

Failure Analysis of 304 Stainless Steel Tubes of Intercooler Carbon Dioxide Compressor

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Abstract— The annual inspection of the petrochemical plant revealed that SS 304 tubes at Intercooler CO₂ Compressor have failed. Some heat exchanger tubes were found during inspection with circumferential cracks. Some analysis methods were performed to investigate heat exchanger tube failures, such as X-Ray diffraction analysis, XRF (X-ray fluorescence) analysis, hardness test, morphology analysis, and numerical analysis. Investigation results revealed that pitting corrosion has been found on the inner surface of the tube that an essential factor in initiating cracks. Moreover, some porosities were observed spread distributed on the surface sample, which is believed to reduce the mechanical and physical value of the material. The corrosion and the fact that cracks were initiated in the relatively small tensile loads sites suggest that the component failure resulted from chloride-induced stress corrosion cracking. Investigation results for heat exchanger parameters, chloride content, and temperature operation have become the main factor in initiating chloride stress corrosion cracking (CSCC). The operational condition of the heat exchanger should not be referred to by the tube material austenitic SS 304. Regarding the systems with SS 304 tube heat exchangers and chloride content

Index Terms—SS 304, chloride stress corrosion cracking, heat exchanger tube, failure analysis.

I. INTRODUCTION

Annual inspection is an essential procedure to ensure every piece of equipment works optimally, including equipment that works for heat exchange [1]. Heat exchanger equipment is a system component that transfers heat from one medium to another effectively and efficiently. Fluids pass through distinct chambers, with the walls acting as a primary heat transmission surface. Secondary heat transfer surfaces, generally in the shape of corrugated metals known as fins within the flow chamber. Air, oil, water, and coolant are heat exchanger equipment most often utilize fluids.

Although this heat management unit has been commonly used, industries that use heat exchangers always face the problem of failure in their units. They are mostly related to

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the temperature gradient, fouling, intergranular corrosion, and stress corrosion cracking (SCC) problems [2-5]. Stress corrosion cracking (SCC) is the cracking of a susceptible material produced by the presence of tensile stress and a corrosive medium simultaneously time. The interaction between environment and alloy composition on the cracking susceptibility of austenitic stainless steel in an aqueous chloride media has been the focus of considerable research [3].

A severe problem in the chemical and petrochemical sectors is the SCC of process equipment consisting of 304 and 316 stainless steel caused by chlorides. Several cases of austenitic stainless steel process equipment and components failing due to chloride SCC have been reported, and various research has been performed to solve the issue [3, 6-11].

Some fertilizer plants have been shut down for six months. Galvanic corrosion on AISI type 304 Stainless steel intercooler and aftercooler become the main cause of failure [8]. In another failure case on Austenitic stainless steel, sensitization was found on AISI 304 stainless steel tube after five years of service at a working temperature of 500°C [12]. Another author reported the failure of 304 stainless steel tubes in a gas analyzer. Stress corrosion cracking results in several cracks on the tube surface that penetrate the inner tube [13]. In addition, the effect of the presence of NaOH and Chloride ions can reduce the mechanical properties of 304 stainless steel tubes [14].

This study aimed to investigate the cause of the failure of 304 stainless steel tubes of intercooler CO₂ compressor through metallurgical and mechanical tests and numerical analysis.

II. EXPERIMENTAL PROCEDURES

Heat exchanger is one of vital equipment in petrochemical industry. Periodic checks are needed to maintain the performance of a heat exchanger.

An annual inspection of a petrochemical plant revealed that the Intercooler CO₂ Compressor's SS 304 tubes had failed. Clogged heat exchanger tubes will be due to a decrease in the performance of the heat exchanger. The circumferential crack position in the heat exchanger system can be shown in Figure 1. Around 94 tubes on the upper side were clogged with deposits, as shown in Figure 2. Commonly, clogged tubes are on the top tube of the heat exchanger.

Mechanical removal was conducted to clean the deposited material in Stainless steel 304 tubes. Furthermore, a leak test was conducted to investigate the tube leaks. One hundred forty-nine tubes were found in leaked condition after testing using compressed air at 7 kg/cm² on the middle area.

