission Scanning Electron Microscope (ZIESS-Gemini 1).**

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Screening Testing

The chemical analysis performed using spark emission spectroscopy results revealed that samples A and B conform to the chemical requirement of API 5L Gr. X65, Table 7.

Table 7: Chemical analysis results of Samples A and B

S. N	C	Si	Mn	P	S	Al	Cr	Ni	Mo	Cu	V	Nb	Ti	N	B	Ca
	Wt. %															
A	0.030	0.336	1.56	0.009	0.00014	0.042	0.178	0.013	0.005	0.01	0.003	0.043	0.013	0.006	0.0002	0.0019
B	0.023	0.325	1.468	0.008	0.0005	0.021	0.165	0.013	0.006	0.015	0.004	0.03	0.013	0.008	0.000	0.002
API 5L Gr. X65	Max. (Wt. %)															
	0.12	0.45	1.60	0.025	0.015	--	≤ 0.50	≤ 0.50	≤ 0.50	≤ 0.50	Nb + V + Ti ≤ 0.15 %.			--	--	--

The tensile test conducted on received samples A & B conformed to the technical requirement of API 5L Gr. X65, Table 8.

Chemical and mechanical properties of any material is a significantly important characteristic for selecting a proper material of intended applications. Therefore, API SPEC 5L specified acceptance criteria for each pipe grades in terms of mechanical and chemical requirement. The conducted chemical analysis that carried out on samples A and B using spark emission spectroscopy revealed the compliance with chemical requirement of API SPEC 5L Table 5 Gr. X65. Carbon (C), Silicon (Si), Manganese (Mn), Phosphors (P), Sulfur (S), Vanadium (V), Niobium (Nb), Titanium (Ti) are considered as a major elements that required to be controlled in order to quantify the quality of each grade. The chemical composition of Samples A and B conforms with the

chemical composition of Gr. X65. Hence, this test result indicates that the material is suitable for the intended surface.

Table 8: Tensile Strength Test results of received samples A & B.

Name	Base Metal						
	Sample	Fracture	YS	TS	Load	YR	EL
	I.D.	Location	MPa	MPa	kN	YS/TS	%
Specimen A	TBT	Base	501	604	480.37	0.83	50.02
Specimen B	TBT	Base	509	569	480.37	0.89	59.42
API 5L Gr. (X65)	--	--	Min. (450) Max. (600)	Min. (535) Max. (760)		Max. (0.93)	Min. (26 %)

Furthermore, the mechanical properties such as tensile strength and hardness of materials play a vital role in the material selection and evaluating the material suitability for the intended applications. It can give assurance that the selected materials can withstand the expected strength and load that could be encountered during normal operation. Hence, API SPEC 5L specified minimum and maximum yield strength for Gr. X65 which are 450 MPa and 600 MPa, respectively. This value determines the maximum load that can be anticipated prior to yielding. Additionally, API SPEC 5L specified the minimum and maximum tensile strength which are 535 MPa and 760 MPa, respectively. The yield strength of the sample A and B are 501 MPa and 509 MPa, respectively. These values fall within the acceptable limit of the API SPEC Gr. X65. It should be highlighted that the elongation of tensile specimen is a critical factor for selecting and accepting the materials. According to API SPEC 5L, the elongation percentage is calculated for this pipe diameter 30 inches to be 26% minimum. However, the test results of elongation for both samples A and B demonstrated that the materials are more ductile with elongation values 50.02% and 59.42%, respectively. This indicates that the ductility of Sample A

and B exceeded the minimum required value stipulated in API SPEC 5L calculation method.

The hardness testing values conform to the technical requirement of API 5L Gr. X65 and NACE MR 0175/ISO 15156, Table 9. It is below 250 HV as required by NACE.

Table 9: Hardness results of Sample A & B

Sample ID	Indentation	Test Value (HV)	Required Value
Sample A	Base	216	Less than 248 HV
	HAZ	173	
	Weld	210	
Sample B	Base	232	
	HAZ	197	
	Weld	210	

The hardness value is a crucial parameter particularly for sour service environment. It determines the resistance of sulfide stress cracking for the selected material. Accordingly, API SPEC 5L specify special annex (Annex H) for mandatory requirement for sour service. In this annex, the maximum hardness value that mandated by API 5L on base metal, heat affected zone and welded area shall not exceed 250 HV. Also, NACE MR1075 / ISO 15156 “Standard Materials Requirements Sulfide Stress Corrosion Cracking Resistance Metallic Materials for Oilfield Equipment” mandates that the hardness value shall not be higher than 248 HV. The hardness testing results for both samples A and B showed acceptable result and conforming with the technical requirement of both API SPEC 5L and NACE MR 0175/ISO 15156. The hardness values for base metals, heat affected zone and welded area for both samples were satisfactory.

Based on the aforementioned facts and findings, it can therefore be concluded that the Samples A and B conforms to the technical acceptable criteria for API SPEC 5L for Gr. X65 and can be utilized for sour services environment.

4.2 Heat Treatment

The heat treatment application is one of the proven methodologies that are extensively used in the industrial application to relief the imposed stresses on the materials. It can be used in some instances to improve the mechanical and microstructural properties of the materials. It is believed that the imposed stresses during welding application of LSAW pipes exaggerated the HIC susceptibility by imposing some reversible hydrogen trapes during manufacturing processes. These pipe that manufactured using JCOE generally goes for mechanical expansion during manufacturing to remove the stresses. However, the mechanical expansion sometimes does not provide sufficient removal of residual stresses. Hence, it was proposed in this research study to investigate the effect of heat treatment/ stress relief and measure whether or not the HIC properties will be improved. The available heat treatment process for piping is American Society of Mechanical Engineers (ASME B31.3 Process Piping). This standard provides the maximum allowable temperature for conducting post weld heat treatment for carbon steel materials as 595 to 650 °C for 1 hours per one inch. This temperature level and duration was selected to ensure that the microstructure and mechanical properties of the material shall not be dropped or impacted. However, the soaking temperature, which is the maximum temperature achieved and hold for one hour, in this research where varied to measure the effect of amending the temperature. The HIC specimens A, B, C and D where subjected to various heat treatment temperatures 650°C, 600°C, 550°C and 500°C, respectively, and hold for one hour. The application of stress relief generally does not impose any changes on the microstructure and mechanical properties of the materials because it is

involving an increase in the temperature below critical point (A_1) for certain period of time. Figure 49 shows the microstructure of the as received and heat treated samples at 650 °C. The microstructure of the as received and heat treated samples shows sign of ferrite pearlite microstructure with more level of ferrite compare to pearlite phase. There was no major changes in the microstructure as a result from applied heat treatment/stress relief application. There was slightly increase in the grain size as shown in Figure 49. Similarly, a study of the microstructure of carbon steel material process piping that was manufactured in accordance of the requirement of ASME SA106 grade B. The microstructure was ferrite and pearlite microstructure and heat treated in accordance with ASME VIII, DIV I, UCS-56 for 30 min as the thickness was 13 mm. The microstructure of the received and the heat treated material was almost similar to each other [67]. To conclude the discussion on heat treatment, the applied heat treatment was meant to relief the residual stresses utilizing the most referenced industrial standard for oil and gas industries which is ASME B31.3. the selected temperature and duration shall not impose adverse impact on the microstructure as well as mechanical properties of carbon steel material. The selected temperatures are below critical temperature which shall not alter the microstructure compare to normalizing and other heat treatment process. The only observed changes in the microstructure was slight increase in the grain size which is obviously expected as a result for exposure to high temperature for a certain period of time.

A) As Received



B) Heat Treated at 650 °C

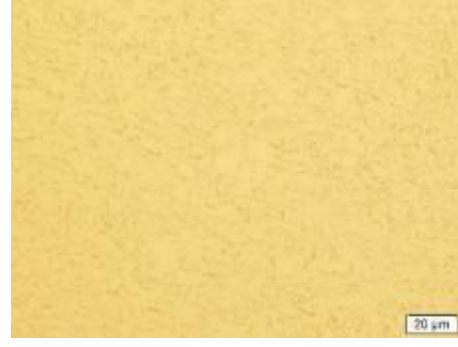


Figure 49: Microstructure of the as received TMCP material and heat treated at 650 °C.

A) As Received



B) Heat Treated at 595 °C

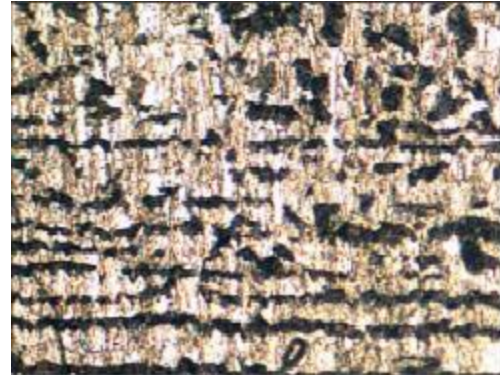


Figure 50: Reported microstructure of carbon steel ASME Process Piping SA106 Grade B in the as received and heat treated conditions [67].

4.3 HIC Testing (NACE TM 0284)

After immersion of the test specimens in NACE solution A, the Crack Length Ratio (CLR), Crack Thickness Ratio (CTR), and Crack Sensitivity Ratio (CSR) were calculated in accordance with the equations below.

$$CLR = \frac{\sum a}{W} \times 100\% \quad (4.1)$$

$$CSR = \frac{\sum (a \times b)}{(W \times T)} \times 100\% \quad (4.2)$$

$$CTR = \frac{\sum b}{T} \times 100\% \quad (4.3)$$

Where: a is crack length, b is crack thickness, W is section width and T is test specimen thickness and can be measured in accordance with Figure 51.

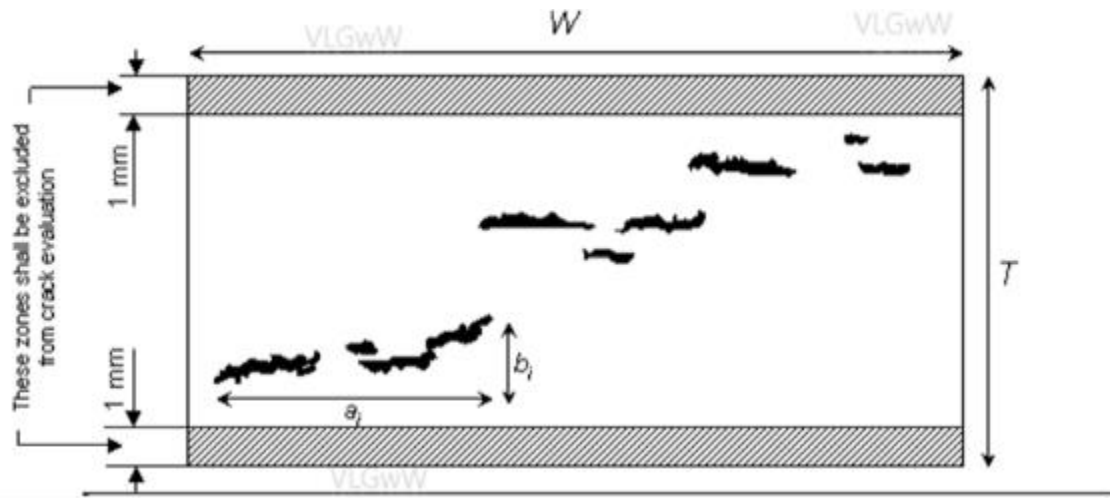


Figure 51: Crack Length and Thickness measurement method.

4.3.1 Calculation Method of Zero (0) Degree or Welded Area

After the immersion in solution, all the zero degree samples shall be sectioned in to three section (Section 1, Section 2 and Section 3) to examine the internal surface of each section, Figure 52.

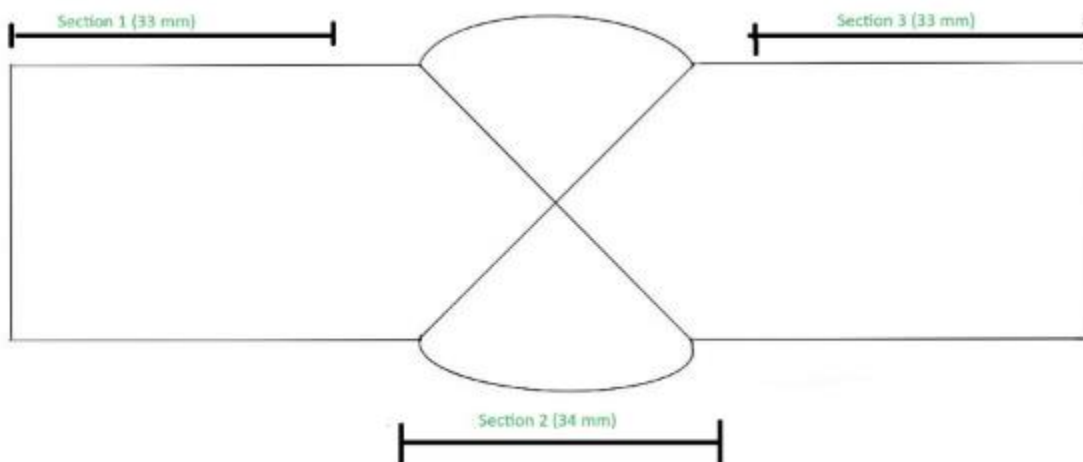


Figure 52: Sections location of Zero degree samples.

Then, the CLR, CTR and CSR shall be determined based on the number of cracks presented on each section. At the end, the average of each CLR, CTR and CSR shall be calculated based on the same equations shown above.

4.3.2 Calculation Method of 90 and 180 Degrees or Base metal Area

After the immersion in solution, all the zero-degree samples shall be sectioned into three sections (Section 1, Section 2, and Section 3) to examine the internal surface of each section, Figure 53.

