Article

Wet H₂S corrosion and degradation of pipeline in amine regeneration system

Juliusz Orlikowski[1], Maciej Kalinowski[2], Igor Lasota[2], Piotr Maruszewski[2], Michal Szocinski[1], Kazimierz Darowicki[1]

[1] Department of Electrochemistry, Corrosion and Materials Engineering, Faculty of Chemistry, Gdansk University of Technology, Narutowicza Str. 11/12, 80-233 Gdansk, Poland

[2] ORLEN S.A., Chemikow Str. 7, 09-411 Plock, Poland

Correspondence: Michal Szocinski, Department of Electrochemistry, Corrosion and Materials Engineering, Faculty of Chemistry, Gdansk University of Technology, Narutowicza Str. 11/12, 80-233 Gdansk, Poland

E-mail: michal.szocinski@pg.edu.pl

Abstract

The paper presents the results of NDT examinations, metallographic tests and risk assessment of degradation related to corrosion of amine regeneration unit in a desulphurisation system. Intensive corrosion resulting from acid gases environment upon water condensation causes perforation of the pipeline. Detailed analysis reveals cracking related to the mechanism of wet H₂S. Hydrogen penetration, resulting from the wet H₂S process, causes a decrease in mechanical properties of steel and an increase in hydrogen content inside steel. Corrosion results mostly from high stream velocity, the presence of acid gases (with high ammonia content), the presence of amine and low stream temperature.

1 INTRODUCTION

Acid gases ammonia wash units are a common part of refinery and petrochemical plants all over the world. The main purpose of these units is removing acid gases (hydrogen sulphide, carbon dioxide) from dry process gases. [1,2] This operation allows processing of hydrogen sulphide in Claus units [1,2] and utilization of the purified gases as a fuel gas in plants' furnaces. Considering corrosion risks, the sections of amine regeneration are the most vulnerable to corrosion and degradation. [3] In these units, acid gases are removed from amine water solutions by increasing the temperature in regeneration columns. [1,2] Leaving the column, the regeneration gases contain significant amount of steam. In these conditions intensive corrosion processes called acid water corrosion and wet H$_2$S degradation occur. [4] As water condenses in the areas where acid gases vapours cool down, the conditions for dissolving of hydrogen sulphide, ammonia and carbon dioxide appear. Due to H$_2$S presence, the following corrosion reactions take place [5-7]:

$$Fe + H_2S + H_2O \; \rightarrow FeSH_{ads}^{-} + 2H_3O^{+} \tag{1}$$

$$FeSH_{ads}^{-} \rightarrow FeSH_{ads}^{+} + 2e^{-} \tag{2}$$

$$2H_2S + 2e^{-} \rightarrow H_2(atomic) + 2SH^{-} \tag{3}$$

The above process also results in the evolution of hydrogen gas, which leads to the degradation processes known as wet H$_2$S degradation. [3]

Wet H$_2$S mechanism can be observed in four different forms: blistering, Hydrogen Induced Cracking (HIC), Stress Oriented Hydrogen Induced Cracking (SOHIC) and Sulphide Stress Cracking (SSC). [8-11] Hydrogen blisters may be formed as surface bulges, cracking due to HIC, SOHIC and SSC processes may occur at many different depths by interconnecting

cracks caused by hydrogen blisters. [8-11] SOHIC is potentially more damaging as appearing as the arrays of cracks stacked on the top of each other. SSC is defined as cracking of metal under combined action of tensile stress and corrosion described above. Those mechanisms result in cracking of the plant's structural elements. The mechanism as well as the rate of corrosion processes are highly dependent on pH and temperature of the aqueous environment. [13] In acidic environments (pH < 7), the acid water corrosion processes occur. [3, 4, 12, 14] The factors further increasing corrosion rate include: presence of oxygen in the stream and increased flow rate. [13] In the situation of higher ammonia concentration in the stream and for aqueous environment pH higher than 7, the Alkaline Sour Water Corrosion process occurs. The dynamics of Alkaline Sour Water Corrosion depends mostly on the flow rate, however, it is also related to the concentration of ammonium bisulphide (NH_4HS). [3, 4, 15] Formation of the salt is connected with the concentration of hydrogen sulphide and ammonia in the aqueous phase. In the streams of amines the Amine Corrosion takes place. It is possible for the following corrosion reactions to occur [16]:

