

A Review Study of Critical Oxygen Content to Stress Corrosion Cracking of Stainless Steel 304

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Abstract: Stress corrosion cracking is so destructive that it can cause severe damage to a component to the point of beyond repair thus the purpose of this research is to investigate the critical oxygen content to stress corrosion cracking to stainless steel 304. The objectives of this study are to determine the corrosion rate of the experimental immersion, to study the effect of temperature to the length of immersion period, to investigate the effect of oxygen in accelerating the corrosion rate and to compare the result if the corrosion rate of experimental immersion and the reviewed electrochemical test. The investigation is done by conducting an immersion test and reviewing previous research. Based on the immersion experiment, the highest corrosion rate was recorded on the 45° U-bend specimen. From the previous immersion research, it was found that the temperature plays an important role in accelerating the corrosion rate. A review of Tafel analysis was conducted for the electrochemical test and the outcome was the longer the specimen immersed in the electrolyte, the higher the corrosion rate. This is due to the presence of the oxygen that accelerate the rate of corrosion. A comparison of corrosion rate was made between the experimental immersion and the reviewed tafel analysis. The result shows similar trend which is the longer the period of immersion, the higher the corrosion rate.

Keywords: Corrosion rate, Immersion, Oxygen, Stainless steel 304, Tafel analysis

1. Introduction

Corrosion is the degradation of material mostly a metal due to the simultaneous exposure to the surroundings [1]. The types of corrosion of a metal is depends on how a part is used and was exposed to what kind of state [2]. Generally, there are two types of corrosion, which are uniform corrosion and localized corrosion. Uniform corrosion usually will attack metal such as carbon steel because it contains iron which when exposed to the environment it will oxidizes and ended up creating rust [3]. Localized corrosion is the attack of corrosion of a passive metal at a much higher rate in a corrosive environment such as liquid [4]. Localized corrosion has several types mainly pitting, crevice, and stress corrosion cracking. In various ways where if the austenitic stainless steel is used incorrectly, stress corrosion

cracking may happen which makes this topic is the most attractive for the researcher to do the investigation [5].

Stress corrosion cracking (SCC) failure happens when the metal has a material vulnerability which involving the tensile stress and an aggressive environment [6]. SCC is cause by the combination from the influence and synergistic interaction of mechanical stress and a corrosive environment [7]. The vulnerability to SCC is usually depends on the chloride content, the temperature and the pH value of the corrosive environment [8].

As in the case of reactors, the effect of SCC can create a huge crack in the structures where the residual stress from the welding is dominating and by comparison, the operational stresses are low. If leaves undetected, the cracks can lead to the failure resulting catastrophe [8].

2. Materials and Methods

The material that being used to perform this study is stainless steel 304 plate. For the immersion test, the plate will be bend into three shape which are U-bend, 45° U-bend and a full circled bend according to the ASTM G30-97. The plates was bended by using a bending machine and flywheel machine. The solution used was 3.5wt% sodium chloride. The immersion period was 14 days and the weight loss was taken in every 7 days. As for the electrochemical test, previous research papers was reviewed. Tafel analysis was conducted and throught data collected, the corrosion rate was obtained.

2.1 Immersion Test

The immersion standard that was used in this research is ASTM G1 where it provides an easy method to determine the corrosion rate in an aqueous condition. The requirement for the immersion testing to be done is from 24 hours and can up to several months. For this research, the immersion test took 14 days to be completed. The samples were immersed in seawater that contain 3.5%wt sodium chloride (NaCl). The calculation of the corrosion penetration rate can be calculated by using the equation 1. The length and the width of the specimens are 17 cm and 3 cm respectively and the area of the specimen is 51 cm².

$$CPR = \frac{W \times k}{\rho \times A \times t} \quad Eq. 1$$

2.2 A Review of Electrochemical Test (Tafel analysis)

The electrochemical test that reviewed in the previous research paper was conducted by using a potentiostat to get the Tafel analysis. This is different from the immersion test where it only needed a test cell to conduct [9]. The potentiostat consists of three electrodes to record the polarization curve which are reference electrode (Ag/AgCl), counter electrode (graphite) and working electrode (sample) [10]. Before the experiment started, the sample needed to be reclean by polishing it with sandpaper to remove any dust or rust that is caused by the environment exposure. The electrochemical test consists of 5 specimens which was undergo the Tafel analysis at different time of immersion. The corrosion rate was then obtained throught the data extracted from the corrosion rate analysis.