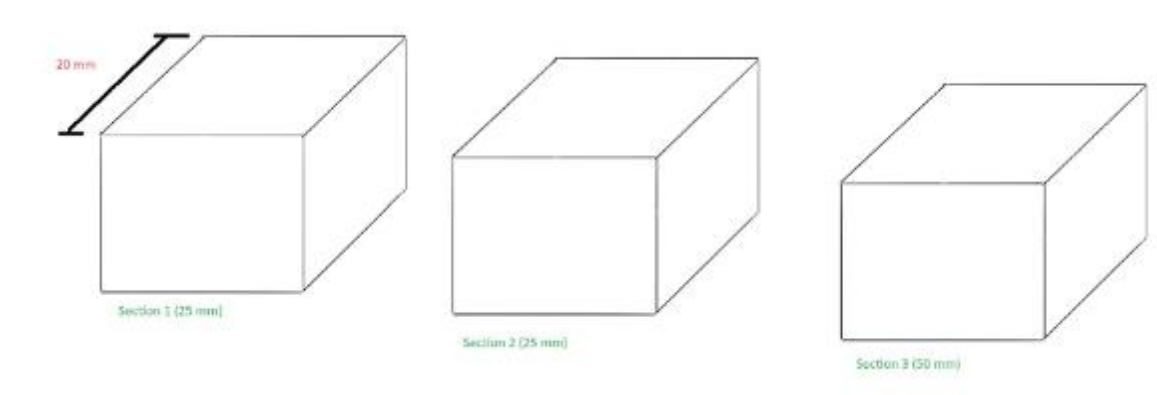


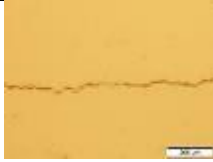
Figure 53: Configuration of sections for 90 and 180 degree samples.

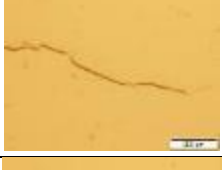
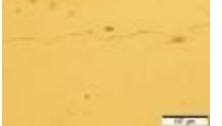
Then, the CLR, CTR and CSR shall be calculated for each section using optical microscope. Thereafter, the average CLR, CTR and CSR shall be calculated in accordance with the equations above.

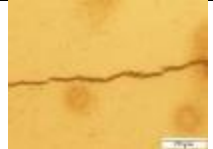
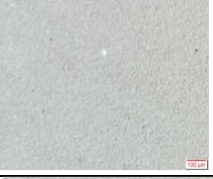
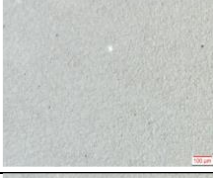
4.3.3 Compiled results for HIC testing

Table 10 shows the calculated average CLR, CTR and CSR for all the eight (8) Samples.

Table 10: HIC testing results for eight (8) samples

S/ N	Sample ID		Orientat ion	Avg %CLR (10%max)	Avg %CTR (3%max)	Avg %CSR (2%max)	Status	Optical Microscope Image at 100X magnification
1	Sample A	Sample A-0 (1-3)	0°	16.79	3.45	1.01	FAIL	
		Sample A-90 (1-3)	90°	0.00	0.00	0.00		
		Sample A-180 (1-3)	180°	0.00	0.00	0.00		
2	Sample A-HT 650°C	Sample A-HT-0 (1-3)	0°-2	0.00	0.00	0.00	PASS	
		Sample A-HT-90 (1-3)	90°-2	0.00	0.00	0.00		
		Sample A-HT-180 (1-3)	180°-2	0.00	0.00	0.00		
3	Sample B	Sample B-0 (1-3)	0°	19.43	4.53	1.46	FAIL	
		Sample B-90 (1-3)	90°	7.47	2.70	0.38		

S/ N	Sample ID	Orientation	Avg %CLR (10%max)	Avg %CTR (3%max)	Avg %CSR (2%max)	Status	Optical Microscope Image at 100X magnification	
		Sample B-180 (1-3)	180°	6.29	3.38	0.64		
4	Sample B-HT 600°C	Sample B-HT-0 (1-3)	0°-F2	0.00	0.00	0.00	PASS	
		Sample B-HT-90 (1-3)	90°-F2	0.00	0.00	0.00		
		Sample B-HT-180 (1-3)	180°-F2	5.45	0.23	0.04		
5	Sample C	Sample C-0 (1-3)	0°	16.91	5.21	1.32	FAIL	
		Sample C-90 (1-3)	90°	11.88	2.62	0.46		
		Sample C-180 (1-3)	180°	5.18	0.65	0.10		
6	Sample C-HT 550°C	Sample C-HT-0 (1-3)	0°-M2	0.00	0.00	0.00	PASS	
		Sample C-HT-90 (1-3)	90°-M2	5.80	0.32	0.03		

S/ N	Sample ID		Orientat ion	Avg %CLR (10%max)	Avg %CTR (3%max)	Avg %CSR (2%max)	Status	Optical Microscope Image at 100X magnification	
		Sample C-HT- 180 (1-3)	180°-M2	0.00	0.00	0.00			
7	Sample D	Sample D-0 (1-3)	0°	15.34	2.19	0.52	FAIL		
		Sample D-90 (1- 3)	90°	0.00	0.00	0.00			
		Sample D-180 (1-3)	180°	0.00	0.00	0.00			
8	Sample D-HT 500°C	Sample D-HT-0 (1-3)	0°-T2	0.00	0.00	0.00	PASS		
		Sample D-HT- 90 (1-3)	90°-T2	0.00	0.00	0.00			
		Sample D-HT- 180 (1-3)	180°-T2	0.36	0.01	0.00			

All HIC samples including heat treated specimens and non-heat treated specimens were immersed in NACE solution A. Thereafter, each HIC specimen was subjected to sectioning/cutting to three sections in order to measure the CLR, CTR and CSR for each section. The CLR, CTR and CSR are measured using optical microscope to measure the value accurately. Then, the average value for these three sections were calculated and

considered for evaluation either being accepted or rejected. This statistical method is a common recommended practice and recommended by NACE TM 0284. The targeted acceptance level for the CLR, CTR and CSR are 10%, 3% and 2%, respectively. If the tested specimen fails one of these criteria, the entire set of samples will be considered as a rejected result.

The average values of CLR, CTR, and CSR for the eight samples that were subjected to HIC testing are presented in Table 10. The HIC testing results indicate that the heat treated specimens passed the HIC testing. On the other hand, the non-heat treated HIC samples failed the HIC testing. It can be highlighted that the failure of HIC testing is always encountered in the welded area which referenced here as zero degree. However, there are some cracks observed on base metals as well particularly on HIC Samples that extracted from steel supplier B. This finding is in line with the published literature data where TMCP Pipe of API 5L Gr. X65 experienced HIC failure after exposure to hydrogen environment as reported by Tonye Alaso Jack et al [4]. They conducted electrochemical hydrogen charging to simulate NACE HIC testing TM0284 on TMCP API 5L Gr. X65 Plate. The reported average length, width and longest cracks are 2053 μm , 7 μm and 2413 μm [4]. On the other hand, T. Hara et al [55] carried out HIC testing in accordance with NACE TM 0284 on TMCP API 5L Gr. X65 pipe manufactured BY LSAW using UOE technology. The calculated Crack Area Ratio, as claimed in the published article for CLR, CSR & CTR, for all the TMCP pipes did not show sign of HIC damage. This findings is in line with our test results for majority of non-heat treated samples particularly for 90° and 180° location that extracted from the base metal. Furthermore, a recent study was carried out by Thomas et al [68] on TMCP API 5L Gr.

X65 plates that were stressed to simulate the expected stresses that will be produced during pipe forming process of LSAW using UOE technology. The result of Crack Area Ratio, which is based on application of Ultrasonic technique prior to conduct the metallurgical calculation post immersion in NACE solution, varies depending on the wall thickness and diameter ratio of the pipe. They reported that pipe forming process could impact the HIC resistance properties particularly when the Wall Thickness (t) and Diameter (d) ratio is above 5%. However, the wall thickness and diameter of the pipes utilized in our experiment are 20.62 mm and 914.4 mm, respectively, which gives t/d 2.2 % [68]. Therefore, it is really difficult to compare the NACE test results with published data because the manufacturing process is some time different, which make some changes in the reversible and irreversible trap sites, and the testing method is different as some authors used different NACE solutions. As a rule of thumb, it can be expected that TMCP API 5L Gr. X65 could experience HIC failure when it is formed as a pipe regardless of whether the plate is HIC resistance material or not [69]. Our results of HIC Testing utilizing NACE Solution A provides evidence that the forming process of pipes imposed reversible hydrogen traps on the HIC resistance steel materials which further be supported by latest experiment by Thomas et al [68]. Evidently, the HIC failures were encountered on the zero degree location which are exposed to welding and excessive bending stresses during forming process of the pipe.

The applied heat treatment was only stress relieving process which requires an increase in the temperature below critical temperature A_1 and hold it for certain period of time to get a homogeneous microstructure and cool it down to ambient temperature either in the furnace or in room atmosphere [4]. It is reported that the application of stress relieving

and post weld heat treatment is always imposing positive results on enhancing the HIC resistance. The so called tempering treatment or stress relieving or post weld heat treatment tends to reduce the dislocation density, hardness and residual stresses which subsequently enhance the HIC resistance of the material [47]. This claim is validated even with the conducted HIC testing on heat treated and non-heat treated samples in NACE TM 0284 Solution A. The results showed significant improvement in terms of CLR, CTR and CSR for the heat treated samples. However, the claim that stress relieving enhances the HIC resistance for pipelines material was criticized by the same authors. They have conducted stress relieving treatment on TMCP API 5L Gr. X70 pipe and evaluated the HIC resistance. They found that acicular ferrite microstructure are susceptible to HIC cracking even post application of stress relieving treatment [47]. The microstructure of the examined material was ferrite pearlite not acicular ferrite as shown in Figure 54.



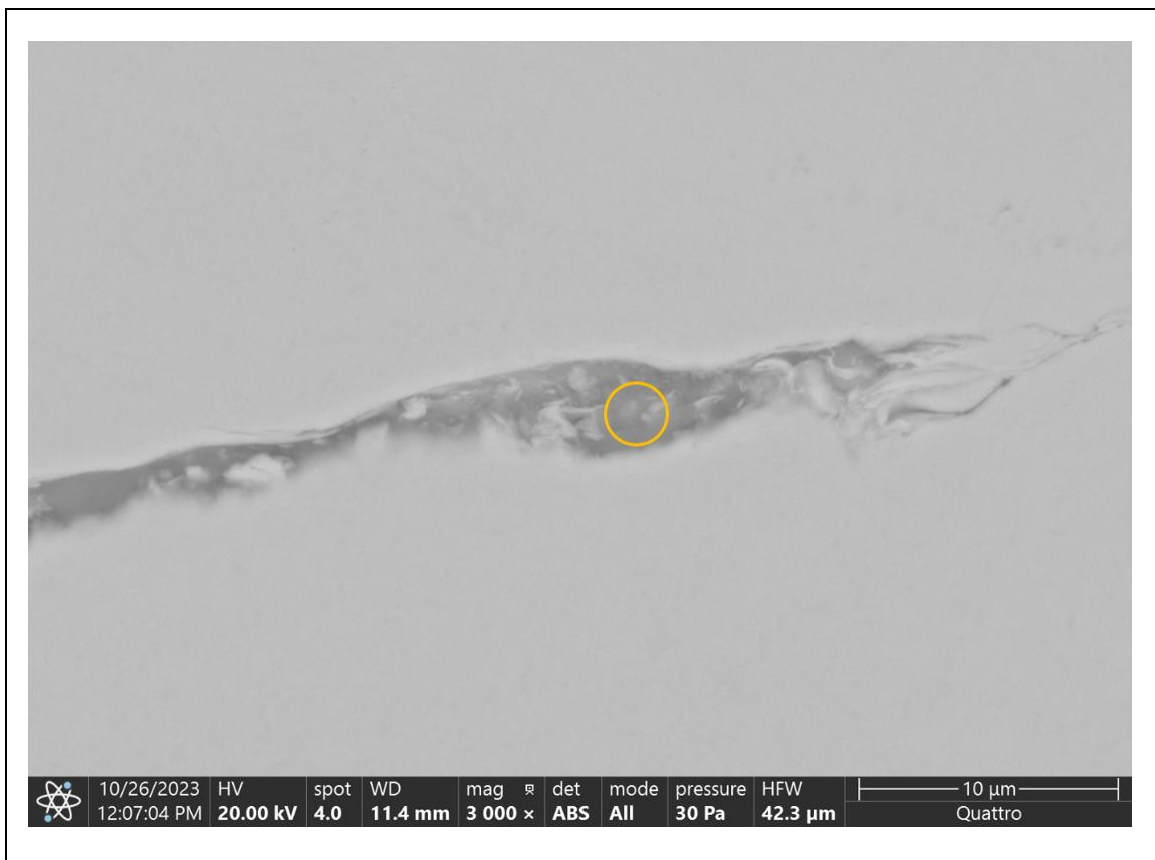
Figure 54: Microstructure of as received sample which shows ferrite pearlite microstructure (5% Nital etched. 500X magnification).

The results of the research support the literature that post weld stress relieving will result in minimizing the effect of reversible hydrogen traps (mainly dislocation density) and reducing the micro-residual stresses imposed during forming and welding processes of the pipe. The results of the research also concluded that cold expansion alone will not minimize the effect of reversible hydrogen traps and will also not reduce the high micro residual stresses in the base metal adjacent to the weld area. This explains the improvement in HIC results subsequent to post weld stress relieving compared to the results of specimens extracted from only cold expanded pipes. It is clear that JCO forming process develops high micro-residual stresses in the base metal area adjacent to the weld area and specifically in the area uncovered by the pre-bending process.

Additionally, the final shape of pipe forming and peaking angle will play vital role in developing high micro-residual stresses that can not be minimized by applying only cold expansion.

4.3.4 Fractography Results for NACE TM 0284

The SEM & EDX analyses of inclusions around HIC crack indicated presence of Ca, traces of Mn, Oxide, Si and Al, Figure 55-58.