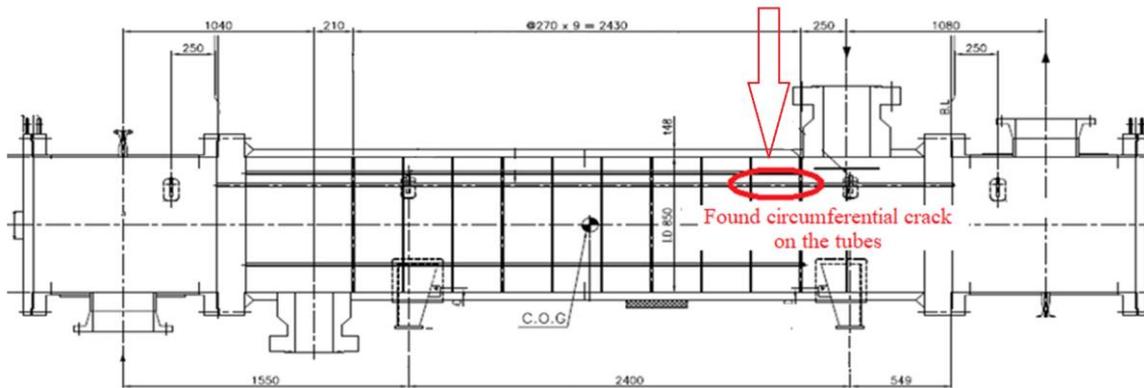


Fig. 1. Circumferential crack position in heat exchanger system.



Fig. 2. Deposits of material-clogged SS 304 tubes on the top side

Some tubes were removed from the bundle to analyze the leaking tubes. Visual observation shows that some circumferential cracks were observed on tubes. Furthermore, a dye penetrant test was carried out to investigate the tube's outer deeply. Circumferential cracks occur on the south tube sheet's 700–900 mm range. Moreover, the other tube occurred 900 and 1200 mm from the south when pulled out.

X-ray diffraction analysis, XRF (X-ray fluorescence) analysis, hardness test, morphological analysis, and numerical analysis were all used to evaluate heat exchanger tube failures.

III. RESULTS AND DISCUSSION

In this work, the analysis begins by analyzing the deposits contained in the SS 304 tubes. Deposit material composition in clogged pipes can be shown in Table 1. the main composition of the deposited material is silicon dioxide (31.88%). Based on the analysis results, the water used is believed to have been polluted by fly ash from the generator located near the heat exchanger system. The high content of SiO₂ evidences this. SiO₂ is the main constituent component of fly ash.

TABLE 1.

DEPOSITS MATERIAL COMPOSITION ON SS 304 TUBES

Element	C	Ni	Cr	Fe	SiO ₂	PO ₄
Wt%	9.1 %	0.48 %	0.06 %	18.2 %	31.88 %	2.49 %

Analysis of operational conditions on the tube exchange system was performed using numerical analysis to determine the heat exchanger tube temperature profile when operating.

A short simulation was performed to investigate the

operating condition of the heat exchanger. Pressure and temperature were selected for input parameters due to having a significant contribution to failure. Figure 3 shows the analyzed heat exchanger model.

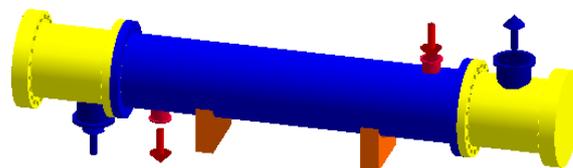


Fig. 3. Analyzed heat exchanger model

Figure 4. shows the numerical analysis result of the temperature gradient shell of the heat exchanger. Shell temperature increases significantly from the outlet heat exchanger. Moreover, the temperature gradient tube rises with increasing distance from the outlet, as shown in Figure 5.

