

Failure Analysis of Heat-Exchanger Sheets at Coke Oven

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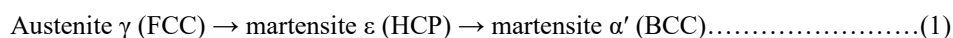
Abstract

The heat exchanger sheets at JSW Steel coke oven is 316L used in ammonia stripping / distillation waste water plate heat exchanger (PHE) with the waste water inlet temperature 110°C and outlet temperature 40°C, with water temperature between 35-43°C. The solution include 250mg/liter Ammonical Nitrogen open to atmosphere. The sheets of two heat exchangers, showed pitting/pin holes all over the plates in just 14 days of use after commissioning. Chemical composition of the sheets showed lower Ni (9.3-9.6% against 10%) than the specification. Varying hardness (189-249 Hv0.5 kgf) on the sheets indicating different phases present on the sheets. The analysis revealed the pits are from the top surface (extrusion side) of the plates. SEM-EDS revealed presence of chlorine on the plate, leading to pitting corrosion of the plates. The sheets were checked for their magnetic properties by a permanent magnet showed attracting nature and with a NDE magnetic device the sheets showed magnetic hysteresis loop, indicating presence of magnetic phase (deformation induced martensite) in the sheets. It is inferred that, martensitic transformation from the austenite due to severe plastic deformation has occurred at the manufacturing stage. Hence, galvanic effect caused by the presence of two distinct phases, austenite and martensite, which exhibit different corrosion potentials lead to formation of pitting corrosion due to presence of higher level of chlorides in the water.

Keywords: sheet type heat exchanger, failure analysis, pitting corrosion, magnetic properties

1. Introduction

A heat exchanger is a device that transfers heat between two or more fluids that are typically at different temperatures, but remain physically separated. This process allows one fluid to gain heat while the other loses it, enabling applications such as heating, cooling, and recovering waste heat from industrial processes or mechanical systems (<https://www.sciencedirect.com/topics/chemistry/heat-exchanger>). A plate heat exchanger is a type of heat exchanger that uses a series of thin, parallel metal plates to facilitate heat transfer between two fluids (Willem Faes, Steven Lecompte, Zaaquib Yunus Ahmed, Johan Van Bael, Robbe Salenbien, Kim Verbeken & Michel De Paepe, 2019). The design maximizes the surface area for heat exchange, making it efficient and compact. These plates are typically corrugated or have pressed patterns to encourage turbulence and improve heat transfer. For plate heat exchangers stainless steel is a common material, however for the high efficiency low thickness of the plates makes them extremely vulnerable to local corrosion attacks in chloride containing water (Georgii Vasyliiev, Ihor Pylypenko, Oleg Kuzmenko & Yuriy Gerasymenko, 2022). The austenitic stainless steels under plastic deformation transformed to martensite as given below:



The amount of deformation induced martensite depends upon several factors such as: temperature, plastic strain, strain rate, stress state, deformation mode, grain size, and grain orientation. The increase of α' martensite by martensitic transformation induced by plastic deformation causes a change in physical properties of austenitic stainless steels. The martensitic transformation causes the rupture of the passive film due to higher density of flaws and generates residual stress or a galvanic effect caused by the presence of two distinct phases, austenite

and martensite, which exhibit different corrosion potentials. The results show that corrosion rate is function of stress level and amount of dissolved chloride ions in water. SCC resistance of tested samples depends on the magnitude of cold work at surface layers; high level of cold work decreases corrosion resistance. The design and production of plate heat exchangers, which are cyclically loaded parts, made of AISI 316 austenitic stainless steel demands a substantial understanding of the correlation between fatigue life, martensitic transformation and damage mechanisms (Nicolae Solomon & Iulia Solomon, 2017).

The chloride/chlorine contents in the cooling tower system have caused pitting corrosion (perforation) of PHE's 316L stainless steel plates on the open circuit side (CT water side). The 316L plate samples also registered very low pitting corrosion resistance in CT feed and CT bleed water during electrochemical testing. The electrochemical results proved the chloride level as a damaging entity. There was complete non-compliance of sound scientific knowledge and practice of the cooling water treatment especially chloride concentration, which cannot be ignored even at ppm level. Hence, water of reverse osmosis (R/O) quality should be used in the CT feed and qualification of feed water system must focus on chloride contents rather than TDS values (K.M. Deen, M.A. Virk, C.I. Haque, R. Ahmad & I.H. Khan, 2010).