3. Results and Discussion

The results and discussion part are divided into two parts of which the first part is the results obtained through the experimental immersion test while the second part will be the discussion on the results obtained from the review of the previous research papers.

3.1 Results of Immersion Test

Based on the result on Table 1, the highest percentage of weight loss occurred at the U-bend stainless steel type. The cause of the weight percentage loss is greater might be influenced by the type of bending where the microstructure of the stainless steel has changed. The highest weight loss occurred at day 14 of the fully circle bend type, which is 0.097%.

The corrosion rate occurred at day 14 of the specimens are more likely higher than at day 7. The highest corrosion rate happened at the 45° U-bend type which is 0.00632 mmpy and followed by fully circle bend (0.0625 mmpy) and U-bend (0.0593 mmpy). The result of the corrosion rate shows that the U-bend has the highest value at the first week, which is 0.0503 mmpy. However, on the second week there was some changes where the corrosion rate of the 45° U-bend and the fully circle bend is higher than the U-bend.

Table 1: Result of the immersion test of stainless steel 304

Type of bend	Period (day)	Mass before corrosion attack (g)	Mass after corrosion attack (g)	Weight loss (g)	Percentage weight loss (%)	Corrosion rate (mm/yr)
U- bend	7	217.517	217.439	0.078	0.036	0.0503
	14	217.439	217.347	0.092	0.042	0.0593
45° U-bend	7	221.170	221.104	0.066	0.030	0.0425
	14	221.104	221.006	0.098	0.044	0.0632
Fully circle	7	223.416	223.269	0.050	0.022	0.0322
	14	223.269	223.366	0.097	0.097	0.0625

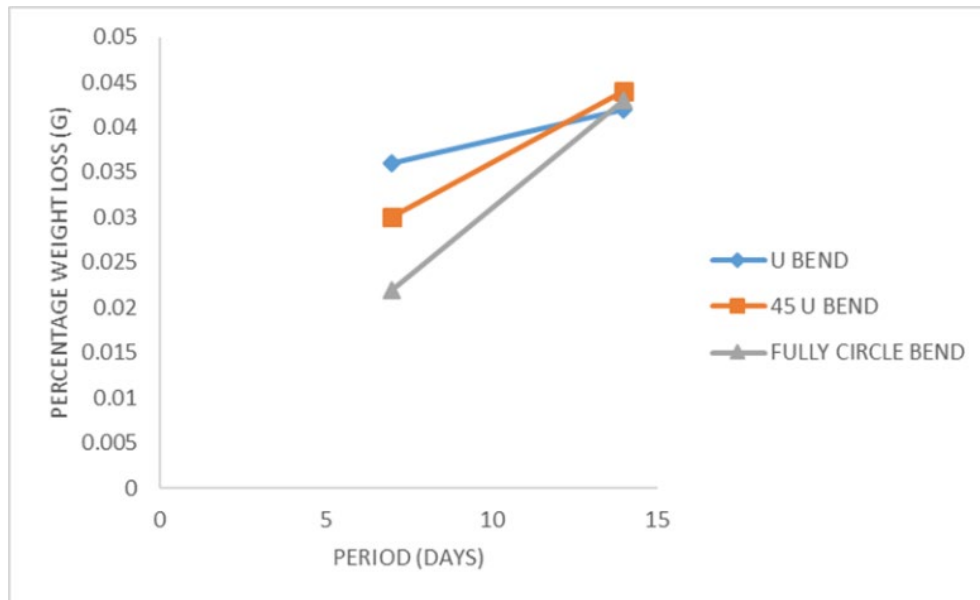


Figure 1: Relationship of percentage weight loss of sensitized stainless steel 304 immersed in NaCl solution against period