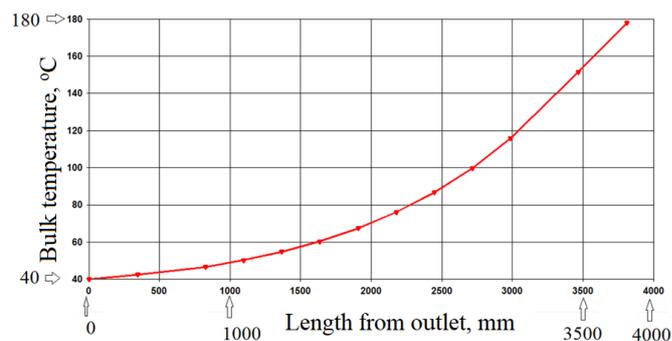


Fig. 4. Temperature gradient shell side

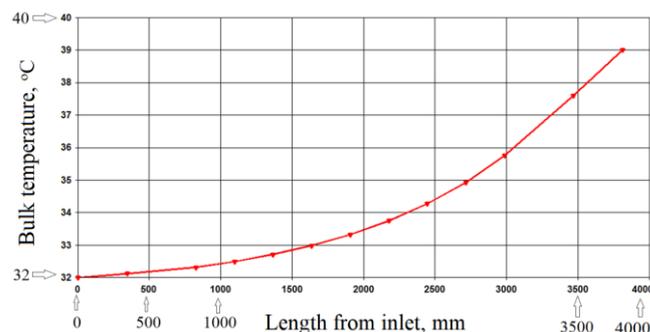


Fig. 5. Temperature gradient tube side

Circumferential cracks positioned on the tube were found between 3480 to 3750 mm. Let's take the length of 3500 mm as a sample position. According to Figures 4 and 5, at the current position, the bulk temperature of the shell side is 155 °C, and the tube side is 37.7 °C. It is assumed that the heat transfer convection coefficient of water flowing in the tube is approximated at 500 to 1200 W/m²K and 10 to 350 W/m²K for gas flow between tubes. The temperature distribution inside the tube wall between 77.7 to 82.6 °C is seen in Figure 6.