$$H_2S + R_3NH \ \text{-->} \ R_3NH^+ + HS^- \tag{4}$$

$$Fe + 2R_3NH^{+\,-} \text{-->} Fe^{2+} + H2 + 2R3N \tag{5}$$

Reaction (4) describes absorption of hydrogen sulphide by amine and reaction (5) is the corrosion reaction. Again, the wet H_2S mechanism occurrence is always initiated by corrosion mechanism and in the case of alkaline pH conditions this is the Amine Corrosion. Corrosion prevention relies mostly on using more corrosion resistant materials and on changing process parameters (reducing flow rate and concentration of NH_4HS). Reducing the risk of wet H_2S corrosion mechanism is also relevant. [17, 18] Stainless steels of much higher resistance to corrosion in aqueous environments containing amines, as well as in acid gases, are also applied in the amine regeneration units. Furthermore, stainless steels do not undergo

degradation according to wet H_2S mechanisms. However, stainless steels are prone to chloride cracking (Cl-SCC) in water environments at elevated temperatures. [19]

The objective of this paper is to study the case of malfunction of the pipeline transporting a stream of condensing vapours from the amine regeneration column based on the standard data from the corrosion and degradation risk assessment. [3, 4]

2 MATERIALS AND METHODS / EXPERIMENTAL

A pipeline which was the subject of the corrosion analysis was made from A333 grade 6 construction steel, following the ASTM standard. The chemical composition of chosen steel verified with the Spektrotest TXC03 spectrometer is given in Table 1.

Table 1

The research was carried out using the following techniques and methods.

- The comparative analysis of the chemical composition of streams was performed with the use of the Aspen Hysys v.12.1 software.

- Pipeline wall thickness tests, using UT-Scan method, were carried out with the Olympus Omniscan X3 32128PR. The length of examined section was 200 cm.

- Steel hardness was measured using the Portable Hardness Instrument type MIC 10.

- Temperature vacuum extraction method was used for evaluation of hydrogen trapped inside the material. The samples were shredded into small pieces by a slow rotating lathe. Obtained alloy chips were kept in an ultrasonic bath with distilled water for 5 min and then dried at 1100 °C for 1 hour. In the next step, the alloy chips were places under 800 mbar vacuum and heated up to 810 °C. Evolved hydrogen and methane were measured using the Agilent 6890 gas chromatograph with the following operation parameters: 2 m x 0.21 mm, molecular mesh 5A, 100 – 120 mesh, TCD

2200C detector, argon 15 ml/min as a carrier gas, 1000 °C column temperature, splitless doser.

- Mechanical tests were performed using the ZD 100/2 tensile testing machine. Stress-strain curves were obtained for 4 service-exposed and 4 non-exposed samples. The strain rate amounted 7.0 MPa/s.

- Metallographic investigations were carried out using the metallographic microscope VHX – 7000E. The samples were prepared by grinding with abrasive paper of gradation up to 4000 and then by polishing with diamond paste of gradation down to 1 mm. The etching involved a freshly prepared mixture of 5 cm^3 of 65 % nitric acid (HNO$_3$) and 100 cm^3 of ethyl alcohol (C$_2$H$_5$OH).

3   RESULTS AND DISCUSSION

3. 1. Process scheme and analysis of the chemical composition of process streams

The simplified scheme of the unit, where the pipeline failure occurred is demonstrated in Figure 1. The purpose of the unit is regeneration of amine (diethanolamine – DEA), the function of which is to remove hydrogen sulphide and ammonia from the stream of acid gases in the hydrodesulphurisation plant. Rich amine (DEA), which contains high amount of dissolved hydrogen sulphide and carbon dioxide, is heated up and directed to the regenerator (stripping column). Regenerated, freed from hydrogen sulphide and carbon dioxide, lean amine gathers in lower, heated by the reboiler, part of the column (Figure 1a). In the upper section of the regenerator, there is evolution of acid gas, which flows to the upper part of the column. Acid gas, together with steam, with the temperature of approx. 121 °C is directed to the air cooler (Figure 1b), where it cools down to 31.5 °C. As a result of cooling, the aqueous phase separates from the stream. The separation of gas phase takes place in the separator. Acid

gas is directed to the Claus unit, while the condensate comes back to the regeneration column as a phlegm.