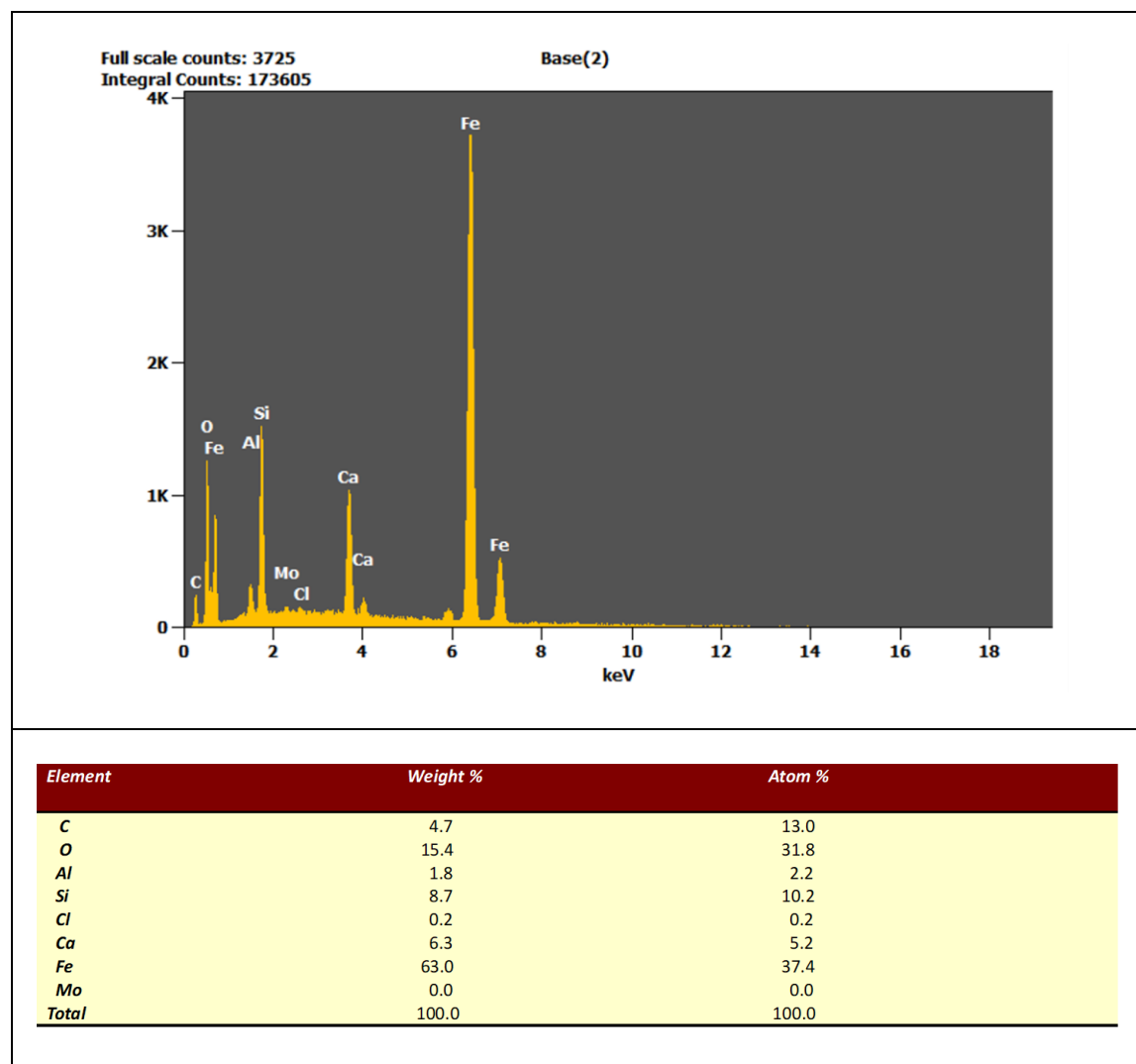
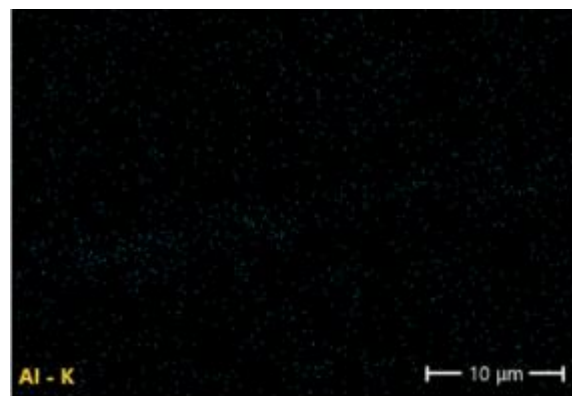
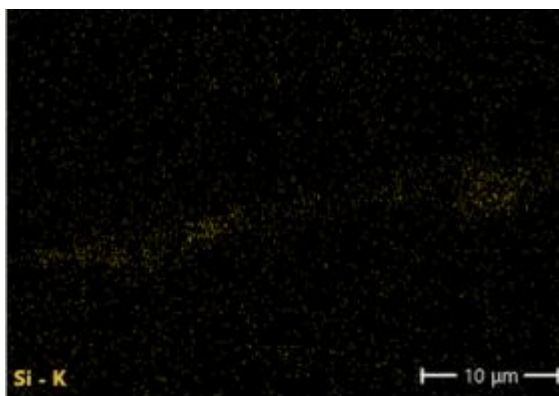
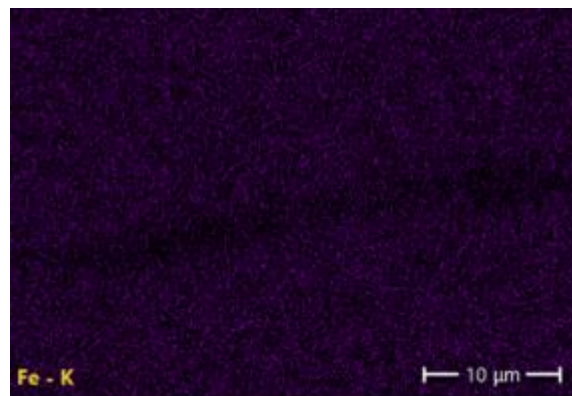
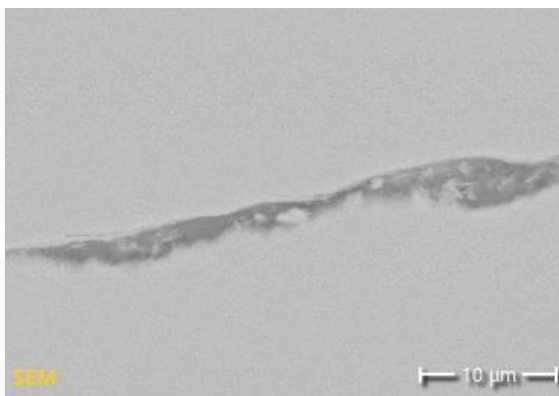
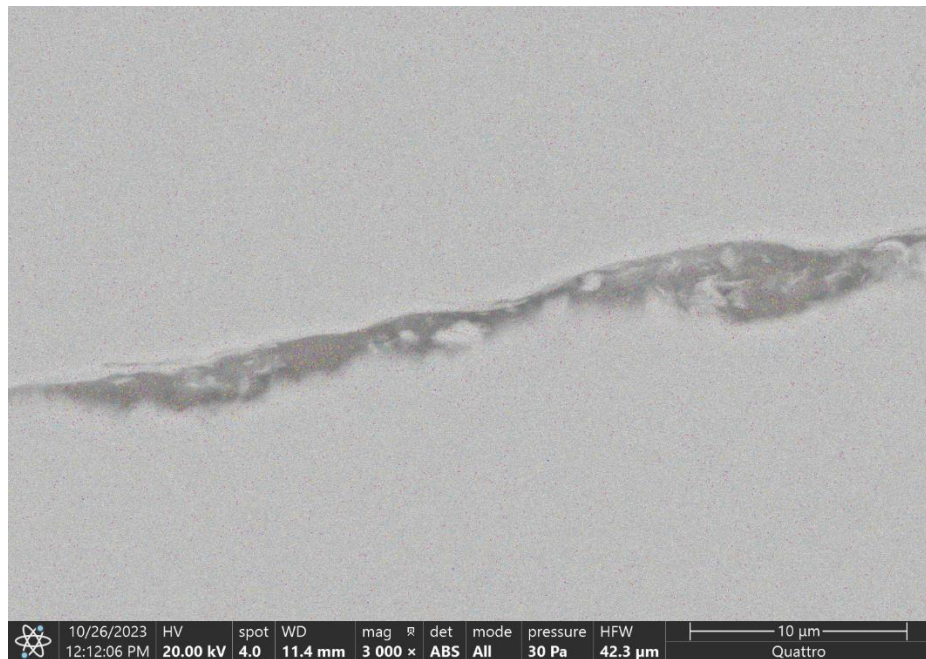


Figure 55: SEM & EDX analyses results for HIC cracked specimens.



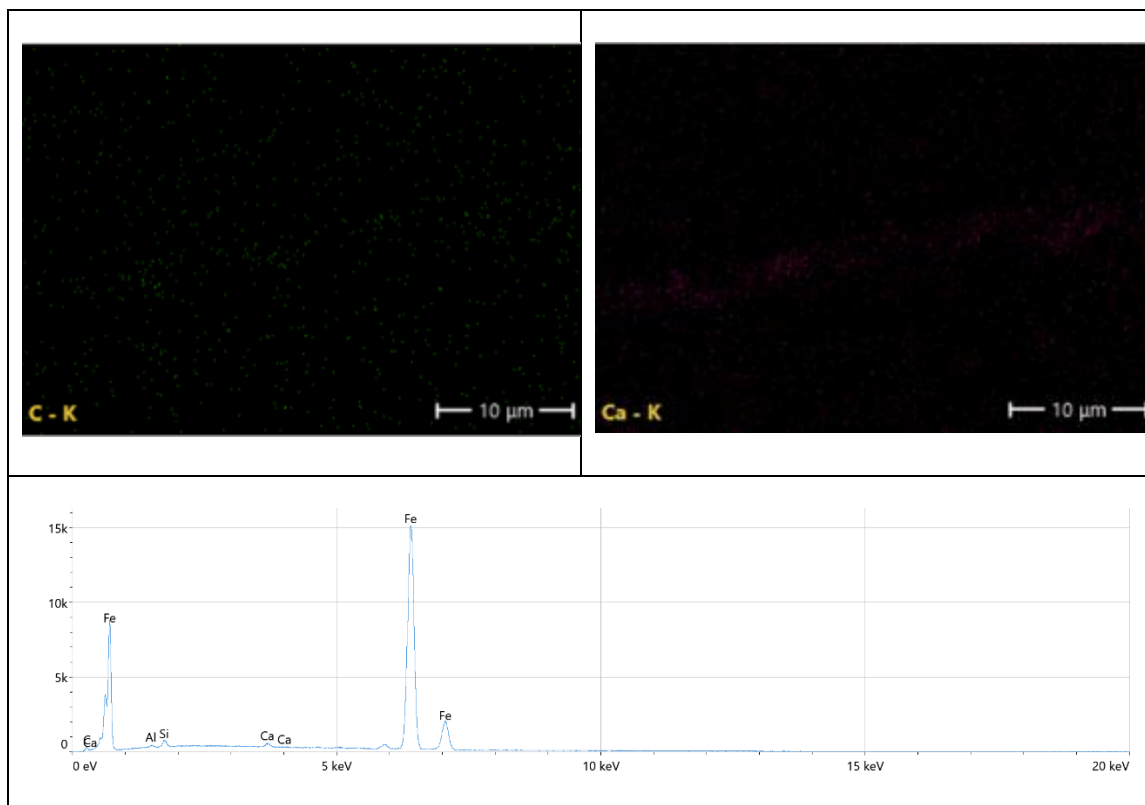
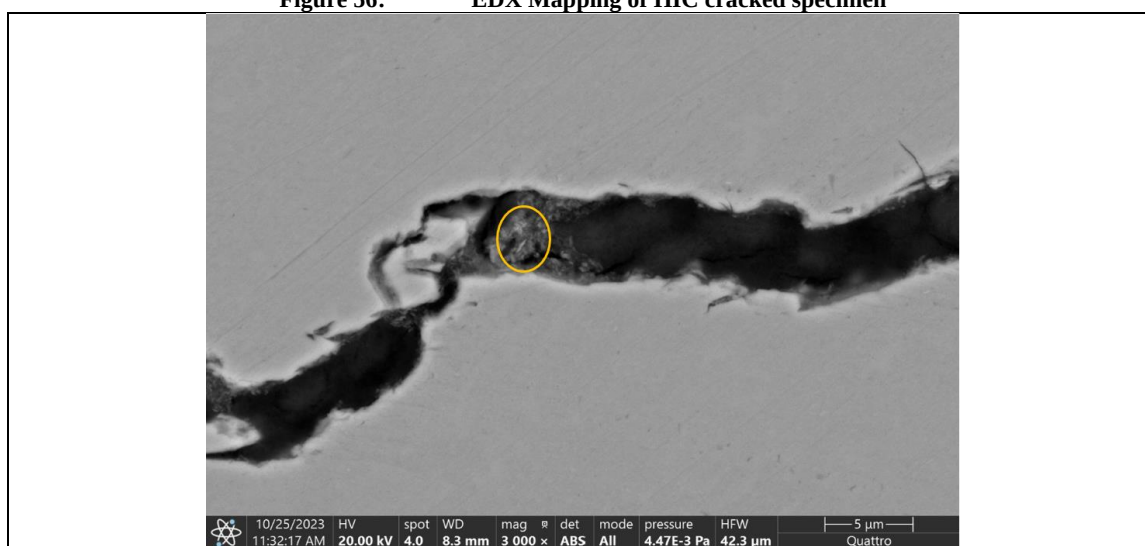