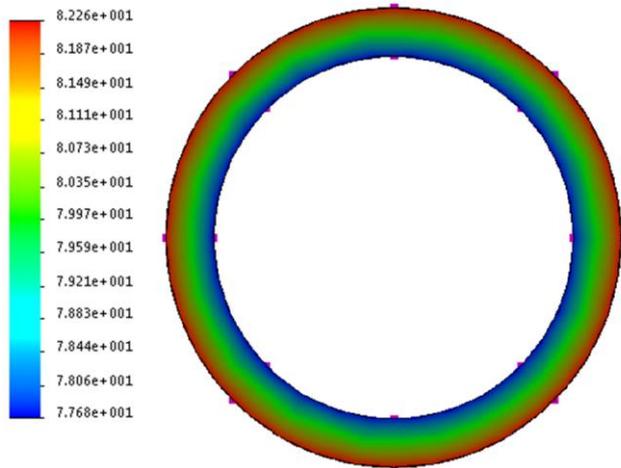


Fig. 6. Temperature distribution inside tube wall

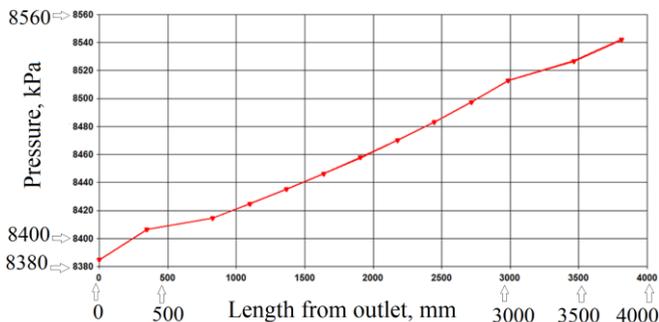


Fig. 7. Pressure Gradient shell side

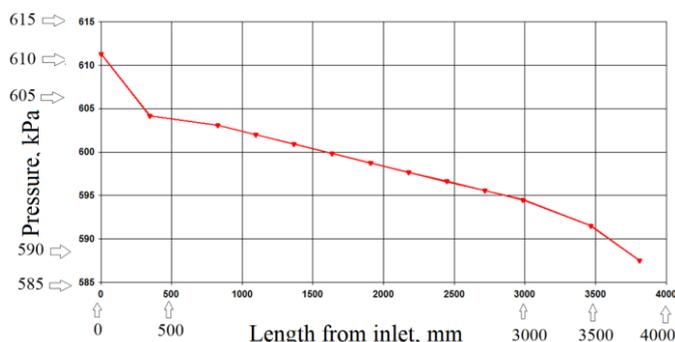


Fig. 8. Pressure Gradient tube side

Figure 7 shows that the pressure increases with the length; at 3500 mm, the pressure was obtained at 8530 kPa. On the other hand, the pressure tube side decreases with the length, as shown in Figure 8. At a position of 3500 mm, a stress analysis is performed with the higher stress distribution located inside the tube wall, as shown in Figure 9.

Operational conditions play an essential role in triggering the occurrent failure mode. Some factors affecting pitting

corrosion include environmental and surface conditions, metal composition, and temperature, besides critical factors such as ion concentration and pH.

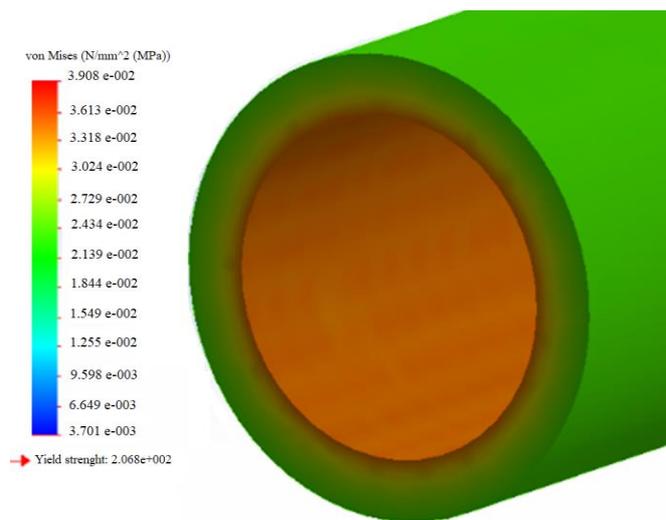


Fig. 9. Stress analysis tube side

In the austenitic stainless-steel case, some factors significantly determine failure modes, such as stress, temperature, chloride concentration, and pH. Referring from API Recommended Practice 581. 2016 [18].

Table 2 shows the susceptibility to cracking chloride stress corrosion cracking, referring to the tube heat exchanger operation condition on 4. It can be categorized as very high conditions susceptible to forming chloride stress corrosion cracking.

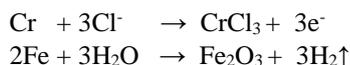
In this case, chloride content in the water of the tube heat exchanger is around 44-68 ppm, and the maximum temperature operation is about 170°C, as shown in Table 3. Based on API Recommended Practice 581. 2016 [18] that tube exchanger operation in the critical zone has passed the A Chloride stress corrosion cracking (CLSCC) thresholds of 304 stainless steels.

TABLE 2. SUSCEPTIBILITY TO CRACKING-CHLORIDE STRESS CORROSION CRACKING (CLSCC) [8].

pH≤10				
Temperature (°C)	Susceptibility to Cracking as a Function of Chloride Ion (ppm)			
	1-10	11-100	101-1000	>1000
≤ 38	Low	Low	Low	Medium
>38 - 66	Low	Medium	Medium	High
>66-93	Medium	Medium	High	High
>93-149	Medium	High	High	High
>149	High	High	High	High
pH>10				
Temperature (°C)	Susceptibility to Cracking as a Function of Chloride Ion (ppm)			
	1-10	11-100	101-1000	>1000
≤ 38	None	None	None	None
>38 - 93	Low	Low	Low	Low
>93-149	Low	Low	Low	Medium
>149	Medium	Medium	Medium	High

The main water pollutant that attacks stainless steel is chloride. Chloride breaks down the passive layer by dissolving chromium and allowing the combination of water with iron to produce the red oxide Fe₂O₃, often known as

hematite. There seem to be two stages to the chemical process. The first includes the oxidation of iron following the dissolution of the passive layer. Furthermore, the second involves the dissolution of chromium by the chloride ion.