Figure 1a, b

The chemical composition of the process stream is crucial for the corrosion analysis. The composition of the process stream in the unit is frequently analysed, however, all available data refer only to the acid gas and the condensate directed to the column. There is little information concerning the chemical composition of fumes from the column, which are directed to the separator. Regarding insufficiency of data, the Aspen Hysys software was utilized to assess lacking parameters. The analysis of the flow of fumes from the column to the separator (Figure 1b) was performed. The software allowed modelling of not only the chemical composition of the fume stream, but also physical conditions (flow rate, phase composition). Chemical composition of the stream that leaves the regeneration column is given in Table 2. The values presented in Table 2 are the results of computer calculation, performed with the Aspen Hysys software, and based on unrevealed in the paper (due to the policy of protecting company's inside data) process data.

Table 2

Fume stream (gas phase only) contains high amount of steam. Apart from steam the stream contains dry gases typical for the amine regeneration units, for instance hydrogen sulphide, carbon dioxide and ammonia. The presence of amine in the stream is caused by relatively high temperature in the upper part of the regeneration column. Computer analysis showed that if the temperature of the upper part of the column was lower than 100 °C, the amine would not be present in the stream. Based on the analysis of chemical composition of rich and lean amine, the thermostable salts (HSAS) are present in the stream, with the concentration up to 4 % of the amine content. The temperature of the stream entering the air cooler is above 100 °C,

while the leaving stream temperature is slightly below 30 °C. The chemical composition of the stream that leaves the air cooler is given in Table 3.

Table 3

Individual mass shares, according to the conducted simulation, were as follows: 0.66 – liquid phase, 0.33 – vapour phase, while molar shares were: 0.64 – liquid phase, 0.36 – vapour phase. Calculated pH value was 9.22. Despite significant amount of hydrogen sulphide, carbon dioxide and thermostable salts, the stream has alkaline properties, what results from the presence of high amount of ammonia and amine, water solubility of which is very high as compared to other acid gases. Furthermore, the dissociation constant of ammonia aqueous solution is over 100 times higher than of hydrogen sulphide solution. For the purpose of further calculations the concentrations of NH_4HS and $H_2S(aq)$ were assumed at the level of 28000 ppm and 27800 ppm, respectively.

3.2 Failure description

There are four pipelines with the diameter of 6 inches that leave the air cooler and then unite in one pipeline with the diameter of 8 inches. Directly after the air cooler in the region before non-return T-pipes (Figure 1b) two outer pipelines were unsealed. Perforations occurred in the region of the welded joint. Figure 2 demonstrates the internal and external side of corrosion damage of the pipelines.

Figure 2a, b

3.3 Non-destructive and destructive tests of the samples from the pipeline

The pipeline plate samples were examined using the UT-Scan technique. Figure 3 demonstrates the exemplary results of the UT-Scan examination of the pipeline that was perforated (Figure 2).

Figure 3

Examination of the samples revealed a significant corrosion loss, mainly at the ends of the plate samples. Ultrasonic tests were also performed on all sections of the pipeline, from the regeneration column to the separator. The average corrosion rate, measured gravimetrically on coupons by corrosion monitoring system operating in the refinery, was 0.3 mm/year, while on the samples from the region of T-tubes it was 1.8 mm/year, whereas on the collecting pipeline, in the region of the elbow before entering the separator, it was 1.56 mm/year. The pipeline walls were the thinnest in the areas where the flow direction changed, which suggests the influence of erosion. In order to assess the overall corrosion process, the metallographic examination of the native material (before the welded joint) in the region of the highest corrosion rate was performed. A metallographic microsection prepared from the sample taken from the pipeline is demonstrated in Figure 4.