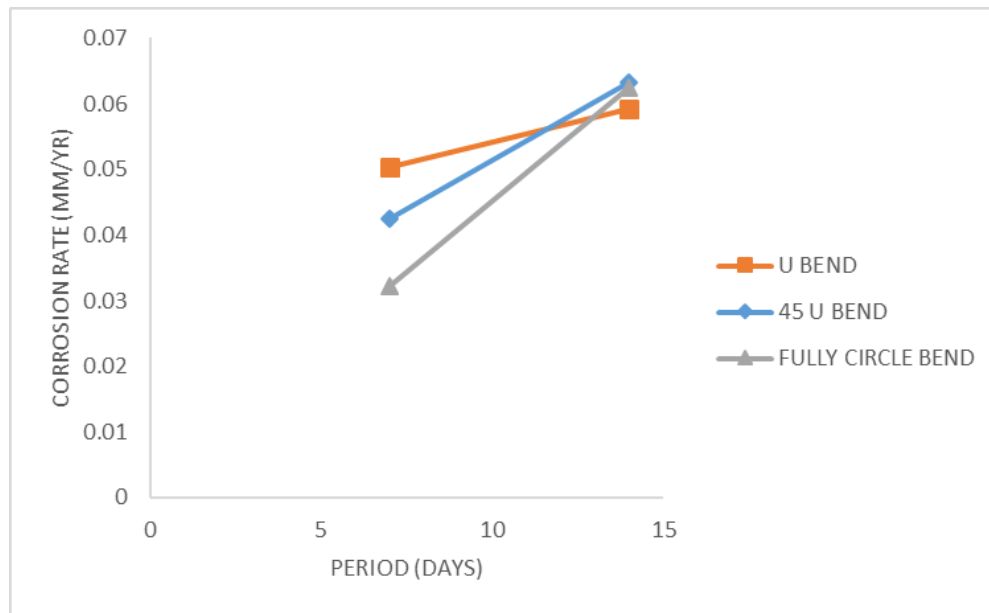


Figure 2: Relationship of corrosion rate of sensitized stainless steel 304 immersed in NaCl solution against period

Based on Figure 1 and Figure 2, it shows that the percentage weight loss and the corrosion rate have similar trends. Both percentage weight loss and corrosion rate increase as the period of immersion increases. The possibility of the increased elements is due to the sensitized materials. As per mention that stainless steel is the ideal material to withstand the corrosion, but when sensitized, the microstructure of the material itself is not stable thus has weaker resistance to corrosion attack through time.

The percentage of weight loss and the corrosion rate of 45° U-bend and the full circle bend have significant increases from day 7 until day 14 while the U-bend weight loss and corrosion rate only have slightly increase throughout the immersion period.

3.2 Comparison of Previous Experiment

A research by Zakaria et al about stress corrosion cracking induced by chloride where this type of corrosion always happened in the 300 series of austenitic stainless steel. The material that used in the experiment are the stainless steel grade 304L that was U-bend according to the ASTM G30 standard.

Table 2: Result after 42 days of immersion [11]

Temperature	28°C	70°C	90°C
Observation	No crack after 42 days	<ul style="list-style-type: none"> • Pitting in 21 days • Crack in 28 days • General corrosion 	<ul style="list-style-type: none"> • Pitting in 7 days • Crack in 14 days • Badly corroded

After 42 days of immersion, the result of the experiment is as shown in Table 2. It shows that when the specimen was immersed in 28°C, there is no sign of crack occurred. While for the high temperature solutions when the specimens was observed under a microscope, it was having pitting as well as cracked. This happen due to the temperature factor since the chemical reaction increases when the temperature rises [12].

For specimen that was immersed at 70°C temperature condition, pitting began to be visible on day 21 and followed by cracked on day 28. The specimen was experienced general corrosion after the experiment was finished. The same thing happened to the specimen that was immerse at 90°C temperature but the pitting and cracked occurred much faster where pitting began on day 7 and cracked on day 14. The diffusion of oxygen was accelerated as the temperature increase eventually degraded the protective property of the film in the steel [13].