X-ray diffraction analysis is a common means to determine the structure and relative configuration of solid compounds in a certain way. XRD result of the tube material is shown in Figure 10. XRD analysis shows another peak in the tube material sample was not found. Regarding this result, the transformation phase is not occurring on tube material. In other words, the material does not experience a state where it can change its phases, such as high temperatures or mechanical processes.

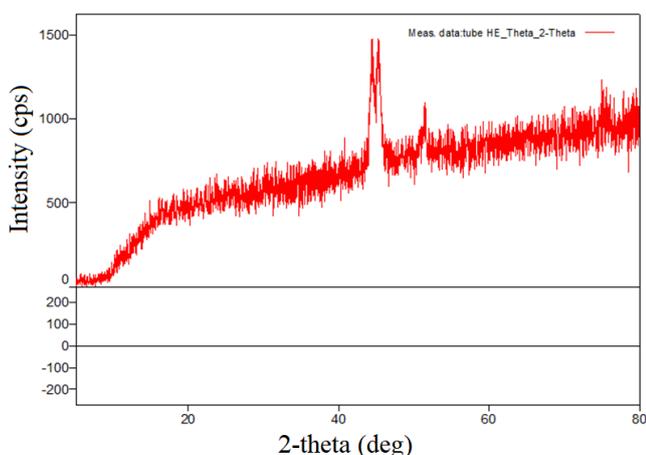


Fig. 10. X-ray diffraction result for tubes material

The initial investigation of failure is to analyze the composition of the tube. XRF (X-ray fluorescence) was utilized to analyze the composition of failure tubes. XRF results as shown in Table 3. Based on the XRF result, the tubes that have the second major element after Fe are Cr and Ni for 18.49 % and 8.06 %, respectively, according to SS 304

TABLE 3.
COMPOSITION OF FAILURE TUBE

Element	Cr	Fe	Ni	Cu	Mo
Content	18.49	72.11	8.06	0.333	0.114

Hardness measurement for SS 304 using the Vickers method at the tube cross-section. The hardness measurement result shows no significant difference in hardness value compared to the ASM Material Data Sheet for SS 304, as shown in Table 4.

TABLE 4.
HARDNESS RESULT USING VICKERS METHOD

No	VHN	Remark
1.	189	ASM Material Data Sheet
2.	198.4	Measurement results
	180.2	
	188.9	
	174.8	

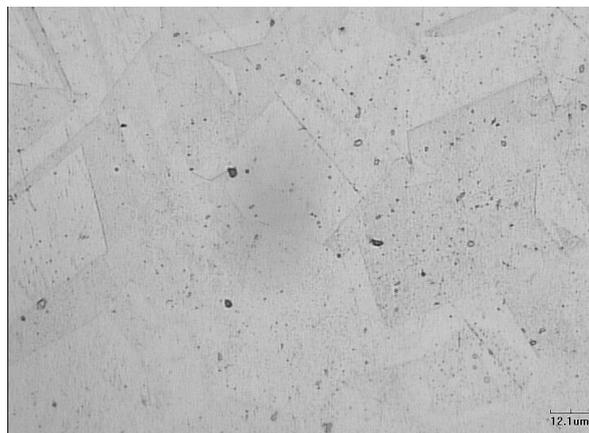


Fig. 11. Microstructure of austenitic stainless steel 304 tube

Morphology observation was carried out utilized by the digital microscope and optical microscope. Figure 11 revealed an austenite structure, characterized as austenitic stainless steel 304.