Figure 4

The microsection in Figure 4 shows the corrosion loss, which is present on the internal surface of the pipeline. The corrosion damage takes an irregular form. Due to aqueous environment and significant amount of hydrogen sulphide, the occurrence of wet H_2S degradation is possible. In order to assess the risk of wet H_2S degradation, the hardness of the pipeline's material was examined using a hardness meter. The average hardness value obtained was 150 HB.

Due to considerable threat of hydrogen embrittlement, resulting from wet H_2S degradation, the hydrogen content in the material of the pipeline was measured. The examinations were carried out for the native material, the material from the heat affected zone (HAZ) and the welded joint material. The residual hydrogen content, after metallurgical process, was 4.1 ppm (measured on the pipeline material in the area were no corrosion damage was observed).

The value of residual hydrogen was subtracted from every result in order to arrive at actual hydrogen content in steel. The results of hydrogen measurement are shown in Table 4.

Table 4

The above results show a significant hydrogenation of ASMT A333 grade 6 steel. Hydrogen content in the heat affected zone is considerably lower, what suggests that post welding heat treatment process contributed to the reduction of hydrogen embrittlement risk.

In order to assess to what extend had the mechanical properties of steel reduced as a result of hydrogen absorption in the metal, the tensile tests were performed. Table 5 shows the tensile response of the tested materials.

Table 5

The results of mechanical tests indicate that steel strength parameters deteriorated. It should be noted that deterioration of the mechanical properties is relevant to the level of hydrogenation of the samples. The extensive analysis of the mechanical results was done by comparing the experimental results with the standards for investigated material. [20] Although the degradation was so severe in the wet H_2S environment, both critical values – the yield strength and the maximum tensile strength – were still at the acceptable level according to the standards indicating 241 MPa and 414 MPa, respectively. [20]

Taking into account chemical composition of the process stream, steel hardness and the fact that welded joints were heat treated after welding, the susceptibility of the pipeline to wet H_2S, HIC/SOHIC and SCC degradation was assessed based on the API 581 standard. [13]

Environmental severity analysis according to the API 581 standard, Table 9.2 yielded the following results:

- pH of water > 9.0 – pH according to experimental analysis: 9.22,

- H_2S content in water > 10,000 ppm – H_2S content according to experimental analysis: 27800 ppm.

Accordingly, environmental severity level was evaluated as high.

Susceptibility to cracking analysis – HIC/SOHIC-H_2S according to the API 581 standard, Table 9.3 provided the following:

- sulphur content in steel: > 0,01 %,

- heat treatment: PWHT,

- environmental severity: high.

Thus, the conclusions on susceptibility of cracking were as follows:

- in the regions of welded joints that connect sections of the pipeline (e.g. connection of a straight with T-tube) – susceptibility to cracking is high,

- for the native material of the pipeline – susceptibility to cracking is low.

Susceptibility to SSC analysis according to the API 581 standard, Table 8.3 provided the following outcome:

- maximum Brinell hardness > 200, experimental hardness: 150,

- welded joints heat treatment: PWHT,

- environmental severity: high.

In conclusion, the material is not susceptible to SSC.

The analysis performed in accordance with the API 581 standard indicated high risk of cracking and hydrogen blistering in the areas of welded joints that connect sections of the pipeline. In the heat affected zone there is practically no risk of SCC, whereas the risk of wet H_2S degradation in the native material is low.

Due to a potential risk of HIC/SOHIC-H_2S process occurrence, the metallographic tests were carried out on the samples collected from welded joint material. Metallographic microsections were examined with the metallographic microscope. The photographs of microsections are shown in Figure 5.

Figure 5a, b

Metallographic microsections indicate the degradation mechanism called Hydrogen Induced Cracking (HIC). The presence of atomic hydrogen inside the welded joint material leads to an increase in internal stress, which results in a crack formation. The crack has a distinct, graduated, step-like appearance, which is common for this type of degradation mechanism. Despite the crack was found on the surface of the microsections, the cracking process was not a direct cause of the failure. Probably the cracking process was significantly impeded in the heat affected zone due to the PWHT treatment, which has considerably reduced hydrogen absorption inside the metal (Table 4).