The principal causes of corrosion failure in the plate heat exchanger (PHE) of Jam Petrochemical Complex (JPC), Asalouyeh, Iran, showed chloride ions build-up in crevices formed between plates and gaskets (at high temperatures), is the main reason for the occurrence of SCC. The suspended soil of the service water settled in the crevices between gasket and plate and provides a favorable position (dead zone) for building-up of the chloride ions. When the level of chloride ions in the water is low, the evaporation of water in the crevices leads to chloride ions build-up. Moreover, the high temperature of the transport water outlet and cooling water inlet results in the acceleration of this building-up. By chloride ions concentration in the crevices, the pitting corrosion under gasket seat occurs. In addition, the simultaneous presence of chloride and sulfide ions intensifies the SCC failure in the heat exchanger plates. Since the appearance of the cracks in the heat exchangers occurs after cooling the TW outlet by the service water (SW), the use of higher number of the plates in the heat exchangers together with the higher flow rate of cooling water is strongly recommended; in this way, at a constant flow rate of transport water, not only the temperature of transport water is decreased but also the SCC cracks resulted from the direct contact of the service water (which usually contains some levels of Cl^-) can be effectively avoided (S.H. Khodamorad, N. Alinezhad, D. Haghshenas Fatmehsari & K. Ghahtan, 2016).

2. Materials and Methods

Sheets of 316 L were received from the JSW Steel coke oven for characterization to sort out the root cause of leakage. The sheets were visually tested for the presence of defects and measured the thickness. The steels were evaluated for their chemical composition through a SPECTRO make optical emission spectroscopy. The sheets were evaluated for their micro hardness through a micro Vicker's hardness tested at a load of 0.5kg. The optical microscopy through a opto-digital Olympus make microscope and scanning electron microscopy (SEM) through a Hitachi make SEM were conducted for the microstructure and defect analysis. SEM-EDS was conducted for the presence of inclusion and corrosion product identification. A permanent magnet and a portable NDE magnetic device (*MagStar*) were used for the magnetic property and magnetic hysteresis loop measurement of the sheets (http://www.nmlindia.org/download/Tech_HB/files/assets/downloads/page0026.pdf; <ftp://ftp.technofour.com/pub/catalogs/MagStar.pdf>). The MHL measurement was carried out at a magnetizing field of 1000Oe and magnetizing frequency of 50 mHz.

3. Results and Discussion

3.1 Visual Examination, Chemical Composition and Hardness

The sheets with the top (extrusion side) and bottom (intrusion side) shown in Figure 1. Pitting marks can be seen on the sheets surface responsible for the leakages.