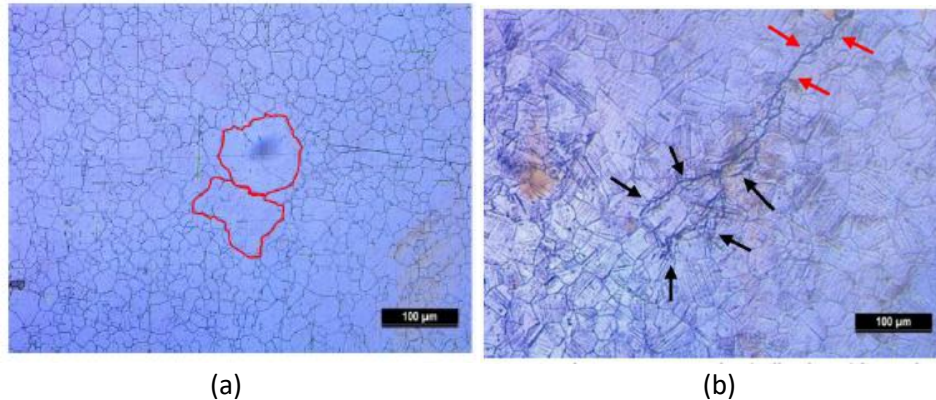


Figure 3: Crack on the specimen at (a) ambient temperature and (b) 90°C [11]

Figure 3 (a) shows the micrograph of the grain boundaries that was observe under 200× magnification by using an optical microscope. The image displayed that the specimen experienced a few grain expand located in the red circled marked in the image. The microstructure of the specimen was change due to the heat treatment and the cooling process. Small grains of atoms moved to large during the cooling process because of a stable and stronger bond. Over time, the small grains will started to diminish.

Meanwhile Figure 3 (b) shows the result of the micrograph of the specimen that was immerse in the NaCl solution at 90°C condition. The specimen shows both of the transgranular and intergranular crack. It was pinpoint that the initiation of the crack was the intergranular crack and the intergranular branching type. By sensitizing the material, it could reduce the crack resistance due to the chromium carbide precipitate along the grain boundaries. Due to the failure of the protective oxide film in the presence of corrosive chloride ions and sensitized microstructure, the crack most likely originated from pitting.

In conclusion, after 42 days of immersion, there was no sign of crack on the specimen that was immerse in the ambient temperature but at 70°C and 90°C temperature condition, pitting and crack began at day 7 until day 28. It indicates that the higher the temperature of the NaCl solution, the faster of the crack initiation to begin.

3.3 A Review of the Electrochemical Test (Tafel Analysis)

Another research publish by Mahesh et al. (2018) manage to define an electrochemical experiment that determined the corrosion rate and stress corrosion cracking of austenitic stainless steel under NaCl solution in electrochemical test [14]. From Table 3, the result of the corrosion rate was obtained through the calculation. It shows the result of different i_{corr} value, corrosion rate in mmpy and the time of immersion each of the specimens.

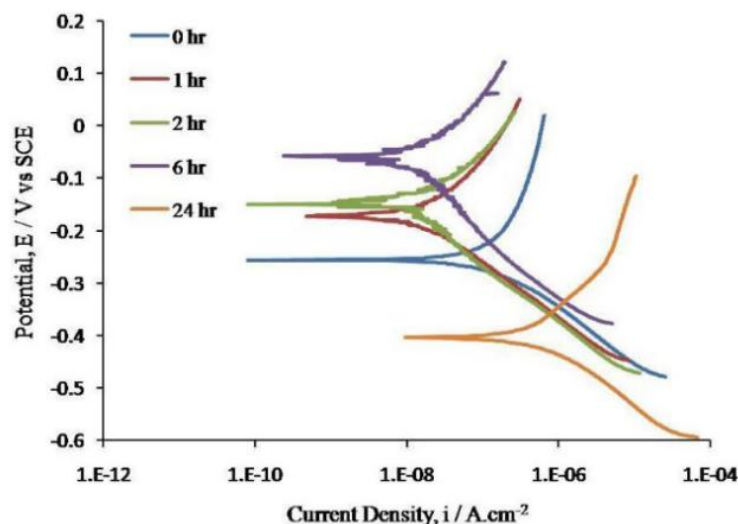


Figure 4: The Tafel graph of voltage against current of the specimens in 35 g/l NaCl solution [14]