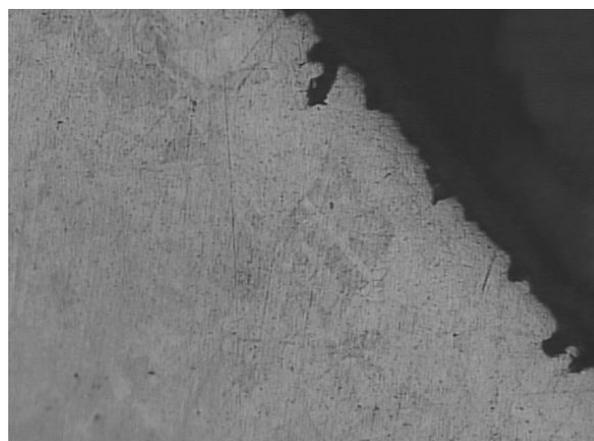


Fig. 12. Microstructure of austenitic stainless steel 304 with some porosity on the surface.

Moreover, porosities and pitting corrosion were evenly distributed on the surface sample, as shown in Figures 11 and 12. The occurrence of porosity was believed to be due to manufacturing results. On the other hand, pitting corrosion occurs due to unsuitable conditions for SS 304.

Figure 13 show some cracks found on the tube's inner surface, which were believed to be stress corrosion cracking. All of the cracking has a similar pattern (circumferential crack).

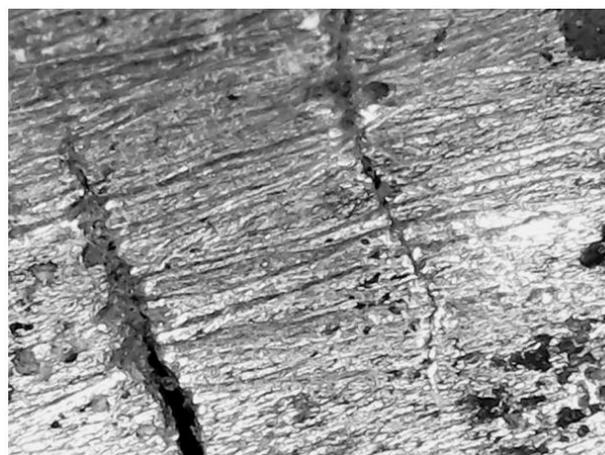


Fig. 13. Inner surface morphology of tube

In the shutdown period, CLSCC can appear when a solution contains chlorides, especially at temperatures above 60 °C. In general, CLSCC has a transgranular formation and many branches. Stainless steel is very susceptible to CLSCC of Austenitic type with a nickel content of 8%, such as Type 300 series, stainless steel 304, and 316. Lower or higher nickel content alloys generally indicate the resistance of stainless steel to higher CLSCC. The CLSCC commonly does not influence duplex stainless steels with a low nickel content like it does alloys with nickel content above 42%.

If the required electrochemical, mechanical, and metallurgical conditions are fulfilled, cracks may initiate already-existing surface flaws, or corrosion processes may generate a surface imperfection through pitting or localized corrosion. Pits, manufacturing defects, and intergranular corrosion, which all contribute to SCC start, are visible and identifiable, as demonstrated in Figure 14 pitting corrosion in an inner tube.



Fig. 14. Cracks start from corrosion pits.

Pitting corrosion is critical in initiating failure, especially under complex conditions such as pressure and temperature. In this case, some cracks were found in the inner surface of the tube. Crack initiation and propagation are different but highly related processes; when the crack has been initiated, it will propagate.

Austenitic stainless-steel components are susceptible to pitting and crevice corrosion in wet, humid environments containing chloride ions. Components with residual stress may crack due to stress corrosion under these conditions. A vessel or piece of pipework may become perforated due to pitting, simply the breakdown of the chromium oxide layer followed by localized corrosion that results in pits. Instead of the macroscopic physical characteristics of a component, pitting is mostly related to the microscopic heterogeneities of a surface. Localized corrosion is followed by breaking the chromium oxide layer in crevices. However, unlike pitting, it only occurs at specific physical features when a partially shielded surface and a stagnant solution are present at the covered area's interface.

Figure 15 shows SEM analysis of the crack on the inner surface of the tube. Morphology analysis for the cracks revealed that the fracture surface is characterized as transgranular fracture, which is the crack propagation along grain boundaries.