Figure 56: EDX Mapping of HIC cracked specimen



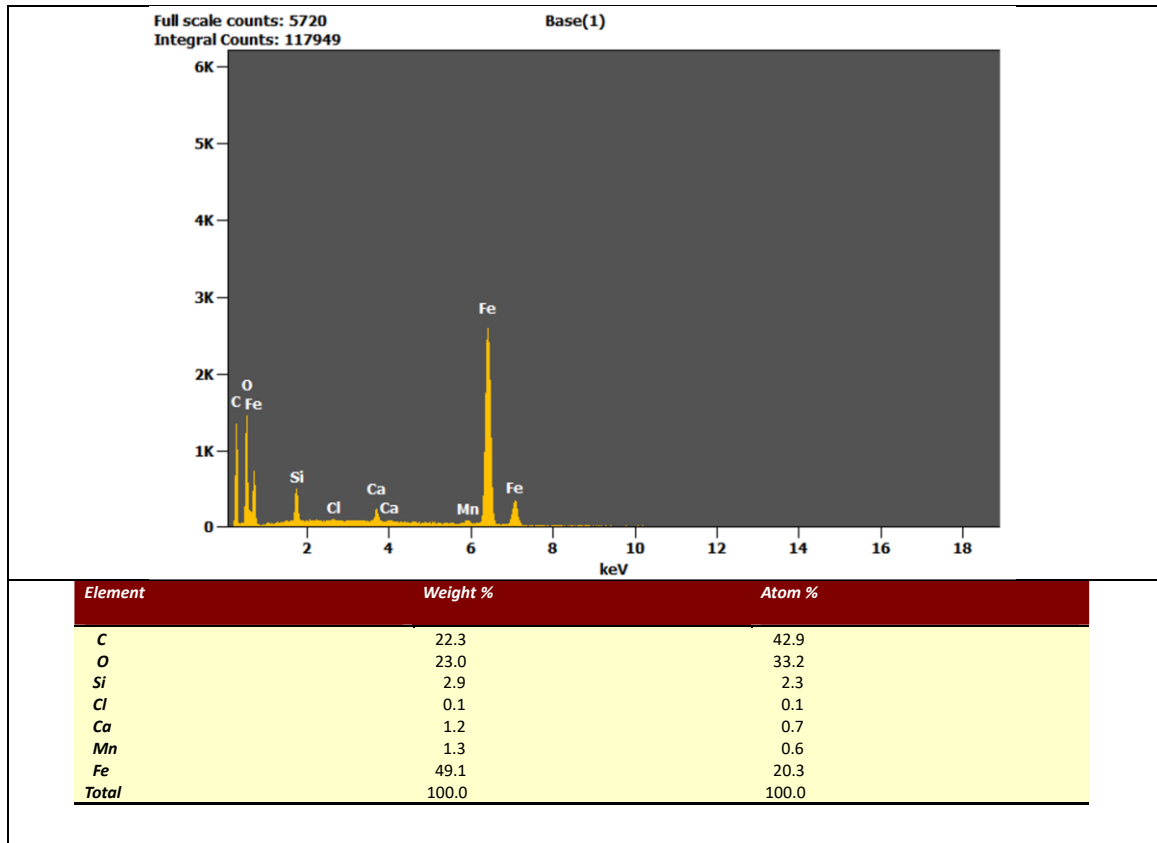
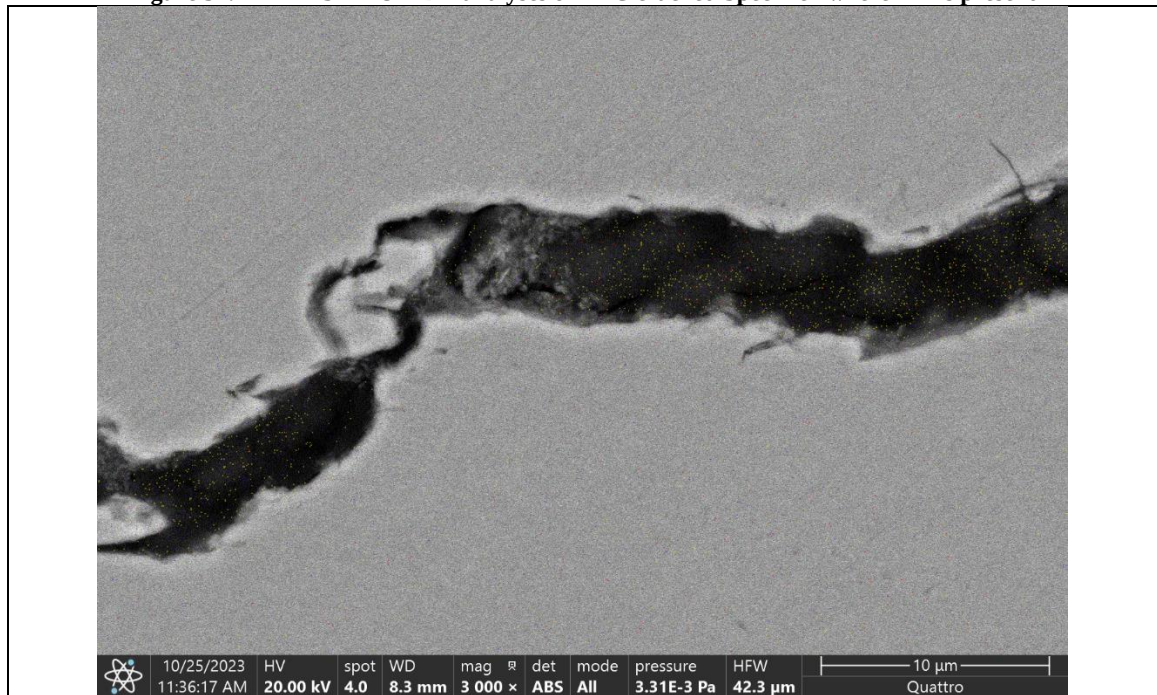
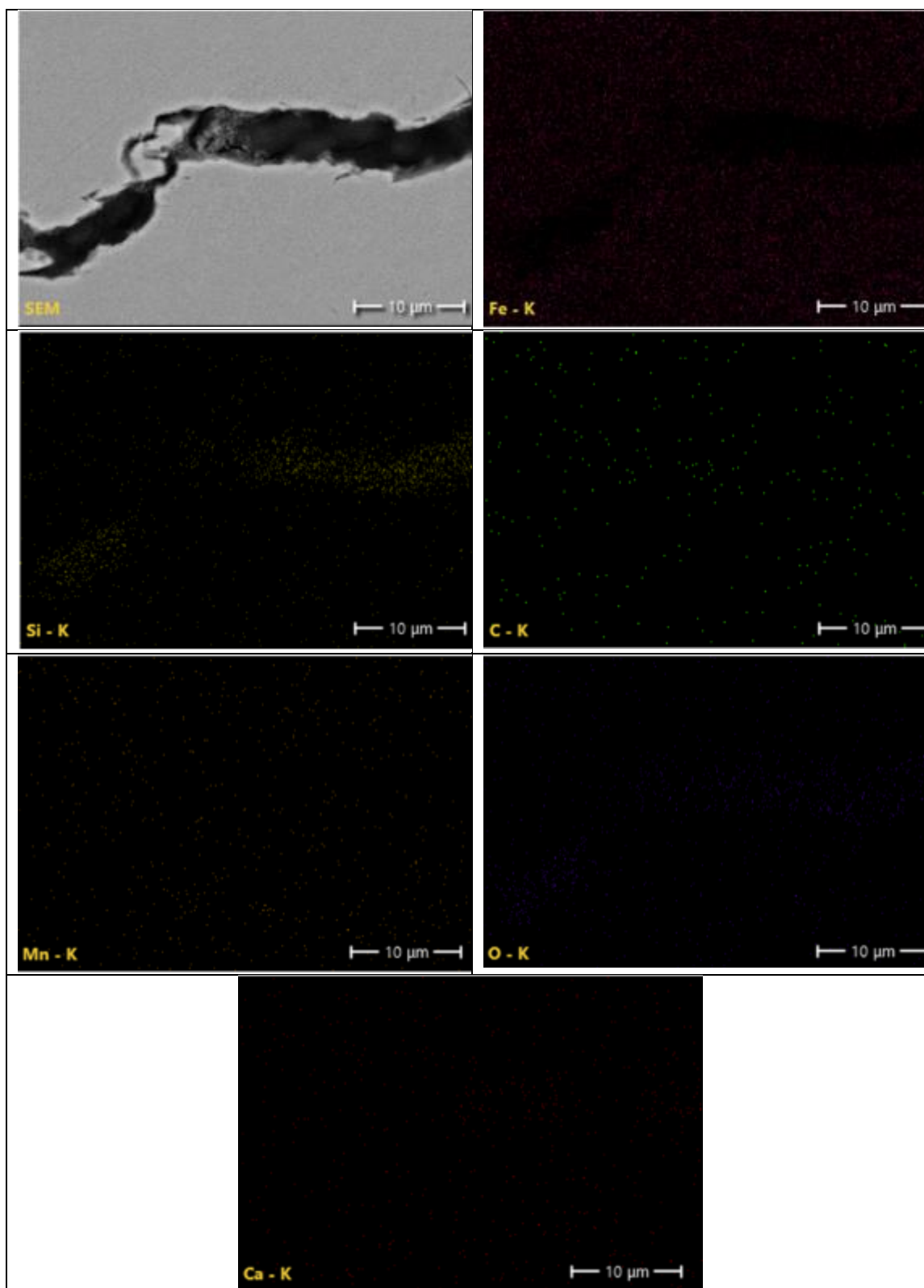


Figure 57: SEM & EDX analyses on HIC cracked Specimen where Mn is present





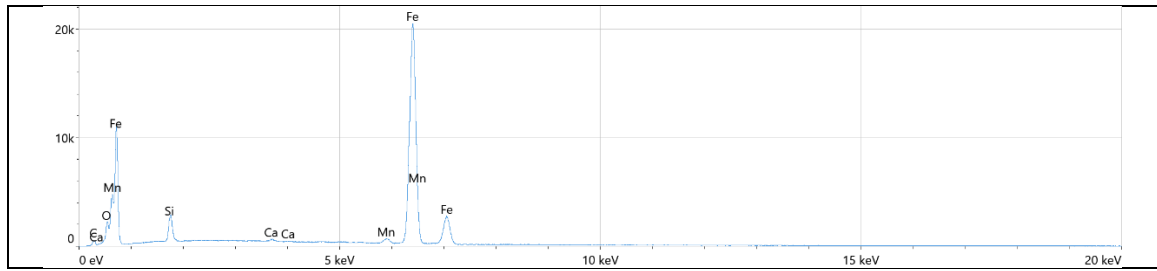


Figure 58: EDX Mapping of HIC cracked specimen where Mn is present.

Inclusions are common material defect that are encountered in any metallic material and can not be avoided. However, certain enhancement on the morphology and shape were reported that could improve the material properties. These inclusions are very tiny and can not be seen by naked eyes. It has to be examined under the microscope to be observed clearly. In the literature, it is reported that these inclusions are considered as irreversible hydrogen trapes and can initiate the Hydrogen Induced Cracking [40]. Hence, it was recommended to characterize the inclusions around HIC crack areas. The EDX results revealed presence of Ca, S, traces of Mn, Si and Al. the most dangerous inclusion is MnS type II which is characterize as elongated shape as the cracks are considerably propagated. As a solution for eliminating elongated MnS inclusion, it is recommended to reduce sulfur level of molten steel and treat the it with Ca addition treatment in order to change the shape of elongated MnS to globular MnS which is known as non-risky inclusions and do not initiate HIC cracks or optimize HIC cracks when exposed to stresses. However, CaS inclusions are encountered while treating the molten steel to change the shape of MnS inclusion but it is not dangerous compare to MnS inclusion. This type of inclusion are enhanced by reducing the sulfur content and adding Ca treatment in the molten steel to change the inclusion shape from elongated to spherical shape [55,56].

The other important inclusion is CaO which has been reported in SEM/EDX for majority of the HIC specimens that failed the HIC testing. Figure 59 demonstrates the influence on Ca/S ratio on the formation of MnS and Ca-Oxysulfide inclusions. It is believed that the increase of Ca/S ratio indicates that the Ca-Oxysulfide inclusions shall be produced. It is believed that the Ca-Oxysulfide inclusion are produced as soon as the Ca/S ratio increased [70].

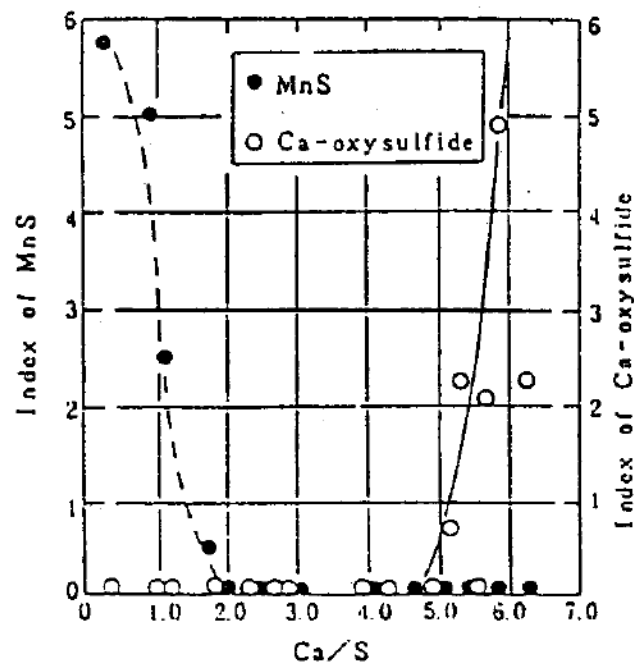


Figure 59: Ca/S ration and index for MnS and Ca Oxide inclusions. (Reference: Tagunchi et al, Tesu-to-Hagane, 1985, A37 [70])

Therefore, lowering the sulfur level of molten steel and introducing Ca treatment were not fully resolving the HIC failures. However, the industry established calcium to sulfur ratio criteria because addition of calcium during treatment of molten steel could adversely affect the HIC properties due to presence of oxides and could be considered as a crack initiation site [31]. It is reported that the Ca/S should be greater than 1.25 in order to have a HIC resistance material properties. However, the maximum value of Ca/S ratio is not very clear. The literature report that the maximum value shall be lower than 8 for

pipelines and 10 for pressure vessel plates. These values are not consistent because the Ca/S ratio for sample A and B are 13.6% and 4%, respectively, and both samples experienced HIC cracks.

However, it is reported that Ca treatment is not an effective method particularly for low sulfur content ($S < 0.001$ wt.%) as the formation of sulfide inclusions is negligible and the shape of inclusion will be globular [71]. This claim is already supported by YOKOTA et al [72] where they carried out HIC testing on TMCP API 5L Gr. X65 pipelines with low sulfur content (0.0006 wt.%) and experienced HIC cracks due to Ca Oxide formation. They recommended that the Ca/S ratio should be maintained in the range of 3 to 8 [72] for low sulfur steel.

The SEM/EDX analyses indicated presence of Ca, Si, Al Mn and Oxide. These elements are well known as HIC crack initiation sites. The peak of Ca content in Figure 55 indicated no sign of manganese sulfide trap sites. This proves that thermomechanical plates with ultra low sulfur that are subjected to Ca treatment will be susceptible more to globular inclusions than the famous Manganese sulfide inclusions. Literature review suggests that plates with ultra low sulfur content shall not be subjected to Ca treatment since the chance of development of manganese sulfide trap sites are minimal.

