Failure analysis of AISI 304 stainless steel pipeline transmission a petrochemical plant

A Arifin1, Gunawan1,*, I Yani1
1 Department of Mechanical Engineering, Faculty of Engineering, Universitas Sriwijaya, Indralaya, 30662, Sumatera Selatan, Indonesia
* Email: gunawan@unsri.ac.id

Abstract. This paper presents the failure analysis of AISI304 stainless steel pipeline that was fabricated by welding and used for CO\textsubscript{2} transmission in a petrochemical plant. After a years of satisfactory operation, significant cracking was observed adjacent to the weld joints of valve and pipeline. The failure investigation was carried out on welded pipe samples using optical and fractography, micro hardness measurements, XRF and XRD testing. The hardness test results show faults occur in the HAZ region. Fractured surface macrostructure and morphology analysis shows inter-granular brittle fracture mode. The inter-granular fault begins with the presence of very intensive inter-granular corrosion, especially on the outer metal surface and progressively propagates to the inside of the metal. The results of XRD found peak of carbide from the secondary phase of SS 304 austenite steel. The material experienced sensitization that is indicated by the presence of carbides at the grain boundary.

Keywords: AISI-304; fractography; inter-granular cracks; Sensitization

1. Introduction
The selection of stainless steels for chemical transmission is based on mechanical properties, corrosion resistance, fabrication characteristics (such as weld ability), working environment, service temperature, and cost. However, corrosion resistance and mechanical properties are generally the most important factors whenever selecting a grade of stainless steel for a given application [1]. Among the many 300 series austenitic stainless steel grades, AISI-304 SS is widely used for the construction of large storage tanks for styrene monomers, because of its corrosion resistance in many environments and it’s relatively low cost as compared to other 300 series steels [2].

The austenite phase in Austenitic Stainless Steel is stable even with very low nickel content, but with an increase in the percentage of manganese. However, this causes a decrease in the chromium solubility limit in the austenite phase. Therefore, the chromium content must be reduced to 15-16% by weight in Cr-Mn Austenitic Stainless Steel to keep the austenite structure intact. Cr content with a range of 18-20 Cr%, found on AISI 304 SS.

Ferritic and austenitic stainless steels are susceptible to intergranular corrosion when heated to temperatures between 400°C and 900°C [3]. The sensitization phenomenon occurs due to the combination of chromium with carbon which forms chromium carbide at the grain boundary. Increased carbide causes chromium grain boundaries that are susceptible to intergranular corrosion. This causes the appearance of microscopic cracks that will propagate at the grain boundary which causes the component to break even if it works below the yield point [4].
Valve (valve) is a component that has a very important role in ensuring a factory can operate. Failure to operate on the Valve will cause the factory to shut down. A valve is a device that regulates, directs or controls the flow of a fluid (gas, liquid, fluidized solids) by opening, closing, or closing part of the flow path. The valve can be operated manually, either by the handle, the lever pedal and others. Besides being able to be operated manually, the valve can also be operated automatically by using the principle of changing the flow of pressure, temperature, etc.

The problem of environmentally induced SCC and sensitization of stainless steel is of prime concerned to chemical, oil, & gas industries and refineries, and should be avoided, if practical, for any application. In general, poor welding practices promote the susceptibility of austenitic stainless steels to SCC. The main objective of this work is to analysis failure of AISI 304 stainless steel pipeline transmission.

2. Materials and Method
The investigation of the pipeline was made to determine the potential causes of the failure. A series experiment procedure performed to examine material properties after breaking. A fractographic based study of the fracture surface used to identify surface features by performing macro and micro structure analysis via both visually or microscopically that indicate the type and origin of failure. The present investigation also included hardness profile on the surface sample by using Vickers hardness tester VKH-2E, while composition analysed using XRF Analyser Niton XL2 and phase of material detected using X-Ray Diffraction Rigaku MiniFlex 600.

3. Results and Discussion
Composition testing was carried out to determine the type of material used using X-ray fluorescent (XRF) analyzers. The test is carried out in two different positions from the pipe, which is in the broken area and un-broken ends. The XRF results as can be seen in Table 1 show that the sample has a composition which is close to the SS 304 Austenitic element and there is no significant difference in chemical composition between the two test points. In the later position a small amount of molybdenum (Mo) is detected. However, the concentration of Mo elements is very low to influence the chemical properties of austenite steel, so the presence of this element can be ignored. In general, this element is added in an amount of at least 2% in austenite steel.

Table 1. XRF result of SS 304 Austenitic elemental analysis

| Element | Broken area | Un-broken area |
|---------|-------------|----------------|
| Cr      | 18.19 ± 0.62 | 18.47 ± 0.56  |
| Mn      | 0.928 ± 0.459 | 1.07 ± 0.41   |
| Fe      | 70.96 ± 1.16  | 70.97 ± 1.06  |
| Ni      | 9.79 ± 0.75   | 9.31 ± 0.67   |
| Mo      | 0.178 ± 0.069 | 0.069 ± 0.069 |

The cross section of an failure pipe is shown in Figure 1.a Based on the visual analysis of macrostructure morphology on the fracture surface, the fracture mode shows brittle fracture. Brittle fractures on the morphology are clearly seen in quadrants I, II and IV. The fractured morphology also shows brown color that is believed to be due to corrosion (Figure 1.b). The presence of this corrosion also proves that the cracks exposed to the air for long time. Meanwhile in Figure 1.b also shows fractured surface due to fatigue as can be seen from the morphology marked by the presence of a beach mark in quadrant II.