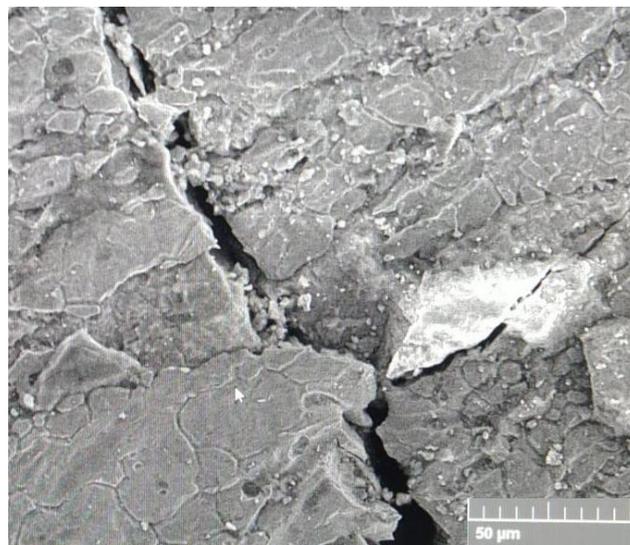


Fig. 15. SEM micrographs of Chloride stress corrosion cracking.

In SCC- and stress-dominated regions, transgranular cracking exhibited a greater elongation rate and higher failure times. In contrast, the failure in intergranular cracking was gradual and frequently occurred in the steady-state elongation area, indicating a minimum of mechanical elongation prior to failure. Intergranular cracking for these austenitic stainless steels consequently showed a more brittle than transgranular cracking.

Transgranular cracking, on the other hand, is unrelated to martensite generated at grain boundaries and is brought on by the growth of fractures initiated at slip steps. Thus, transgranular cracking like this is considered stress corrosion cracking (SCC) in the strict definition of the term.

At lower temperatures, Type 304 experienced intergranular cracking, and at higher temperatures, transgranular cracking. The formation of martensite and the competition between the material's dissolution at slip stages, which induces SCC, and hydrogen entrance, which causes HE, determines the cracking mode. The difference between hydrogen absorption and hydrogen escape determines the hydrogen entry. For type 304, transgranular SCC was generated by spreading fractures that started at slip steps rather than martensite grain boundaries. The cracking mode would thus be determined by the competition between the dissolving rate at slip stages and the hydrogen entry rate at grain boundaries with martensite.

According to a commonly understood literature review, the time to failure in samples subjected to SCC reduces as the applied tensile stress increases [15-17]. According to data, increased applied tensile stress does not result in accelerated fracture propagation or higher crack density. Since stress is thought to play a crucial role in breaking the oxide coating that shields the bare metal, higher applied tensile stress should increase fracture density and a shorter time to failure.

Environmental cracking of 300 Series SS and various nickel-based alloys under stress, temperature, and an aqueous chloride environment promotes surface-initiated cracks. The presence of dissolved oxygen increases the potential to crack [18].

IV. CONCLUSION

The investigation has been conducted to analyze the failure of SS 304 tubes at the Intercooler CO2 Compressor. Some significant results can be concluded. Based on analysis using XRF (X-ray fluorescence), X-Ray diffraction analysis, and hardness measurement, tube heat exchanger material is according to SS 304.

Visual observations reveal that pitting corrosion has been found on the tube's inner surface, an essential factor in initiating cracks. Moreover, porosities were observed to spread and distribute on the sample surface, which is believed to reduce the mechanical and physical value of the material. Cracks start from the tube's inner surface to the outer surface in the circumferential direction.

The presence of corrosion and the fact that cracks developed in the areas of relatively light tensile loads imply that the component failed as a result of chloride-induced stress corrosion cracking. Investigation results for heat exchanger parameters, chloride content, and temperature operation have become the main factor in initiating chloride stress corrosion cracking (CSCC). The operational condition of the heat exchanger should not be referred to by the tube material austenitic SS 304. According to the heat exchanger system's operating temperature and chloride concentration.

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