4.3.5 Result of Tensile Test Specimen (ASTM A370)

Post immersion in NACD solution A, the tensile specimens were tested in accordance with ASTM A 370. Table 11 shows that the tensile strength of the immersed models drastically increased compared to the original specimen. In addition, the elongation of tested samples revealed that the material was exposed to an embrittlement environment.

Also, the absorbed materials were cracked faster than the original material, as shown in Figure 60.

Table 11: Tensile Test Results after immersion in NACE Solution A

Name	Base Metal						
	Sample	Fracture	YS	TS	Load	YR	EL
	I.D.	Location	MPa	MPa	kN	YS/TS	%
Original Material (B)	TBT	Base	509	569	480.37	0.89	59.42
TS HIC 1	TBT	Base	561	588	623.32	0.96	50.88
TS HIC 2	TBT	Base	544	584	613.09	0.93	32.90
TS HIC 3	TBT	Base	555	593	625.68	0.94	45.39

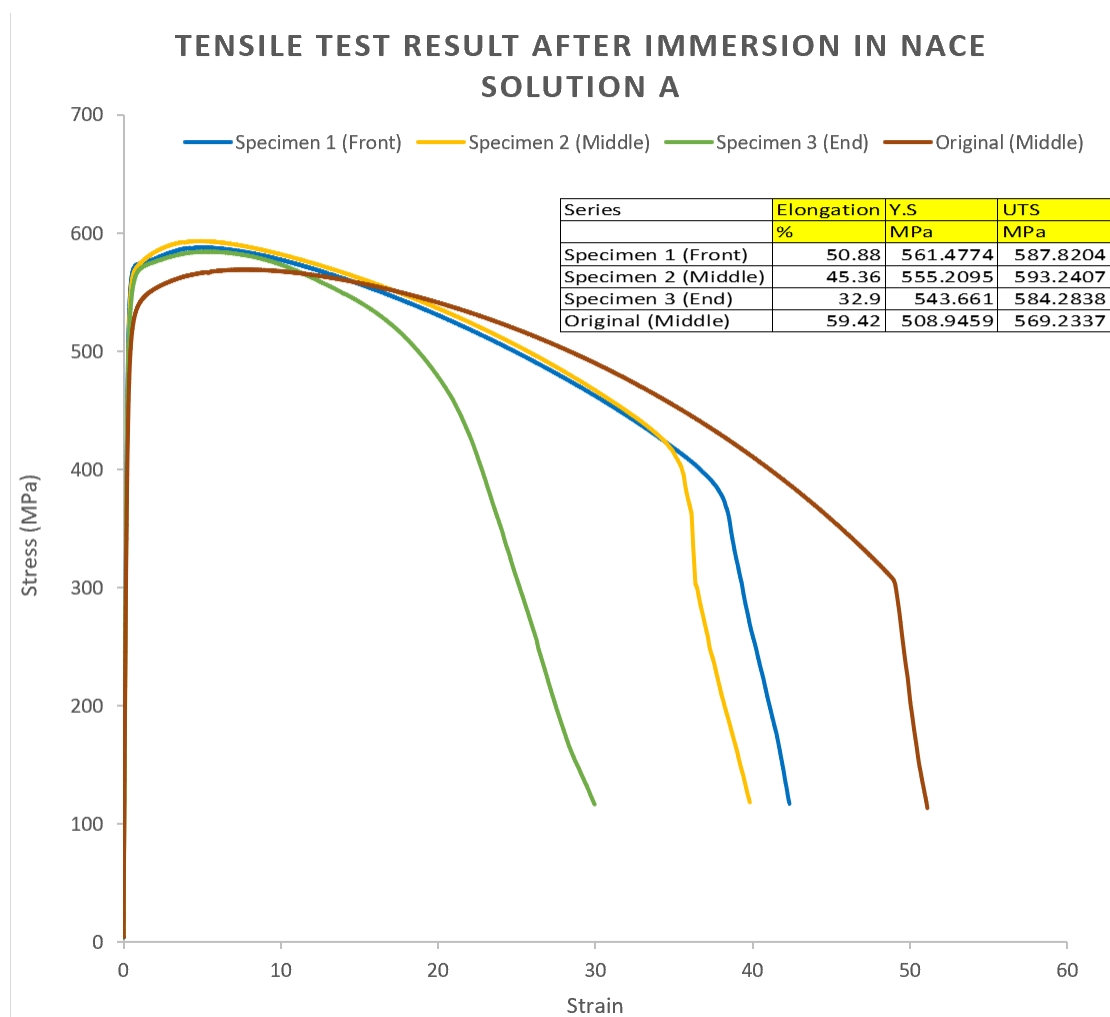


Figure 60: Stress Strain Curve of Immersed tensile specimens and original material.
The results of tensile test post immersion in NACE solution is in line with the recently published test results on carbon steel material with different grade and pipe

manufacturing process. The authors conducted tensile test specimens after immersion in NACE Solution A for TMCP API 5L Gr. X60 manufactured by Electric Resistance Welding (ERW) methodology. The ultimate tensile and the yield strengths were increased in the range of 4 to 17 % compare to original material specification. Also, the elongation percentages were decreased in the range of 1 to 2.86 % compare to the original material on the samples extracted from base metal area [73].

4.3.5.1 Fractography Results of Post Immersion in NACE Solution A

The fracture surface of the immersed tensile specimen in Solution A was examined post Tensile Testing. All the specimens, T1, T2 & T3, shows typical fracture behavior. It is believed that the fracture started in a brittle manner and ended in a ductile approach. The brittleness features were hardly discovered because the samples shows a lot of corrosion which eliminate the visibility of brittle fracture features despite a lot of cleaning conducted using ultrasonic methods, Figure 61.

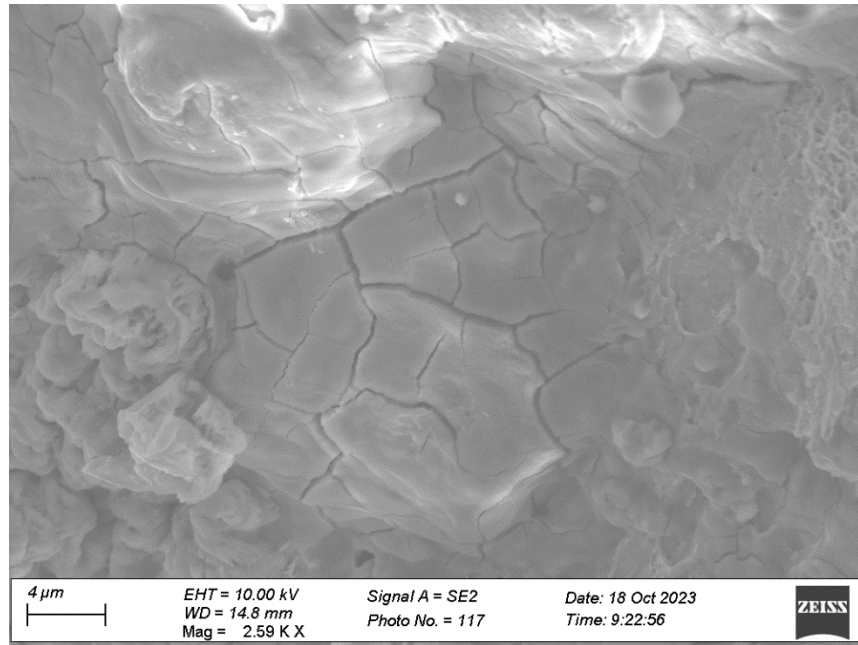


Figure 61: Fracture Surface of tensile specimen post immersion in NACE solution A in the crack initiation area.

On the other hand, the fracture surface on the core area shows typical ductile features. The dimples, which is commonly known as a feature of ductile over load failure, where clearly observed, Figure 62.

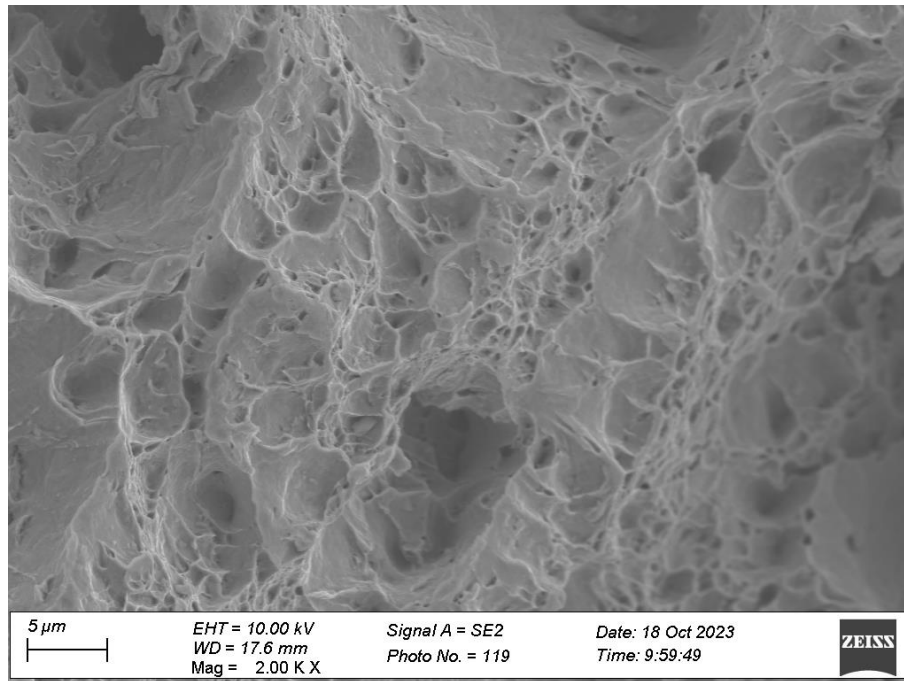


Figure 62: Fracture surface of Tensile Specimens post immersion in NACE solution A, Core Area of Fracture Surface

The tensile test results post immersion in NACE solution result indicates that the material experienced sign of embrittlement. The significant increase in tensile and yield strengths and severe reduction in elongation percentage shows as evidence that the material got embrittlement although some literature refer to HIC as not embrittlement failure mode. Furthermore, the tensile strength of the material that immersed in NACE Solution A exhibit higher strength compare to un-immersed specimen. The analysis of fracture surface for the immersed specimen indicates that the failure is attributed to ductile manner. However, it is believed that the failure initiated as a brittle failure but the brittleness feature on the cracks were not be visible due to exposure to corrosive environment that make brittleness features vanished. On the other hand, the fracture surface of the central area where end of crack occurred clearly shows ductile feature where dimples that are common feature for ductile over load were detected.

In principle, the fracture surface of the failure is showing only ductile features. However, the tensile testing results indicate some sort of brittleness. This could be explained as the duration of the testing was not enough to make the material more brittle. It could show significant increase in the brittleness if the exposure time is increased.

4.4 Electrochemical Hydrogen Charging

The tensile strength of the charged materials has significantly increased and the ductility was drastically decreased.

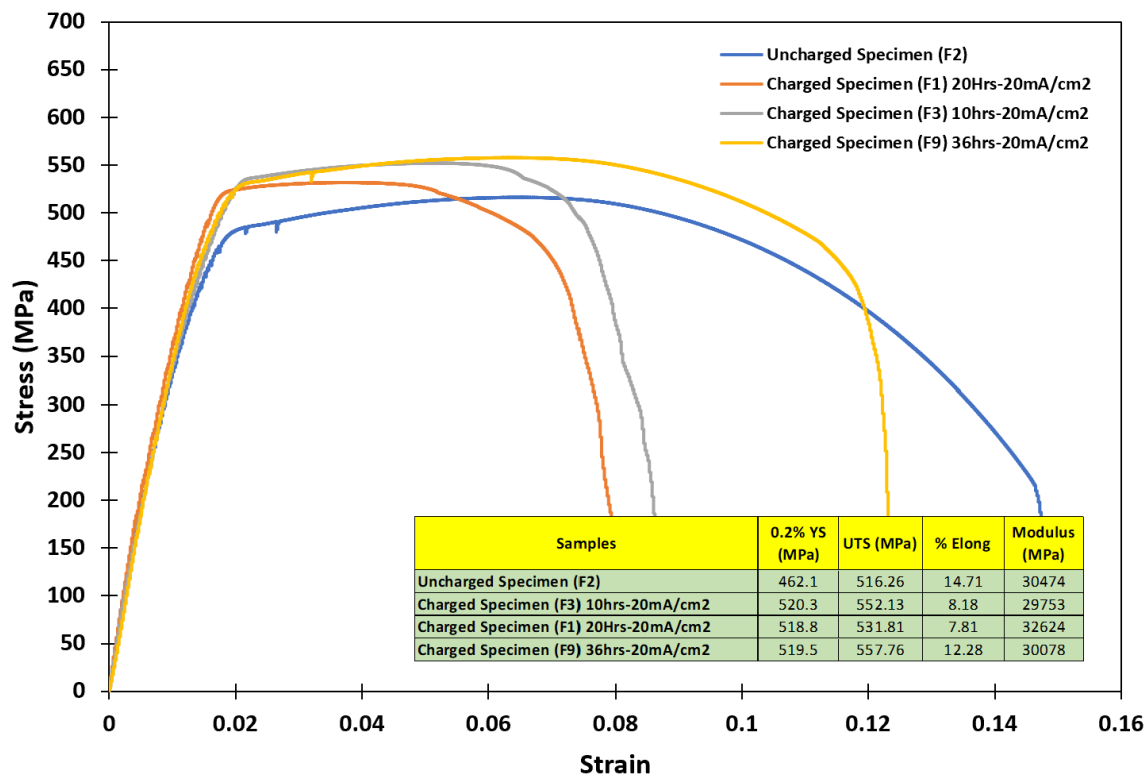


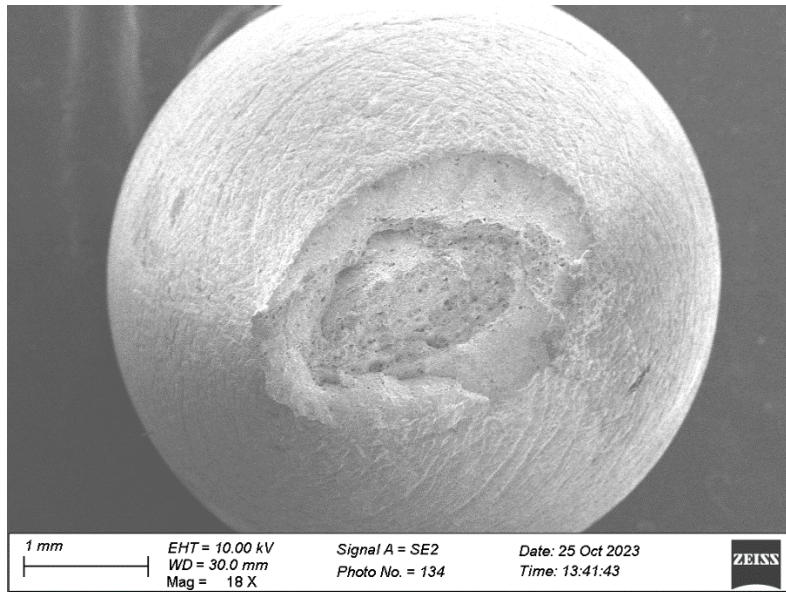
Figure 63: Tensile testing results of electrochemically Hydrogen charged specimens. The tensile testing of electrochemical charged specimens was vary with changing the charging time. Figure 63 shows the impact of tensile strength and charging time at 20 mA/cm² current density. The charged specimen for 20 hours was the most brittle material

behavior. On the other hand, the charged specimen at 36 hours showed the lowest brittleness characteristic. This could be explained that the material after 24 hours charging got saturated with hydrogen and resulted in increasing the ductility behavior. Similarly, a recent study was carried out to evaluate electrochemical hydrogen charging of carbon steel and low alloy steel materials. It was found that the hydrogen content on charged specimen was reduced after 20 hours from 2.5 ppm to 1.9 ppm after 168 hours of charging in same electrolyte solution. They did not figure out the exact reason for such drop in the hydrogen level. However, they assumed that the hydrogen level was reduced due to the reduction of sulfide content in the solution as a result of circulation of electric current [74].