The results of the hardness testing of fracture surface samples were carried out with the Vickers Method. The SS 304 material hardness is generally 189 VHN. As can be seen from figure 2 the Vickers value increases in longitudinal to the arrow direction compared to the hardness of material that is not
exposed to welding heat. This increase occurs due to phase changes due to high exposure to heat and at the same time causes the brittle value of the material to increase.

![Fracture Morphology](image1.png)

**Figure 1. Fracture Morphology**

![Vickers hardness test result](image2.png)

**Figure 2. Vickers hardness test result**

XRD testing is used for analysis of phase composition, compounds or material types as well as crystal characterization. The XRD result is depicted in figure 3 where material shows phase characteristics commonly found in austenitic steel namely phases of $\alpha$, $\gamma$ and $\text{Fe}_2\text{O}_3$. However, the results of XRD also found peak of carbide from the second phase (secondary phase) of SS 304 austenite steel. This phase usually occurs because the material has been exposed to high temperatures in a certain time range [5,6].

Observation of microstructure aims to analyze the microstructure present in the material can be seen at figure 4. Austenitic stainless steels do not respond to magnetic treatment without heat and are influenced by cold working. The steel microstructure consists of austenite grains which can show twinning. Twinning microstructure is detected in a number of places which are characteristic of austenite
Phase, this is in accordance with structures owned by SS 304 which have FCC structure. In this observation we also found the distribution of carbides in austenite and ferrite granules.

![Figure 3. XRD test result](image)

In addition, precipitation (carbide formation) is also found in grain boundaries. This precipitation occurs usually due to high temperatures that reach the range sensitization of SS 304 (600 - 850 °C). If this happens the carbon will diffuse towards the grain boundary to form Cr Carbide. Cr carbide has brittle properties that are very detrimental to the mechanical properties of the material.

![Figure 4. Microstructure observation](image)

Stainless Steel SS 304 material Austenitic having a content of more than 0.05% C which is susceptible to inter-granular (weld decay) corrosion in the HAZ region. In addition, because of the high percentage of Cr in broken part, it is very vulnerable to the occurrence of the process of Cr carbide precipitation at the grain boundary, commonly known as sensitization. From the results of XRD testing it is known that the carbide phase Cr and carbide spread into grain and grain boundaries as seen in previous microstructure observations. In the temperature sensitization range carbon atoms will diffuse very quickly towards the boundary of the grain where they will join the Cr atom which forms Cr carbide.
SS 304 has a range of temperature sensitization between 600°C to 850°C. Visual observations obtained information that valve failure occurred on the HAZ. This confirms that failure can be ascertained due to precipitation of Cr carbide (sensitization). Phenomenon sensitization does not appear in the fusion zone because this position is the highest temperature peak. Sensitization will appear in the area closest to the fusion zone which has a lower peak temperature. This can be explained based on the thermal cycle of welding in figure 5.

Zone no. 2 is a zone where sensitization arises because it has a longer time range that allows precipitation. While zone 1 has a very short time range for precipitation and zone 2 peak temperatures are too low to reach zone of sensitization. Testing of hardness also proves that there is a significant increase in hardness towards the fault (weld) which proves that there is also a phase change in the material. Cr carbide itself has brittle properties.

After experiencing sensitization, there will be Cr buildup at the grain boundary which causes a lack of Cr atoms in forming a layer of Cr oxide. This results in inter-granular corrosion which causes microscopic cracks to appear. The propagation of this crack is compounded by the vibrations received by the structure. Corrosion at this grain boundary starts from the metal surface then propagates towards the direction in the metal as shown in the figure 6.
4. Conclusion
XRF results indicate the material used is SS 304. The hardness test results show fracture occurs in the HAZ. Damaged surface macrostructure analysis and fault morphology shows inter-granular brittle fracture mode. The inter-granular fault begins with the presence of very intensive inter-granular corrosion, especially on the outer metal surface and progressively propagates to the inside of the metal SS 304 material experienced a sensitization indicated by the presence of carbides at the grain boundary.

Acknowledgment
The authors would like to convey their great appreciation to Universitas Sriwijaya for supporting this research.

References
[1] -----2004, Handbook of Comparative World Steel Standards ASTM DS67B Third Edition John E. Bringas, Editor, ASTM International, West Conshohocken, PA.
[2] Kou S 2003 Welding Metallurgy (New York:John Wiley & Sons, Inc)
[3] Queiroz A A D 2012 AISI 304 Steel Intergranular Corrosion (Failure Analysis). In: 21th SAE BRASIL International Congress and Exhibition October, 2nd to 4, São Paulo, Brasil
[4] Callister W D and Rethwisch D G 2013 Materials Science and Engineering: An Introduction, 9th Edition: Ninth Edition (New York: John Wiley and Sons, Incorporated)
[5] https://www.phase-trans.msm.cam.ac.uk accessed at 22 September 2018
[6] Karimi, N., et al. 2008. "Characterization of the oxides formed at 1000°C on the AISI 304 stainless steel by X-ray diffraction and infrared spectroscopy." Applied Surface Science 254 2292-2299.