3.4. Corrosion risk assessment

Determination of the stream's flow rate is a relevant factor in corrosion risk assessment, since the flow rate has a significant impact on a corrosion rate, especially in ammonia streams, as well as in acidic and alkaline waters in refinery plants. Regarding the mass balance, pressure value and inner pipeline diameter, the flow rate was 11 m/s for each of four pipes in the area where the failure had occurred. Such high flow rate is a consequence of a two-phase flow in this part of the unit (vapour and aqueous phase). In order to assess erosive conditions, the calculating procedure in accordance with the API RP-14E [21] standard was applied. The threshold value for erosion (for the value of factor C = 100) was 62.3 m/s. Therefore, the flow rate can be considered too low for the erosion processes to occur. However, such high flow rate can considerably impact corrosion processes.

Most likely, the dominant corrosion mechanism is Alkaline Sour Water Corrosion (environment pH > 7). The aqueous phase of the stream contains significant amount of hydrogen sulphide and ammonia, which form unstable chemical compound – ammonium bisulphide NH_4HS. Due to stream temperature (slightly above 30 °C) this compound is stable in the aqueous solution and causes corrosion attacks. Corrosion rate is strongly related with the flow rate. The rate of corrosion was determined by the calculation method according to the API 581 standard. [13]

Data for calculation obtained in computer simulation in the Aspen Hysys software included:

- flow rate: 11.1 m/s,

- NH_4HS concentration: 2.8 %,

- stream temperature: > 66 °C.

Based on extrapolation of the data from Table 2.B.7.2M [13], corrosion rate was 0.53 mm/year. Because the value of flow rate exceeds 6.1 m/s, localised corrosion will occur (Table 2.B.1.2). [13]

Another corrosion mechanism which impacts corrosion rate is Amine Corrosion. The content of amine in the stream is low, however, it contains considerable amount of thermostable slats. The rate of corrosion depends on a ratio of acid gases content to amine concentration (acid gas loading), stream temperature, flow rate and the amount of thermostable salts. [13] Due to very low amine content, as compared to high amount of acid gases (CO_2 and H_2S), it is not possible to assess corrosion rate. However, this mechanism may have impact on elevation of overall corrosion rate.

In the environment with high pH value and the presence of carbonates (caused by the presence of CO_2), the process of Alkaline Carbonate Stress Corrosion Cracking (ACSCC) can also occur. However, due to the PWHT treatment, the risk of ACSCC occurrence is low.

4 CONCLUSIONS

The intensive corrosion process, which took place was estimated on the basis of partially analytical and process data regarding corrosion rate assessment, along with the level of cracking risk. The corrosion attack was the result of synergistic effect of several factors that had occurred simultaneously. Among these factors the following ones can be specified: high flow rate, presence of acid gases – additionally containing high amount of ammonia – and presence of amine, as well as low stream temperature, in which formed NH_4HS is stable. In order to reduce corrosion rate, the following would be considered: decreasing flow rate (by changing the pipelines diameter), reducing temperature in the upper part of the regeneration column to eliminate amine flow, as well as application of steel compatible with the NACE MR0103 standard to reduce threats related to wet H_2S degradation mechanism.

AUTHOR CONTRIBUTIONS

Juliusz Orlikowski: Methodology, Investigation, Formal analysis, Visualization, Writing – review & editing; **Maciej Kalinowski:** Writing – original draft, Resources; **Igor Lasota:** Resources, Supervision; **Piotr Maruszewski:** Formal analysis, Visualization; **Michał Szociński**: Writing – original draft, Writing – review & editing; **Kazimierz Darowicki**: Conceptualization