It should be highlighted that the elongation percentage for the charged specimens demonstrated lower value compare to the uncharged specimen. This is a clear indication that the material exposed to embrittlement.

4.4.1 Fractography Results of Post Electrochemical Hydrogen Charging

The fracture surface of the tensile specimens after hydrogen charging was pretty much different from each charging time. However, the overall indication is a brittleness of the specimens where necking was not shown. On the other hand, the original sample clearly showed ductile nature of failure where dimples, typical feature of ductile overload, were observed, Figure 64.



1 mm

EHT = 10.00 kV
WD = 30.0 mm
Mag = 18 X

Signal A = SE2
Photo No. = 134

Date: 25 Oct 2023
Time: 13:41:43



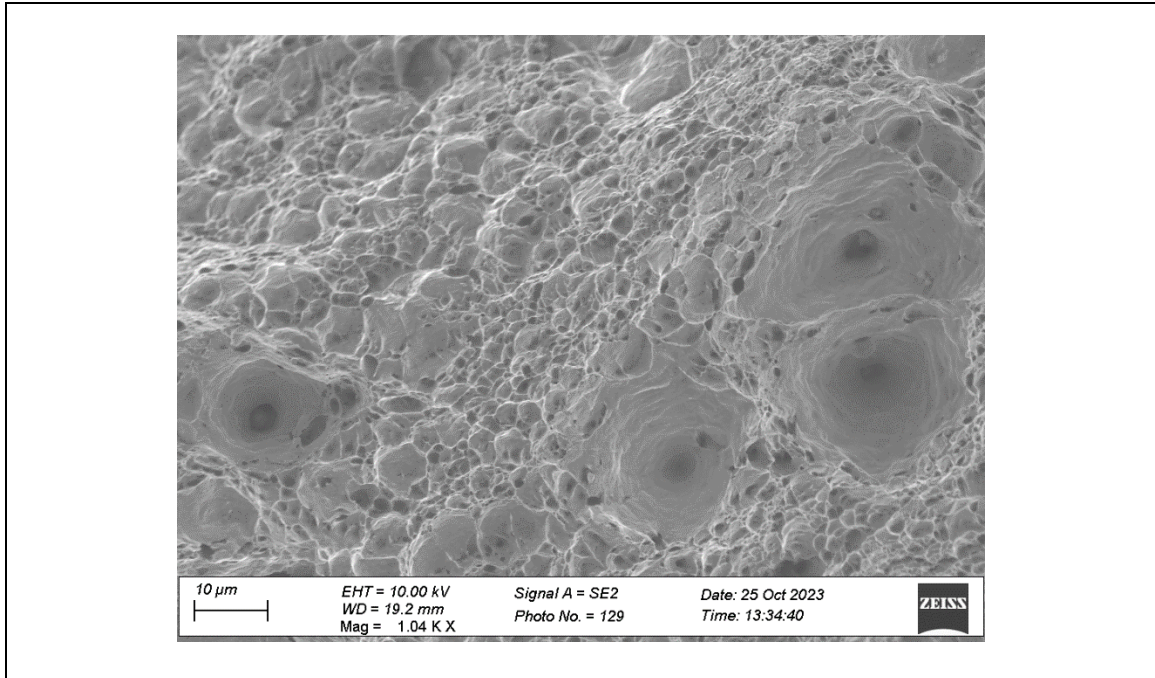


Figure 64: SEM analysis of the fracture surface of original material (without charging) F2.

The fracture surface of the charged specimen at 20 hours showed brittle fracture feature without necking and flat surface, Figure 65.



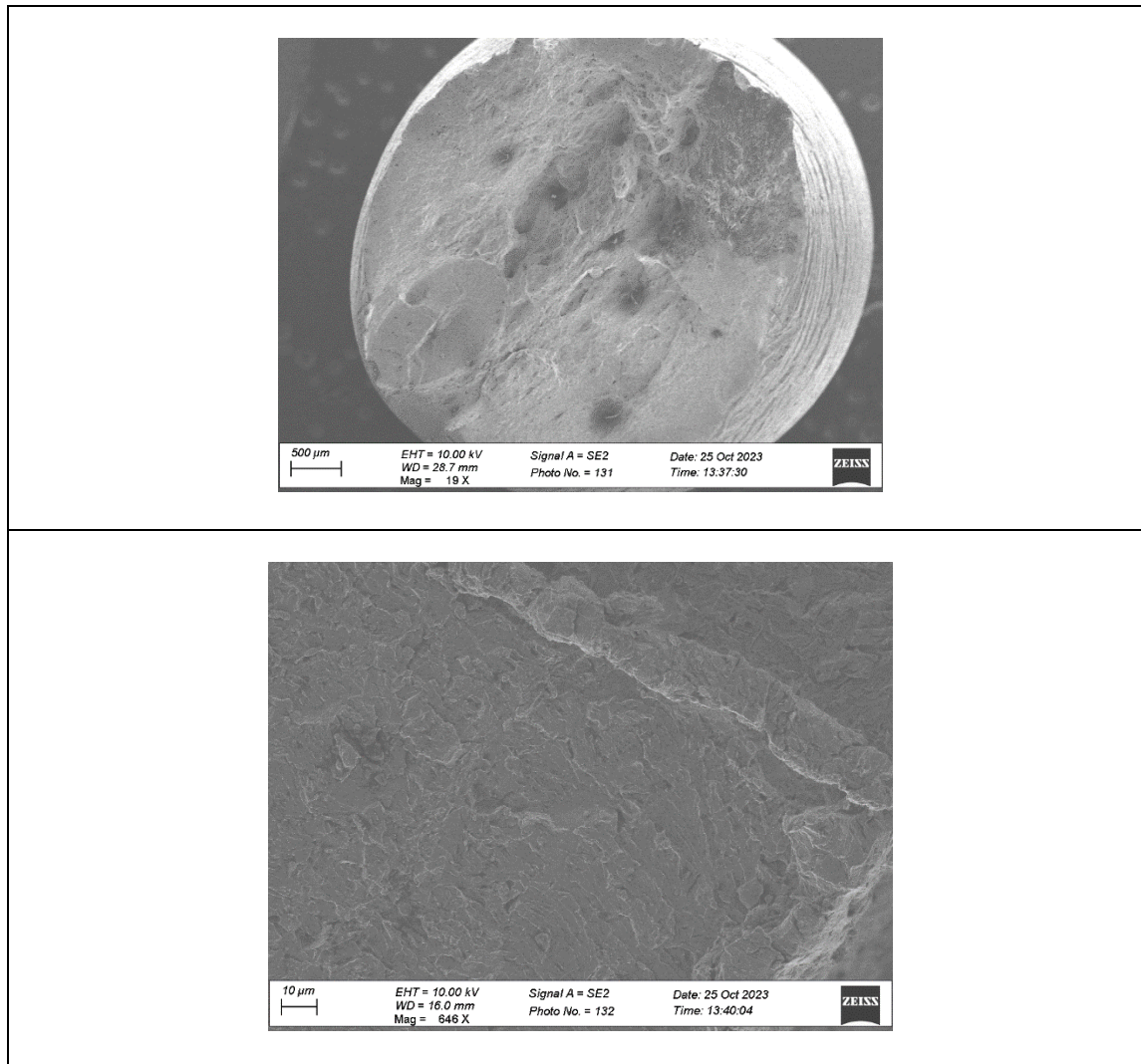


Figure 65: SEM result of fracture surface of charged specimen for 10 hours (F3).

For the charged specimen for 20 hours (F1), the fracture surface showed a little necking. However, the overall fracture surface is brittle., Figure 66.

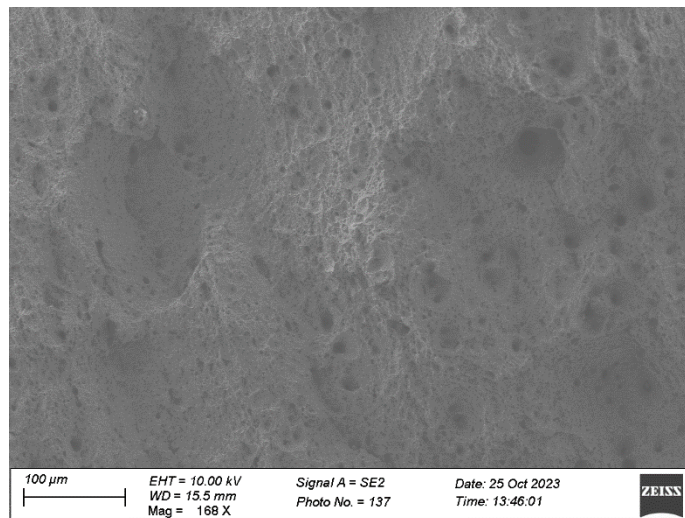
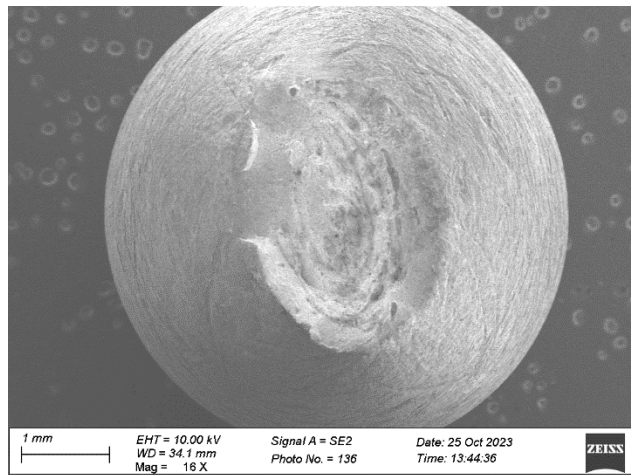


Figure 66: SEM result of charged tensile specimen for 20 hours (F4).

The specimen that was charged for 36 hours showed combination of brittle and ductile fractures features, Figure 67.

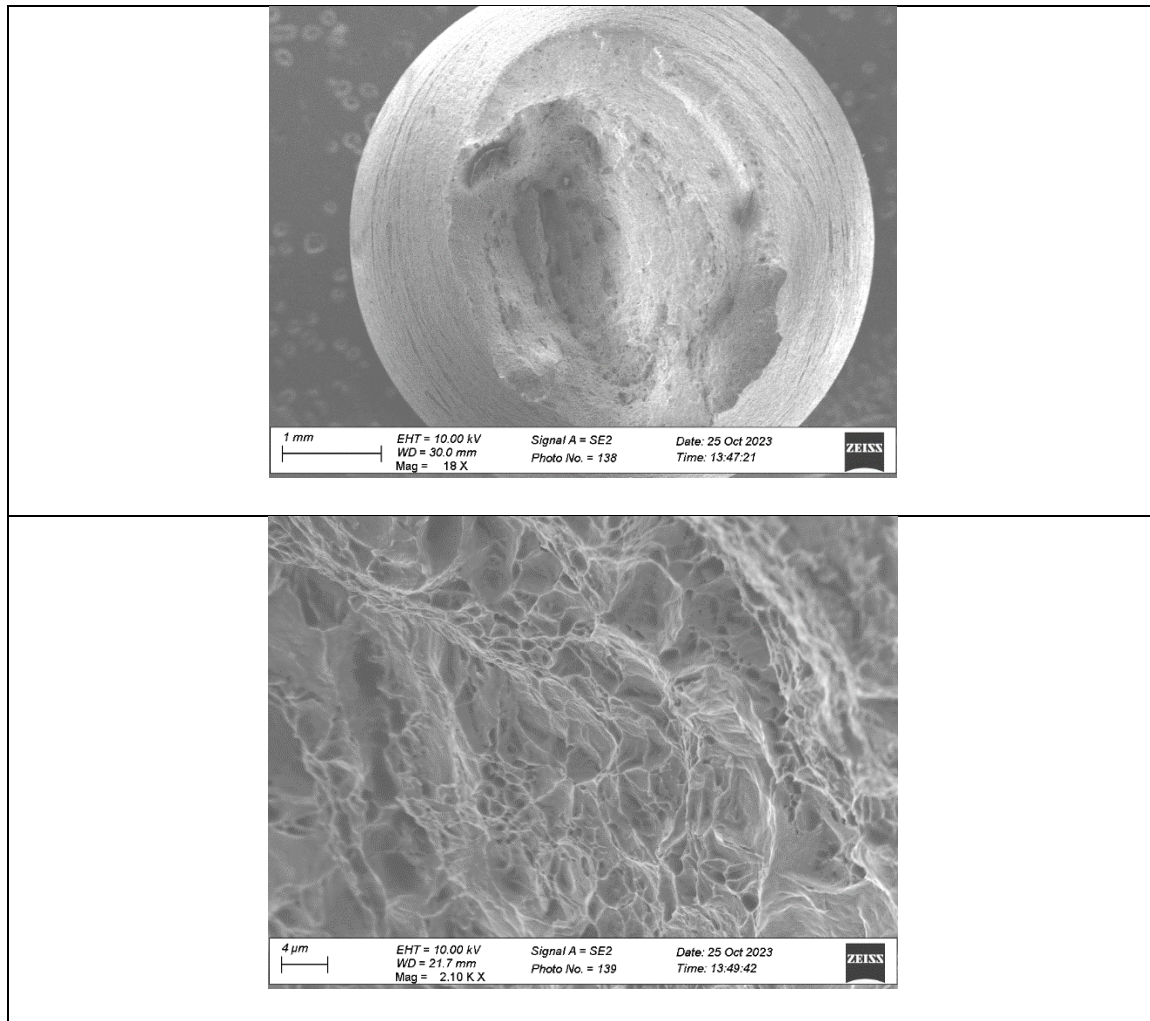


Figure 67: SEM results of fracture surface of charged specimen for 36 hours (F9). The fracture surface of all the charged specimens showed dimples, which are known feature for ductile overload failure. However, the general photo of the uncharged specimen demonstrated clear necking feature as shown in Figure 64. The necking is known as a ductile type of failure. This indicates that the material is able to absorb the energy and endorse deformation prior to failure. On the other hand, the charged specimens did not show any sign of necking features, which is indication of brittleness as shown in Figure 65.