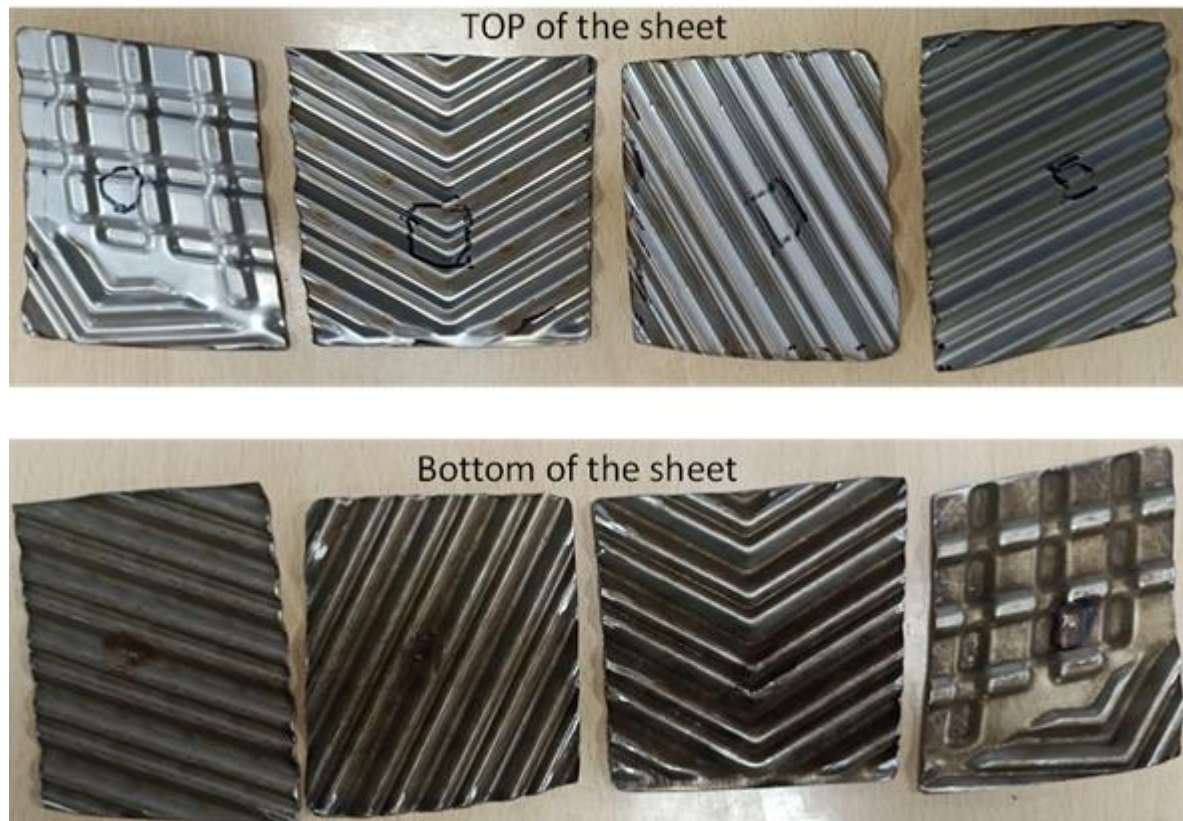


Figure 1. Heat exchanger plates received from JSW coke oven with pitting marks on it

Chemical composition of the sheets is shown in Table 1. The chemical composition shows a small deviation in Ni content in the sheets (lower-9.3-9.6 wt. % Ni) compared to the specification (10-13%). Thickness of the sheets measured through SEM is shown in Figure 2. The thickness of the sheets were found to be in the range of 0.592-0.601mm. Micro hardness of the sheets are shown in Table 2. As per the specification the hardness has to be 217 BHN which is 222Hv0.5kgf. It can be seen that the hardness is different at different regions varying in the range of 189-249 Hv0.5kgf, indicating presence of hard and soft phases in the sheet leading to different level of hardness.

Table 1. Chemical composition of the heat exchanger sheets comparison with 316L

Sample ID	C	Si	Mn	P	S	Cr	Ni	Mo	Al	Cu	Ti	V	N	W	Fe
316L	0.03	1	2	0.045	0.015	16.50-18.50	10.0-13.0	2.0-2.5					0.1		
Sample-1	0.014	0.397	1.416	0.037	0.008	17.421	9.642	2.499	0.000	0.108	0.000	0.089	-	0.025	68.344
Sample-2	0.014	0.397	1.415	0.034	0.007	17.484	9.270	2.445	0.000	0.109	0.000	0.091	-	0.026	68.708
Sample-3	0.014	0.399	1.423	0.035	0.007	17.485	9.313	2.431	0.000	0.109	0.000	0.091	-	0.027	68.666