CONFLICT OF INTEREST

The authors declare no financial or commercial conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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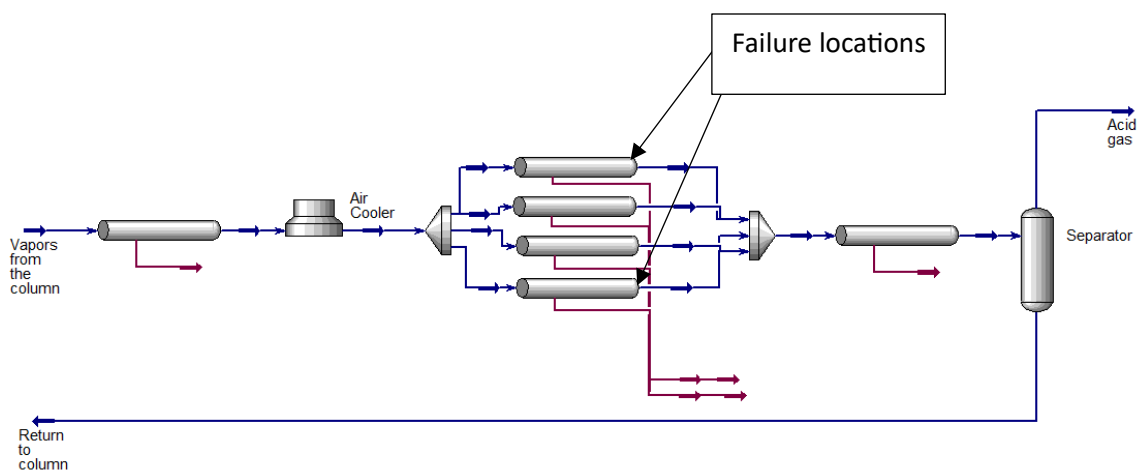
FIGURES

(a)
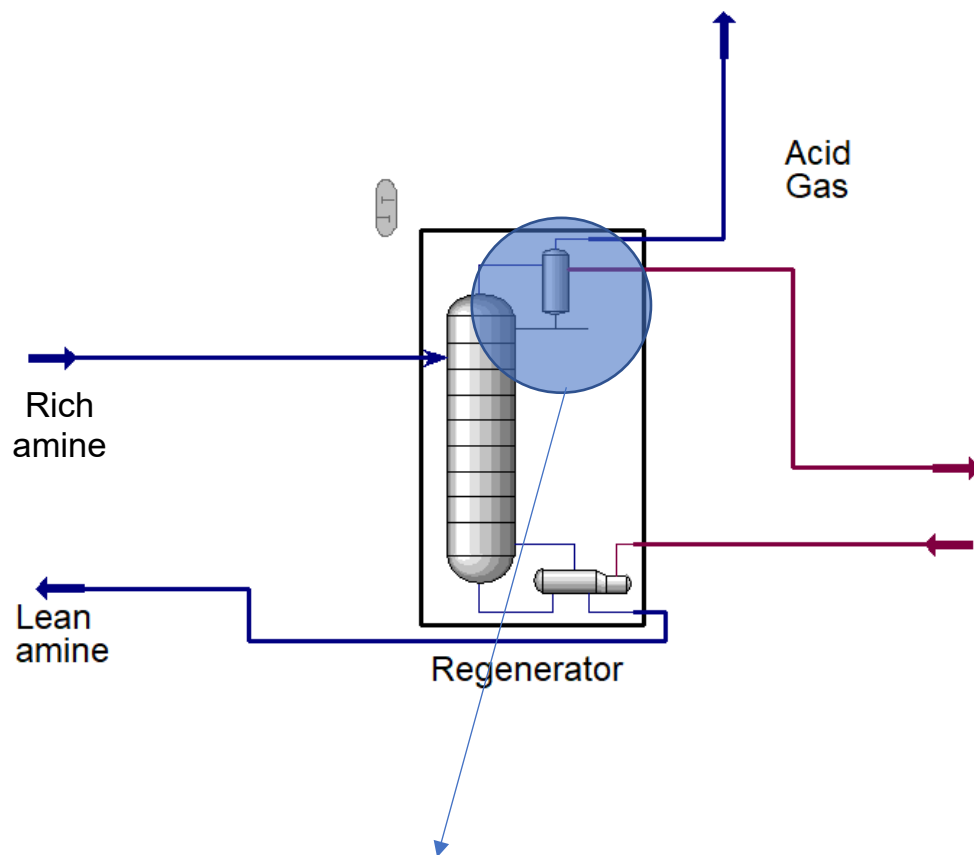
(b)