4.5 Grain Size and Inclusion Content Measurement

4.5.1 Grain Size

The grain size was measured using Planimetric Method for non heat treated specimens and heat treated specimens. The ASTM grain size number of non-heat treated specimens for both steel A and B was 8.44 μm , Figure 68.

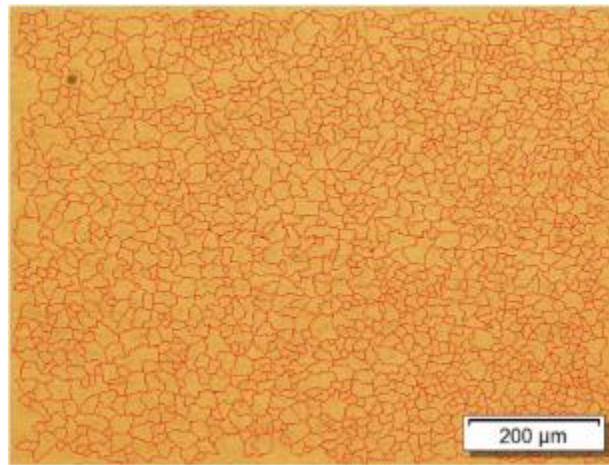


Figure 68: Grain Size Measurement for non-heat treated specimen.

On the other hand, the grain size has been increased when the samples were heat treated at different temperatures. The ASTM grain size number for the 650 °C, 600 °C, 550 °C and 500 °C are 9.27 μm , 9.05 μm , 8.80 μm and 8.46 μm , respectively. Figures 56-59 shows the calculated grain size for the specimens. This is a clear evidence that stress relieving treatment is slightly increased the size of the grains for the materials.

It is reported that grain size has an effect on hydrogen diffusion in the steels that could play a vital role in HIC resistance. The increased in grain size generally results in reducing the hydrogen trapping sites and increasing diffusivity and permeability in the steel [4,31,45,75]. This hypothesis is proved in the conducted research work where the

heat treated samples that have larger grain sizes illustrated excellent resistance to HIC cracks compare to the non-heat treated samples where the grain size was smaller.

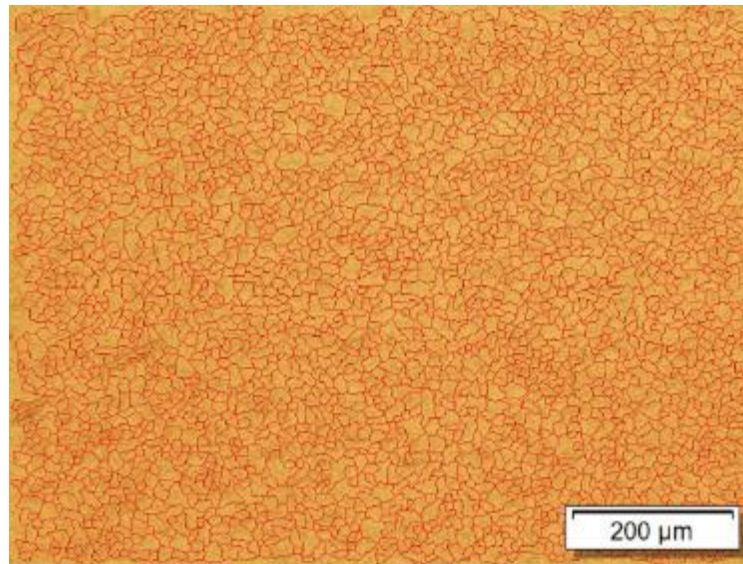


Figure 69: Grain Size Measurement for 650 °C.

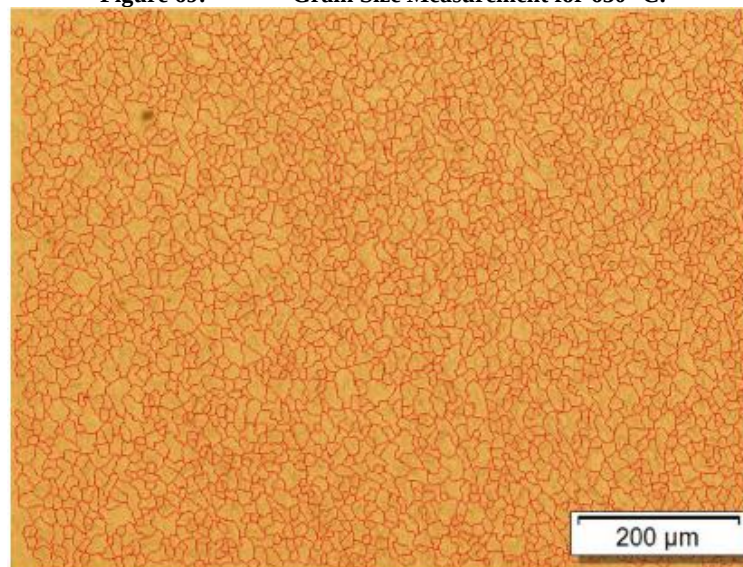


Figure 70: Grain Size Measurement for 600 °C.

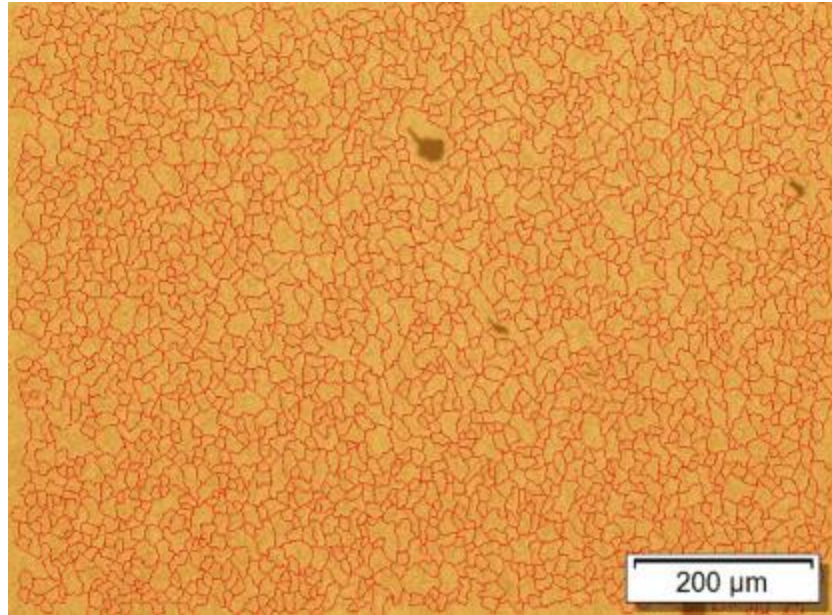


Figure 71: Grain Size Measurement for 550 °C.

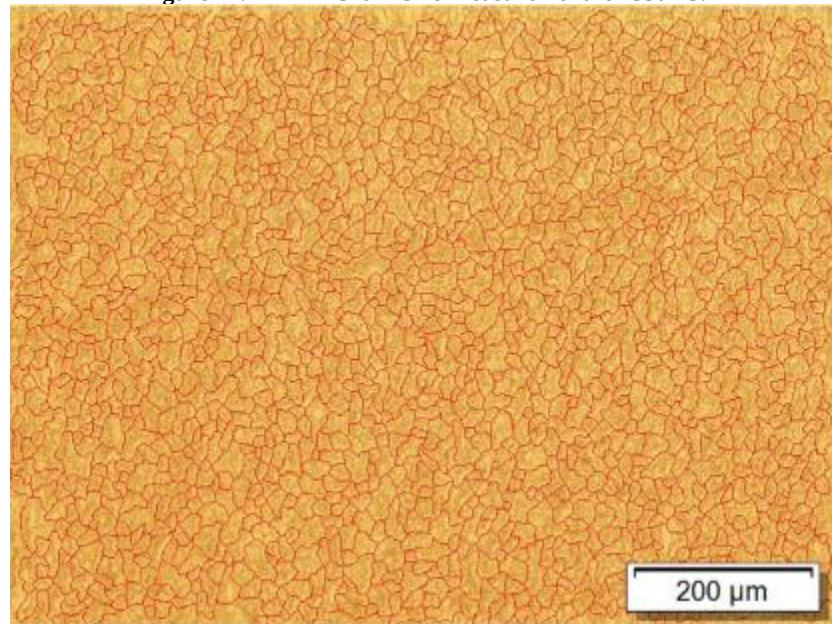


Figure 72: Grain Size Measurement for 550 °C.

4.5.2 Inclusion Content Measurement

The inclusion content measurement were carried out on the samples that demonstrated HIC cracks post immersion in NACE Solution A for 96 hours in accordance with ASTM E45. It is worth to highlight that the measured inclusion are based on the dimension, shape and

number on the microscope. It is not meant to provide chemical analysis of the inclusion.

Table 12 shows the severity level of each type of inclusion observed.

Table 12: Severity Level of inclusions observed after HIC testing.

Sample ID	Position	Section	Sulfide		Alumina		Silica		Globular Oxide	
			Thin	Heavy	Thin	Heavy	Thin	Heavy	Thin	Heavy
Sample A	0	1	0	0	0	0	0	0	1.5	0.5
Sample A	0	3	0	0	0	0	0	0	1.5	0.5
Sample B	0	1	0	0	0	0	0	0	2.0	0.5
Sample B	0	3	0	0	0	0	0	0	2.0	0.5
Sample B	90	1	0	0	0	0	0	0	2.0	0.5
Sample B	90	3	0	0	0	0	0	0	2.0	0.5
Sample B	180	2	0	0	0	0	0	0	2.0	0.5
Sample B-HT	180	2	0	0	0	0	0	0	2.0	0.5
Sample C	0	1	0	0	0	0	0	0	3.5	1.0
Sample C	0	3	0	0	0	0	0	0	3.5	1.0
Sample C-HT	90	2	0	0	0	0	0	0	1.5	0.5
Sample C-HT	90	3	0	0	0	0	0	0	1.5	0.5
Sample D	0	1	0	0	0	0	0	0	2.0	0.5
Sample D	0	3	0	0	0	0	0	0	2.0	0.5
Sample D-HT	180	3	0	0	0	0	0	0	0.5	0.5

The measured inclusion contents for each cracked samples revealed that globular oxide inclusions are the main founded inclusions. This is the expected result particularly for HIC resistance materials where the inclusion shape should be changed from elongated to spherical. It is reported that globular inclusions have higher HIC resistance compare to elongated shaped inclusion due to stress concentration is more in the crack initiation sites [63]. However, the inclusion content indicated that all these inclusions were in the form of globular oxide despite the claim in the literature where globular inclusion considered less risky to initiate HIC cracks. The initiation of HIC cracks from globular inclusions indicates that HIC resistance materials are not fully immune of HIC damage. This finding is also reported as a major challenge for hydrogen damage related to Oil and Gas Industry [69].

CHAPTER 5

CONCLUSION

5.1 Overview

This conclusion chapter is meant to summarize the entire research project by providing outline of each chapter, sharing the challenges and future opportunities on HIC, summarizing the research outcomes and providing recommended future work.

5.2 Outline

This research focuses on investigating the Hydrogen Induced Cracking failure of HSLA steels pipes that manufactured from HIC resistance materials. The targeted grade was API 5L Gr. X65 and the mother plate passed successfully the HIC testing. It is believed that the cold forming and welding processes of HSLA steel pipe resulted in developing micro-residual stresses that adversely impacted the HIC resistance of the material. The first chapter of the research demonstrates general technical knowledge on wet H₂S damages that encountered when exposed to sour service environment. Hydrogen Blister, Hydrogen Induced Cracking (HIC), Stress Oriented Hydrogen Induced Cracking (SOHIC) and Sulfide Stress Cracking (SSC) are the main damage mechanisms that affect the integrity and safe operation of metallic components that operate in sour service environment. Additionally, this chapter discusses the manufacturing processes of HIC

resistance plates and HSLA steel materials. The HIC resistance materials generally manufactured using basic oxygen furnace, pretreatment at the Kambara Reactor, vacuum degassing, and slab continuous casting. This manufacturing process usually produce clean steel with less or controlled inclusions. The research objective was focusing in investigating the HIC cracks on as pipe condition while the mother plate passing the HIC testing.