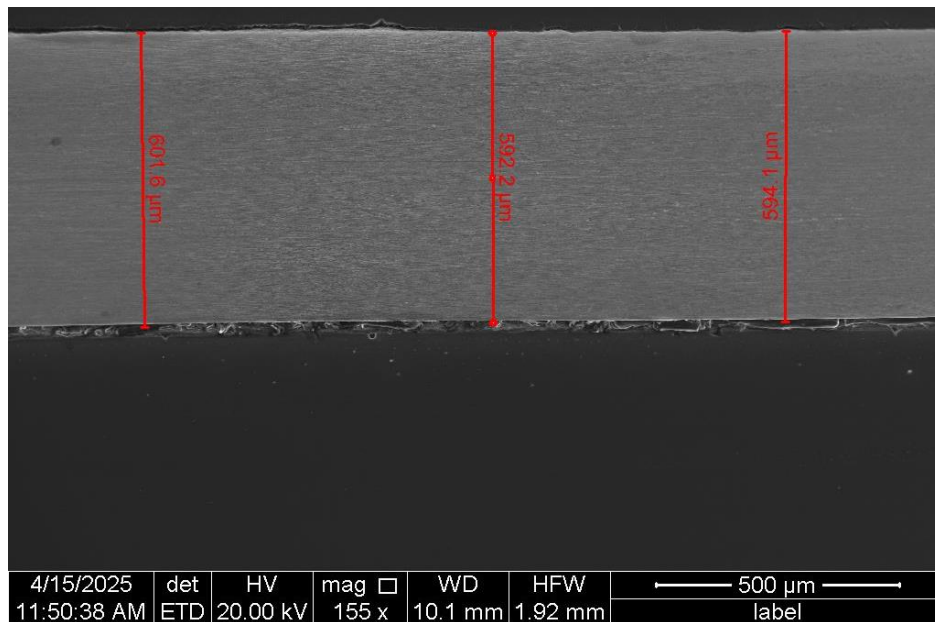


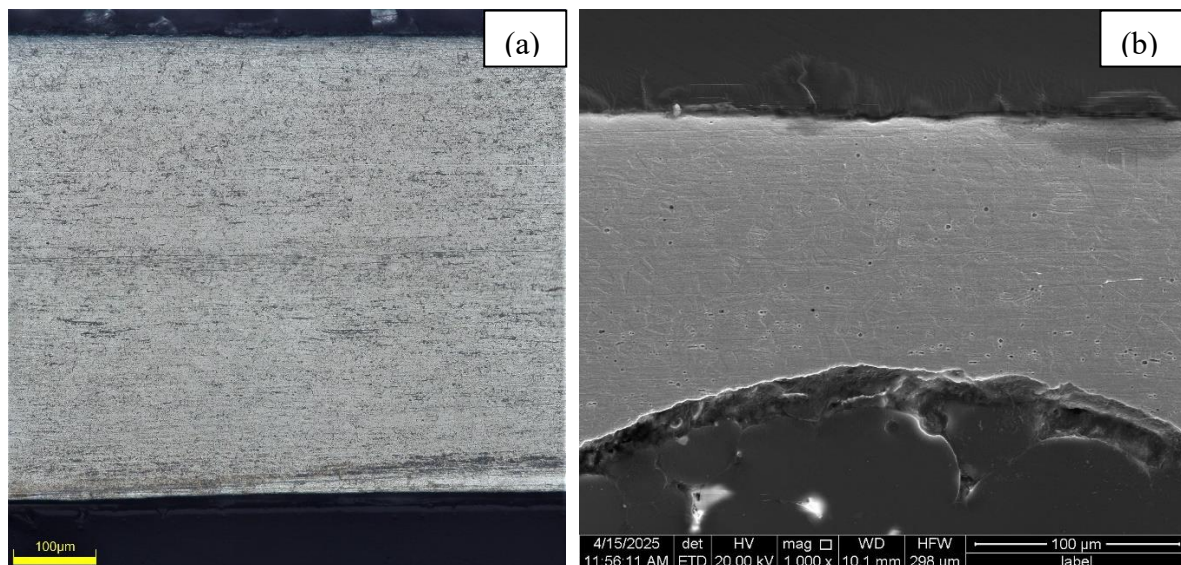
Figure 2. Thickness of the heat exchanger sheets measured through SEM

Table 2. Micro hardness of the sheet

SL NO	Weight - 0.5kg
1	189 Hv
2	211Hv
3	249Hv

3.2 Microstructure

Microstructure of the sheet is shown in Figure 3 (a) with the SEM micrographs in (b) and (c) respectively. Small pits can be seen on the sheets at high magnification SEM micrograph (Figure 3(c)). Corrosion pits can be seen in un-etched and etched condition as shown in Figure 4 (a) and (b) respectively. SEM micrograph of the sheets with the corrosion pits are shown in Figure 5. It can be seen that some of the pits are not having through hole whereas some are having through holes.