Figure 1. Simplified scheme of amine regeneration unit (a) with the areas, where the failure occurred (b – zoomed in piece of scheme 1a).
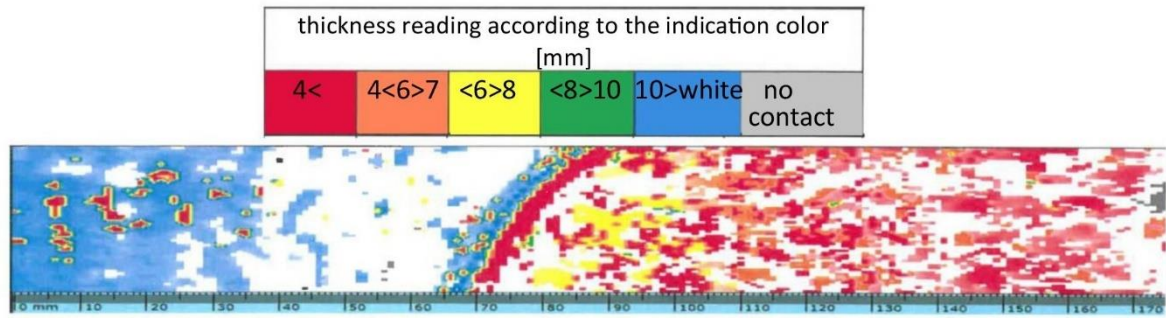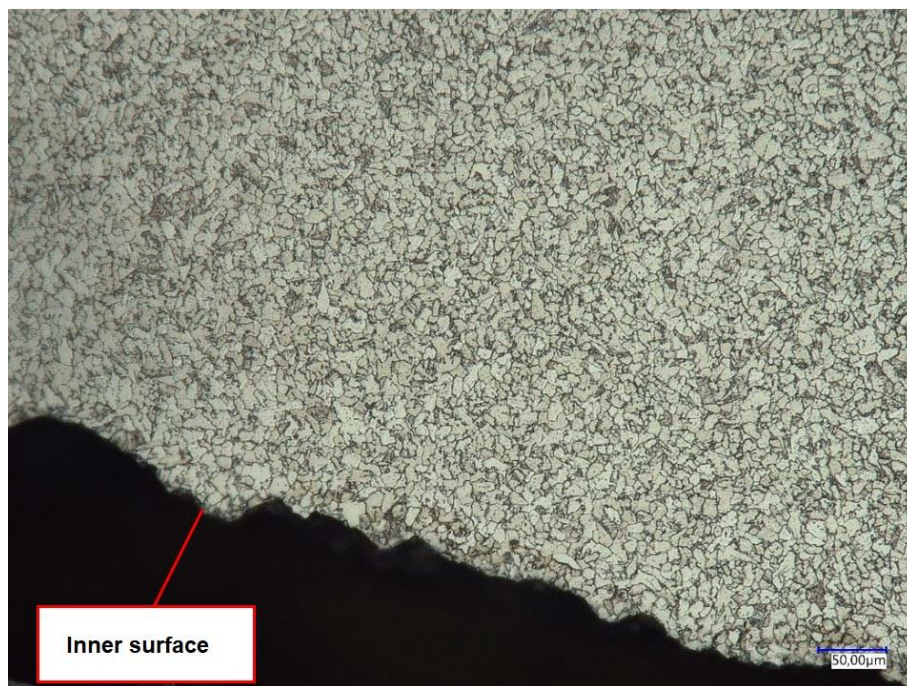
(a)

(b)