Table 3 Result of electrochemistry and corrosion rate [14]

Sample	Immersion time, hr	β_c, V	β_a, V	$i_{corr}, \mu A/cm^2$	Corrosion rate, mm/py
1	0	9.855	3.794	0.11310	0.0017135
2	1	9.658	6.705	0.01499	0.0002171
3	2	7.736	7.605	0.01676	0.0002427
4	6	5.935	6.579	0.02048	0.0002964
5	24	3.209	6.909	0.02066	0.0002989

The corrosion rate was determined by using Tafel extrapolation method with Faraday’s law equation. The result of the electrochemistry test is then derives from IV Man software. From Figure 4, after getting the polarization resistance obtained from the IV Man software, the value of corrosion current is calculated by using Stern and Geary equation.

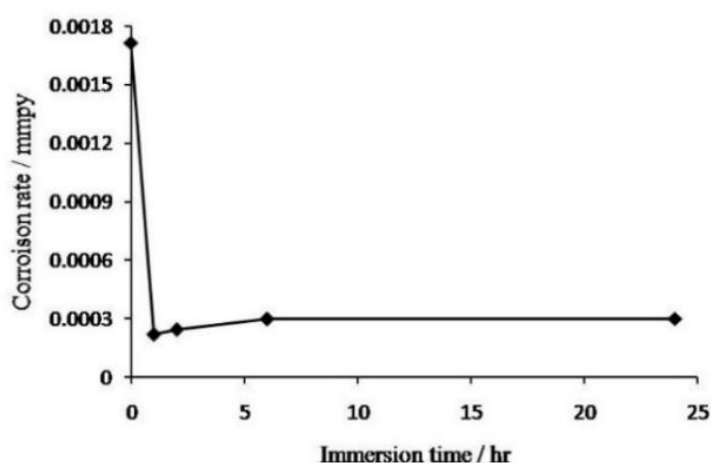


Figure 5: Relationship of corrosion rate of sensitized stainless steel 304 immersed in NaCl solution against period [14]

The result of the corrosion rate plotted in the graph in Figure 5 shows that the corrosion rate from 0 hour to 1 hour is decreasing from 0.0017135 mmpy to 0.0002171 mmpy rapidly prior of the

presence of strong passive protective layer formation on the specimen when in contact with the electrolyte (NaCl) over time. When the immersion period was increase up to 2 hours and 6 hours, the specimens was having a slightly increased in the corrosion rate value which are 0.0002427 mmpy and 0.0002964 mmpy simultaneously. For the immersion for 24 hours, after 6 hours of immersion, it shows that the corrosion rate is nearly constant at 0.0002989 mmpy but compared to at 0 hour corrosion rate, it is lesser. This can be conclude that the highest corrosion rate occurred at between 0 hour to 1 hour time of immersion and the lowest corrosion rate was in the immersion of 24 hours.