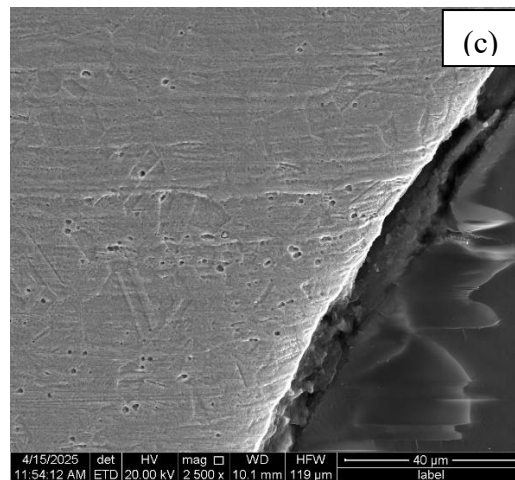


Figure 3. (a) Optical microstructure, (b) SEM micrograph and (c) high magnification SEM micrograph of the heat exchanger sheets

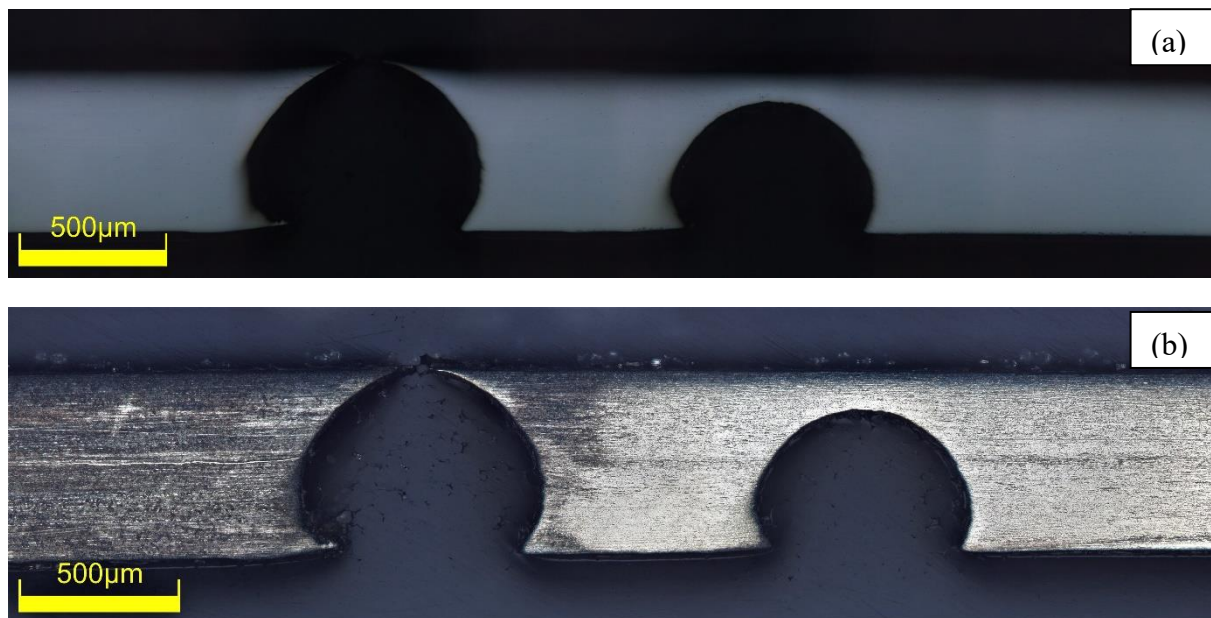


Figure 4. Corrosion pits on the sheet (a) un-etched and (b) etched condition

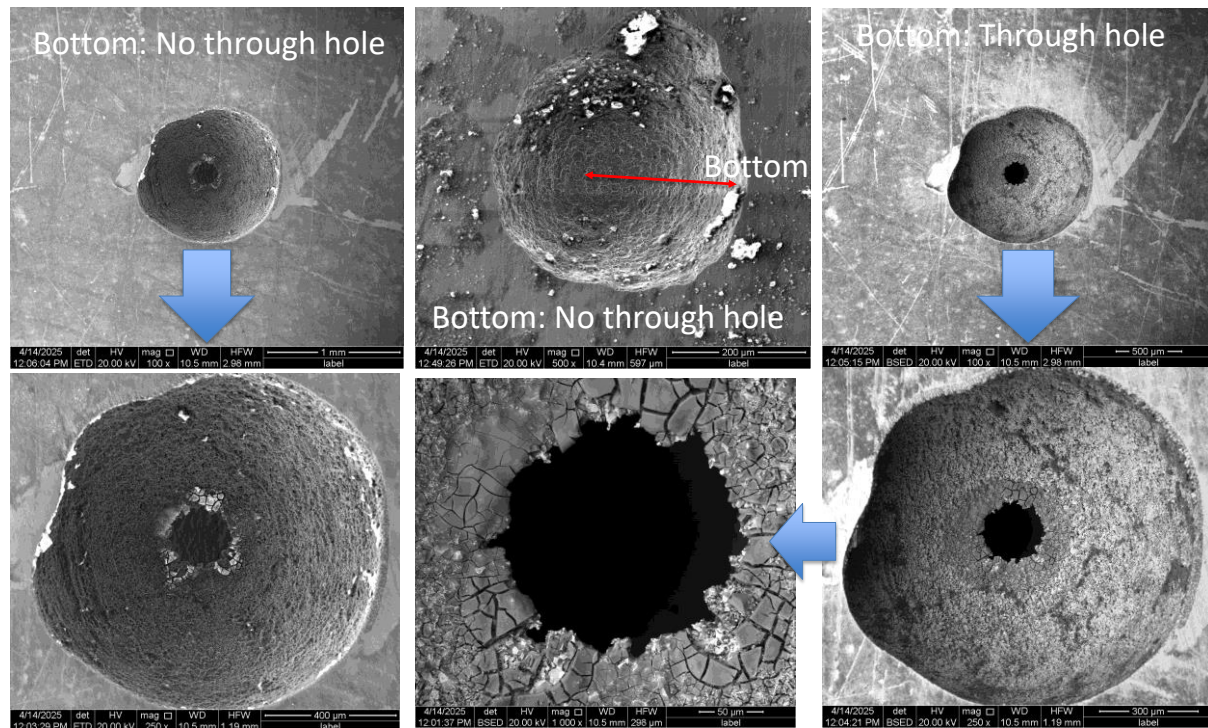
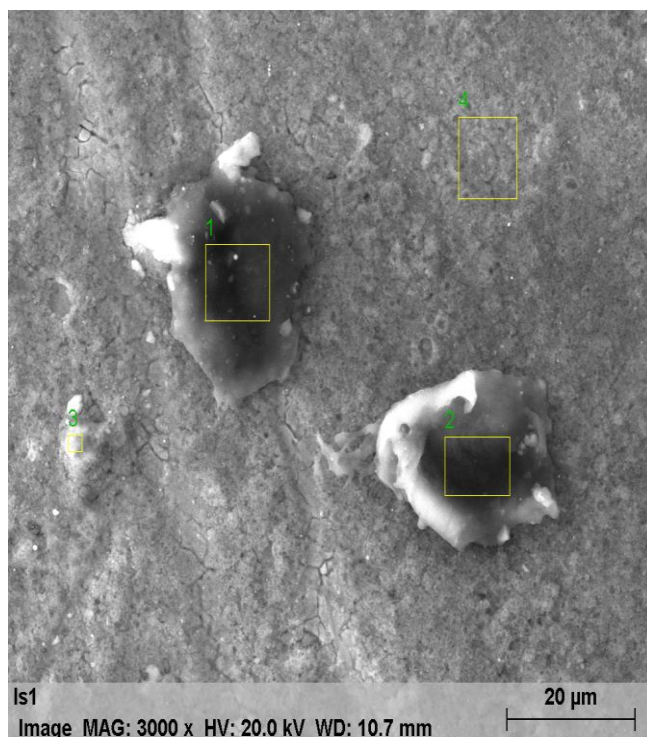