Figure 2. Photographs of external (a) and internal (b) side of the perforation in one of failed pipelines.
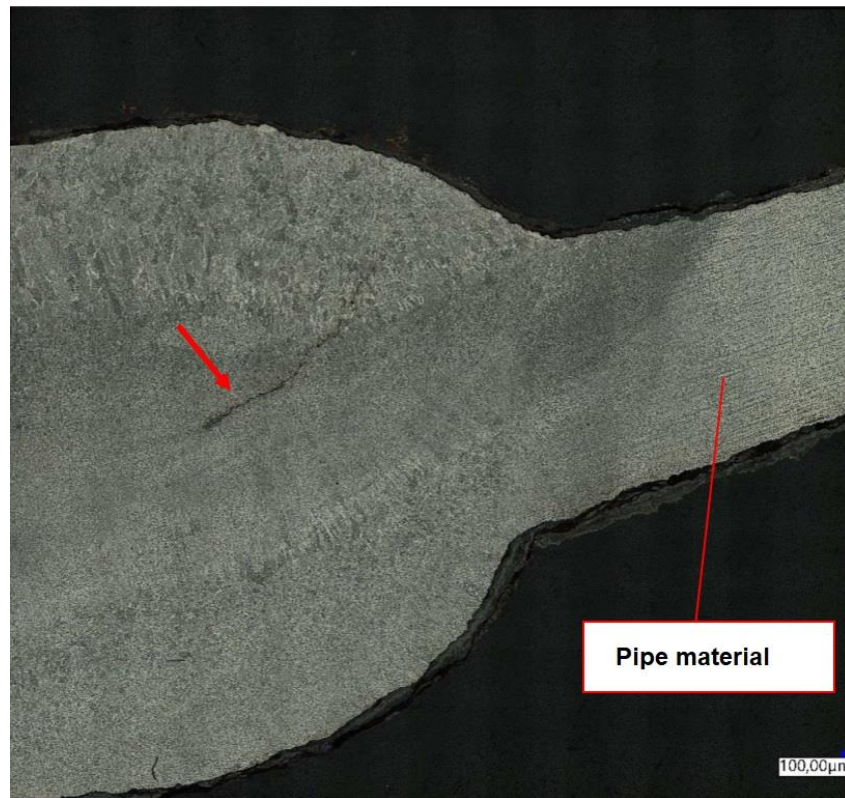
Figure 3. Results of the UT-Scan of the perforated pipeline.

Figure 4. Metallographic microsection of the native material in the area with the highest corrosion rate (internal surface).

(a)

(b)

Figure 5. Metallographic microsections of welded joint area (T-tube/straight) engulfing joint material, heat affected zone, native material of the pipeline (a) and magnified region of the identified crack (b).

TABLES

Table 1. Chemical composition of the metal samples from amine regeneration unit.

Type of the steel	Elemental composition [%]									
	C	Mn	Si	V	Cr	Cu	Ti	Ni	S	Mo
A333 gr. 6	0.3	0.89	0.09	0.07	0.05	0.17	0	0.08	< 0.01	0.03

Table 2. Chemical composition of the stream (temperature 110 – 130 °C, pressure 80 - 90 kPa) that leaves the regenerator, obtained via computer analysis.

Component	Mass fraction
H_2O	0.6239
Ammonia	0.0306
H_2S	0.0417
CO_2	0.0082
DEA	0.0150
HSAS	0.0006
Hydrocarbons, N_2, H_2	0.2800

Table 3. Mass fractions in the stream that leaves the air cooler, divided into vapour and liquid phase, obtained via computer analysis.

Component	Sum	Vapour Phase	Liquid Phase
H_2O	0.6239	0.0652	0.9076
Ammonia	0.0306	0.0249	0.0335
H_2S	0.0417	0.0689	0.0278
CO_2	0.0082	0.0096	0.0075
DEA	0.0150	0.0000	0.0227
HSAS	0.0006	0.0000	0.0009
Hydrocarbons, N_2, H_2	0.2800	0.8314	0.0000

Table 4. Hydrogen content in the material samples from failed pipeline.

Sample origin	Hydrogen content [ppm]
Welded joint	19.5
Heat affected zone	7.4
Native material	24.6

Table 5. Tensile test results for reference steel and H_2S exposed material (average values).

Material	$\sigma_{\%0.2}$ (MPa)	$\sigma_{Max.}$ (MPa)	Elongation (%)
A333 gr. 6	498	708	21
H_2S exposed A333 gr. 6	420	560	15

GRAPHICAL ABSTRACT

Any material failure in refinery and petrochemical plants is associated with health and environmental hazard as well as considerable financial loses due to periodic shut down for repair. Identification of the failure causes is crucial from the damage prevention standpoint. In the paper, the authors investigate the case of wet H_2S corrosion of a pipeline in amine regeneration system, correlating the material degradation with process parameters of the installation.