Chapter 2 demonstrates the technical literature on HIC behavior in industrial applications. Different characteristic of various wet H_2S damages were defined and classified. The hydrogen blister is a common form of all these damages or it is considered as initial stage of hydrogen related failures. The hydrogen blister generally started when hydrogen atom get trapped inside the inclusion and reacted with other hydrogen atom H^+ to produce hydrogen molecular H_2 . This reaction results in developing excessive stresses inside the material and result in bulging the material. The HIC is encountered when two hydrogen blister connects with each other and propagate in a through thickness cracking without presence of external tensile strength. The SOHIC failure is generally known as the ladder type cracking which connect pre-existing HIC crack in a perpendicular way to the applied stress. The SSC is other different type of wet H_2S damages which can be encountered when the material exposed to corrosive environment, tensile strength and high hardness area. This type of cracks generally encountered on Heat Affected Zone where the residual stress after welding is high and the hardness is high as well. The industrial experience to prevent SCC is to maintain the hardness level to be less than 248 HV. Also, this section shied the light on the importance of testing and inspecting HIC damages in the laboratory scale and industrial applications. The HIC testing is mainly classified to tow sections

which are metallurgical and immersion testing using electrochemical techniques. The industrial practice prefers the metallurgical and immersion testing while evaluating HIC failure in the presence of H_2S gases. The HIC failure is always depending on several important factors that determine the crack initiation and propagation. These factors include the microstructure of material, applied heat treatment while producing the steel plate, alloying elements, inclusion and precipitates and finally the process environment where these materials are going to be installed or operate.

The third chapter discusses the experimental method applied on this research study. The research were conducted on several steps which mainly can be categorized in to tow main parts. The first part suggests to conduct HIC testing in accordance with NACE TM 0284 with heat treatment and non-heat treatment conditions. The second part is to apply Electrochemical hydrogen charging on tensile testing specimens to quantify the effect of hydrogen charging on the mechanical properties. According to literature review, the safest method to apply this hydrogen charging in the lab is to use 0.2Mol sulfuric acid with 3g/L ammonium thiocyanate at 20 mA/cm^2 . Then, other metallurgical techniques were employed to classify the inclusion type and grain size. Finally, the fracture surfaces were examined using the Scanning Electron Microscopy and Energy Dispersive X-Ray. The fourth chapter demonstrates the outcome of these testing conducted throughout the research study. The results found in line with the research objective and provided a promising feedback. The heat treatment resulted in enhancing the HIC resistance of as manufactured pipe. The micro-residual stresses imposed on the pipe during the manufacturing process has adversely affected the HIC resistance characteristic.

In Chapter 5, the screening testing were conducted to prove that the received materials conform to the technical specification Of API SPEC 5L Gr. X65 in terms of chemical and mechanical properties. The conducted heat treatment enhanced the HIC resistance properties of as manufactured pipes condition. The HIC crack showed sign of Ca, Oxide, Mn, Si and Al elements on the crack surface while performing EDX analysis. These elements are known and reported to be a crack initiation site for HIC crack. The presence of CaO inclusion resulted from Ca treatment that imposed to enhance the HIC resistance for the mother plate. The Ca treatment is implement in industrial application to control the shape of MnS including and convert it from elongated shape to globular shape. This shows a suspect whether or not the Ca treatment is beneficial particularly for low sulfur grade HSLA steels. The Ca treatment is common and proven technology method for enhancing the shape of inclusions and improve the resistance for the HIC crack. However, special attention shall be taken to quantity and develop acceptance criteria for Ca treatment particularly for the low sulfur grade materials. Other doubt is whether or not the crack observed that resulted from CaO is considered as a HIC crack. It is believed that the crack is a HIC crack as the morphology looks like step wise cracking. However, SOHIC cracks are not generally follow ladder like cracks. It can be propagated in different direction depending on the stress location. Figure 3 shows that SOHIC cracks propagation can vary depending on the type of stress that imposed on the material. However, the crack propagation should be perpendicular to the applied stress and depending on the level of stress the propagation could be different. To conclude this research, the observed cracks are most likely HIC cracks as the crack morphology implies the step wise cracking characteristics. The globular oxide inclusions

are the predominant inclusions that initiated the HIC cracks. The tensile strength properties were adversely impacted after immersion in NACE Solution A. The results revealed that the material showed sign of embrittlement. Evidently, the elongation percentage was drastically reduced from 59.42% to 32.9%. Additionally, the impact on the tensile and yield strengths values for the immersed specimens has slightly increased compare to the original. The fracture surface of the final stage revealed dimples which is known as ductile overload features. However, the area close to initiation of cracks shows intergranular cracks which can be classified as brittle failure mode. However, the presence cleavage or river like manner were not observed during scanning the fracture surface. This could be attributed to internal corrosion that occurs on the specimens after immersion in the NACE Solution A.

The mechanical properties of tensile specimens post electrochemically hydrogen charging showed different characteristic than the original materials. The elongation percentages of charged specimens and uncharged specimen were reduced almost to half. The lowest elongation percentage was encountered for the charged specimen at 20 hours which is 7.81% compare to uncharged specimen 14.71%. This is a clear evidence for the impact of hydrogen on the mechanical properties which makes the material follow brittle nature. The uncharged specimens showed necking which is a common observation for any ductile material. Whereas, the charged specimens did not demonstrate any sign of necking which indicates that the material followed brineless cracking features. Also, it can be concluded that the immersion test and electrochemical charging test provided similar results in terms of mechanical properties. This indicates that the electrochemical hydrogen charging can be utilized in lie of NACE TM 0284. However, the charging

current density and time shall be defined prior to implement such testing. This will provide alternative and safe way to evaluate HIC cracking failures.

To summarize, this research provides sufficient knowledge on the HIC examination and like it with the mechanical properties of the materials. The results were optimistic and aligned with published literature.

5.3 Challenges and Future Opportunities

5.3.1 Challenges

Many challenges are encountered by scientists and engineers working on understanding and preventing HIC damage. However, preventing HIC damage is crucial to eliminate sudden catastrophic failure of components that operate in wet-sour environments. These challenges include, but are not limited to;

- Using HIC resistant steels, which are costly but still not completely immune to HIC damage
- The high cost of NDT techniques and their proper utilization for accurate identification as well as the requirement of specialized tools for examination.
- Experimental challenges due to obvious challenges related to H₂S use in the lab environment
- Difficulties in transferring the present HIC models to actual industrial applications for accurate prediction of lifetime of HIC damage.
- Producing HIC resistant pipes requires various controlling steps to be conducted, which include severe restrictions on the cleanliness of the raw material.

- Industrial experiences show that HIC resistance plates pass the HIC testing (NACE TM 0284) and the pipe manufactured from same HIC resistance plate failed the HIC testing.
- HIC damage assessment is a challenge, owing to its dependence on several factors, even though API 579 Fitness for Service and other models exist.
- In several cases, replacing the metallic components with HIC resistant steel that operate in wet sour environment is a cost-effective option compare to continuous monitoring with NDT, h However, it has its challenges related to work delays, material costs, etc.

Despite the availability of various experimental techniques for HIC, there is only one reliable technique to simulate the actual environmental conditions.

Traidia et al. reported two major challenges that prevent using available HIC models for industrial applications, which include 1) difficulty in identifying boundary conditions and model parameters and 2) complexity of actual field cases. The obstacles related to model parameters include microstructure, steel composition, defect size, Non-Destructive Testing (NDT), hydrogen charging condition, fracture toughness, cohesion properties, hydrogen diffusivity, residual stress, and initial state. Additionally, the hindrances to the complexity of the actual field case include the small size of the crack, load dynamic, and dK/dt hydrogen charging conditions. Inclusion and segregation at the grain boundaries are a major part of the hydrogen trapping sites in the microstructure. The available multiscale statistical HIC models need a lot of improvement to quantify and consider such types of defects [76]. Figure 73 summarize the current challenges encountered on HIC damage.

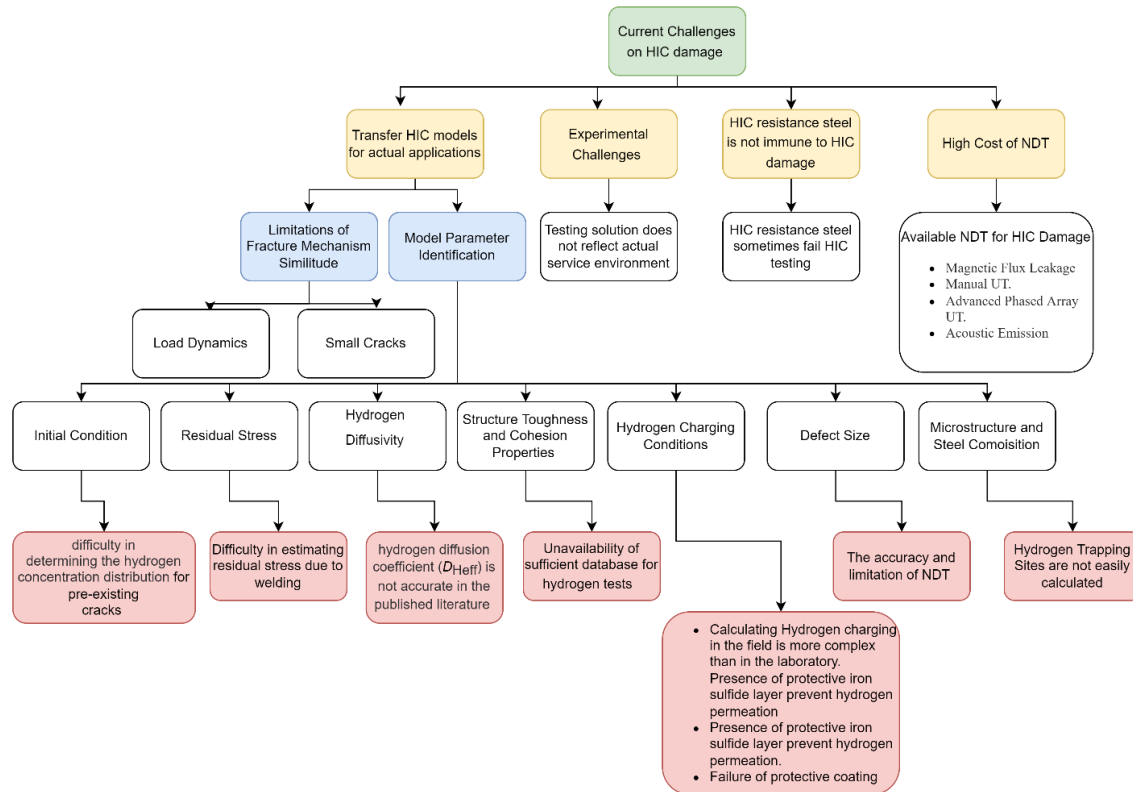


Figure 73: Flowchart of Current challenges on HIC damage

5.3.2 Future Opportunities

The future approach for preventing HIC damage is to develop a cost-effective technology that can inhibit HIC damage. This can be achieved by creating a clean raw material, either plate or coils, to minimize the presence of inclusions. Such an approach shall reduce the trapping site of Hydrogen atoms inside the steel and reduce the possibility of having HIC failure. Additionally, providing a reliable HIC assessment model for non-HIC resistance steels is a viable option to control HIC failures. The current available HIC models are based on experimental techniques and need more information about steel-making practices for raw materials. Finally, the cost of assessing and monitoring HIC failures is extremely high, and it is difficult to determine huge pressure vessels. It is reported that replacing the

pressure vessel with HIC resistance materials is still cheaper than monitoring the HIC failure. Hence, developing cost-effective NDT techniques is deemed necessary.

5.4 Summary

The main objective of this research work is to investigate the HIC behavior of HIC resistant LSAW API X65 pipeline steel samples through applying NACE TM 0284 and Electrochemical Hydrogen Charging Experimental. Also, the HIC samples were subjected to heat treatment or stress relieving treatment to reduce dislocation density and remove residual stresses. The following summarizes the findings of the conducted research work.

1. The pipe forming and welding processes could alter the HIC resistance properties of the materials by introducing or changing the reversible trap sites.
2. Stress Relieving Treatment is an effective method to release residual stresses and enhance dislocation density to increase the HIC resistance properties of the materials.
3. The observed cracks during the experimental work were attributed to Hydrogen Induced Cracking mechanism that could be distinguished by presence of stepwise cracking. The cracks morphology was classified as classical HIC failure where it occurred in the center line. These cracks can not be classified as Stress Oriented Hydrogen Induced Cracking (SOHIC) despite the presence of stresses during forming and welding processes to make pipes. The crack morphology did not show ladder-like cracks that connects pre-existing HIC damage.

4. Globular inclusions could support the HIC cracking initiation although many researchers emphasized that globular inclusions are resistant to HIC cracking. This finding is inline with various researchers who claimed that HIC resistance materials is not fully immune to HIC failure as its cracks in severe environment.
5. Ca Treatment is introduced and being used in the industry to make HIC resistance materials from changing the inclusions shape from elongated to spherical. This method is proven to be not effective method particularly for low sulfur grades ($S > 0.001$ wt.%) as demonstrated in this research work and supported evidence in the literature.
6. The mechanical properties of the TMCP Gr. X65 gets altered when immersed in NACE Solution A for 96 hr. The tensile and yield strengths and elongation percentages indicated that the material experienced some sort of embrittlement although the fracture surface of the immersed specimens showed ductile overload manner.
7. The mechanical properties of TMCP Gr. X65 steels of electrochemical charged specimens shows similar results compare to immersed specimens in NACE Solution A. The tensile and yield strengths and elongation percentage indicated that the material encountered embrittlement. This indicates that the electrochemical hydrogen charging experiment could obtain similar results as NACE TM 0284.
8. The charging time for electrochemical hydrogen charging is very sensitive. The effect of hydrogen diffusion is reduced as soon as the charging time is increased. The exact reason for this phenomena has not yet been discussed in the literature.

However, it is believed that the trapping sites get saturated with hydrogen atoms which prevent further hydrogen atoms from diffusion.

5.5 Future Work

The outcomes of this research work suggest a further study on the following topic.

1. Calcium to Sulfur ratio needs to be precisely identified in particular for low sulfur grades steel. The literature claims that low sulfur grades should not be treated with Ca treatment because the Sulfur level is low and will not produce elongated MnS inclusion. This will eliminate the HIC initiation from CaO inclusions. However, extensive research shall be carried out in order to homogenize this understanding.
2. The charging time of electrochemical method is effective in determining the results. The samples post charging are sent to tensile testing lab which gives ample time for the diffused hydrogen to be released from the sample. In this research, the lead time between hydrogen charging and tensile testing was fixed at 5 minutes. Therefore, it is suggested to do in situ hydrogen charging while conducting the tensile testing to eliminate the effect of lead time.
3. The effect of excessive hydrogen charging duration needs to be further examined and identify root cause(s) of such behavior.

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