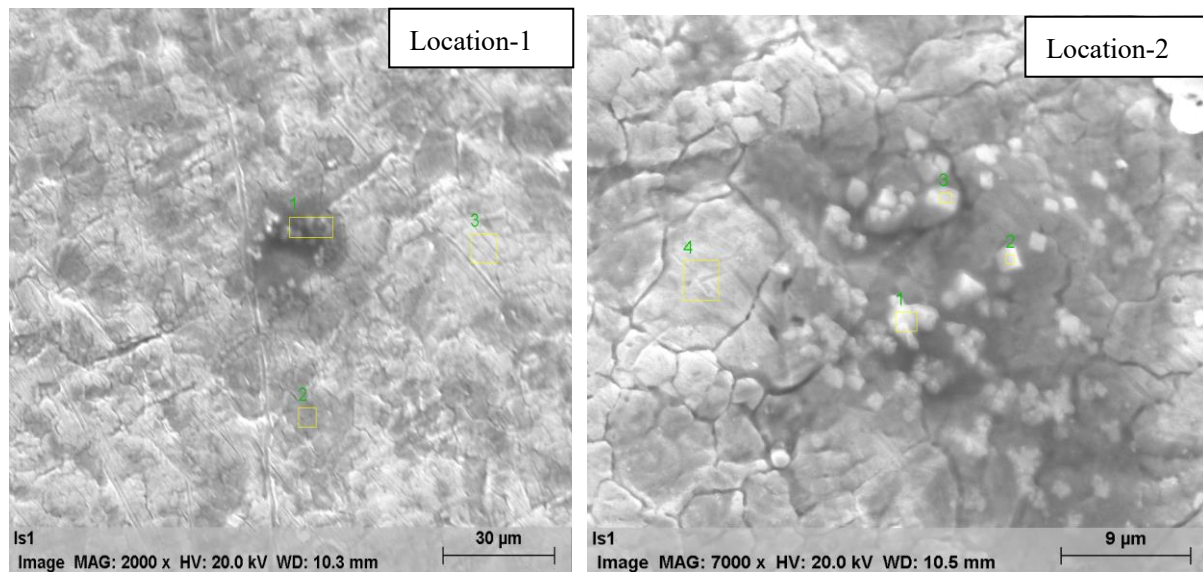
Figure 5. SEM micrograph of the pits with through hole on the sheet and partial pits

SEM EDS of the on the sheets near to the pits and away from the pits are shown in Figure 6 and 7 respectively. It shows presence of chloride near to the pits and such chlorides are in very high level found away from the pits. It indicates that the high level of chloride responsible for the pitting corrosion of the sheets. The source of the chloride was found to be the water. The higher level of chloride in the water leading to pitting corrosion of the sheets.



	P1	P2	P3	P4
O	6.33	8.97	10.42	5.20
Na	9.23	10.06	0.57	0
Al	1.19	1.79	2.48	0.56
Si	1.15	1.41	2.15	0.36
S	2.53	3.43	5.33	1.19
Cl	2.29	2.94	0.05	0
K	3.06	3.26	2.17	0
Ca	0.97	1.34	1.60	0
Cr	12.94	13.22	19.97	15.48
Mn	0.83	0.64	0.43	1.03
Fe	53.18	47.55	50.70	67.19
Ni	6.30	5.40	4.12	9

Figure 6. SEM-EDS analysis revealing presence of chloride on the sheet surface near to pit

**Mass percent (%)**

Spectrum	O	Na	Al	Si	S	Cl	K	Ca	Cr	Mn	Fe	Ni
1	11.46	19.01	0.35	0.28	1.11	13.32	6.98	0.88	8.08	0.77	33.38	4.37
2	2.36	-	0.50	0.46	0.85	0.00	-	-	14.83	1.04	69.72	10.24
3	0.00	-	0.01	-	0.84	0.00	-	-	15.35	-	73.23	10.57

Mass percent (%)

Spectrum	O	Na	Al	Si	S	Cl	K	Ca	Cr	Mn	Fe	Ni
1	10.14	14.55	0.94	0.77	0.93	17.01	14.99	0.47	8.07	0.41	28.20	3.52
2	4.18	5.76	0.00	-	0.75	11.34	11.41	-	10.99	0.54	48.43	6.59
3	6.53	7.51	0.03	-	0.67	14.09	14.73	-	9.53	0.49	40.98	5.44
4	0.53	-	-	-	0.77	0.00	-	-	15.89	1.23	71.18	10.40

Figure 7. SEM EDS revealing presence of high level of chloride on the sheet surfaces away from the corrosion pit

3.3 Magnetic Properties

A permanent magnet was used to check the magnetic properties of the sheets. Figure 8 shows that the sheets are getting attracted by a permanent magnet. Generally 316L steel is non-magnetic in nature due to the fully austenitic phase. As it is getting attracted by a magnet, it indicates presence of ferromagnetic phase such as martensite in the steel. Figure 9 Shows formation of hysteresis loop on the sheet with the application of cyclic magnetic field through a magnetic NDE device (*MagStar*) indicating presence of ferromagnetic phase on the sheets. Such martensites are generated due to transformation of austenite to martensite due to severe plastic deformation during the manufacturing stage. However, after deformation the sheets need to be annealed above the A_3 temperature to remove the martensites, which has not been done in the present case.

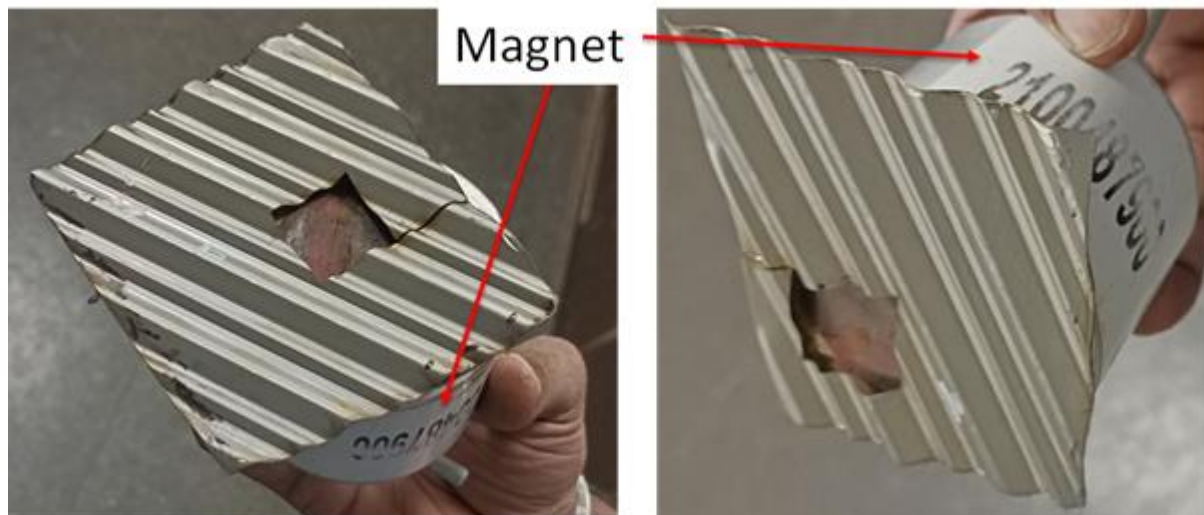


Figure 8. Permanent magnet shows attraction of the sheets indicating presence of magnetic phase (Martensite)

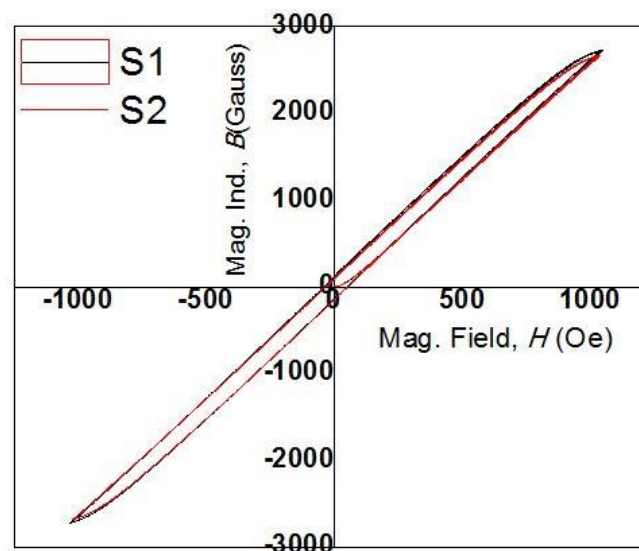


Figure 9. Hysteresis loop formed on the sheets with a portable magnetic NDE device indicating presence of ferromagnetic phase (Martensite) in the sheets

4. Conclusions

Leakage of the plate/sheet type exchanger tube were analyzed in the present study to sort out the root cause of failure in service in a very short period. It was found that pitting corrosion on the sheets responsible for the leakage/failure. Such pitting corrosion occurred due to the presence of high level of chlorides in the water. The pitting corrosion are sever due to the presence of martensitic phase which were generated at the manufacturing stage due to severe plastic deformation and acts as galvanic effect for the presence of two phases such as austenite and martensite. The presence of martensitic phase was conformed through the attraction of such heat exchanger sheets to a permanent magnet and further conformed by the formation of hysteresis loop through a magnetic NDE device by the application of cyclic magnetic field.

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