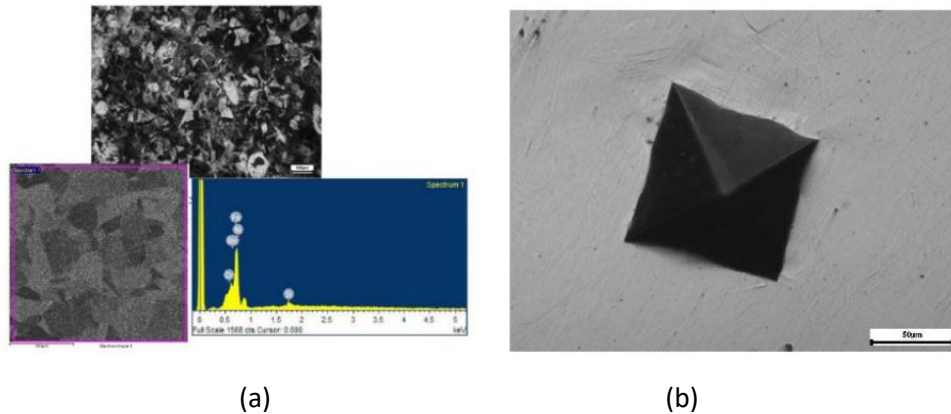


Figure 6: (a) EDX of the specimen and (b) Optical microscope of the indentation of the specimen [14]

Figure 6 (a) shows that Fe was the highest peak in the spectrum graph. Figure 6 (b) presents the image of the optical microscope (OM) that shows the micro visual of the Vicker hardness test of the specimen that have 189.5 ± 5 Hv measurement. From the image, it shows obvious indentation cause by the load when performing the hardness measurement and the specimen was deformed with a striations parallel to the shape of the indentation rhombus.

In conclusion, to understand the various characteristic of corrosion of the metal alloy in an electrolyte, the experiment was conduct by varying the time of immersion for each of the specimen. In order to foreseen the likelihood of the dissolution, the scale of potential from cathode to anode is near to the corrosion potential value.

3.4 Comparison of experimental immersion and reviewed Tafel result

From the data obtained in the experimental immersion and the reviewed research Tafel experiment, the result of both corrosion rate can be compare. Both of the electrolyte used are the same which is 3.5wt% of NaCl and it is using the same material which is sensitized stainless steel type 304. Although the length of immersion time is not the same, but the graph shown in Figure 2 and Figure 5 show the same trend.

From the review of the Tafel experiment, it shows that a drastic decreases of the corrosion rate at the 0 hour, but as the longer the length of the immersion time, the corrosion rate continued to increase. The same trend obtained from the experimental immersions test, where the corrosion rate is having constant increase from day 1 until day 14.

Through the data collected, it can be seen that the corrosion rate of the immersion test is higher than the electrochemical test. This is probable due to the differences in terms of dimension and the length of the immersed period.

3.5 Effect of oxygen to stress corrosion cracking

The cracking of the stainless steel in chloride solution was accelerate by the presence of oxygen at high temperature condition [15]. However, according to Neuman and Griess, the cracking susceptibility for the Type 347 stainless steel U-bend specimen was unchanged when the content of the oxygen ranging

from 1 ppm to 1200 ppm in a chloride solution that having 100ppm Cl^- at 300°C with pH value at 2.8 or 10.5. Crack can happen even in small amount of oxygen concentration that is present in the solution. An investigation showed that they have nearly constant number and pattern of crack in the specimen at all oxygen levels. William stated that only when the chloride is coexist with oxygen or other easily reducible species, the crack could happened. This statement was proven with the fact that there is no evidence of crack when the oxygen was completely removed from the system [16].

3.6 Effect of temperature to stress corrosion cracking

The effect of temperature is one of the main cause that contribute to time of crack of material. It has significant influence to a given system on the time to failure. This is not unpredictable since it is an electrochemical reaction and the rate of reaction is increase when the temperature is also increase [17]. A research conducted by Thomas et al showed a trend of crack decrease in time with a slightly increases of temperature in $\text{MgCl}_2(31\%\text{Cl}^-)$ solution. When the chloride concentration was decrease, the effect on temperature was decrease and increased the time of failure.

4. Conclusion and recommendations

The present work was aim to investigate the critical oxygen content to stress corrosion cracking to stainless steel 304. The study was completed by conducting an experiment and by reviewing previous research papers. The significant conclusion that can be point out are:

- i. From the experimental immersion for 2 weeks, it can be identified that the 45° U-bend have the highest corrosion rate and followed by fully circle bend and U-bend.
- ii. A review from the previous research stated that oxygen is causing the acceleration of the stress corrosion cracking even if there is only a small amount of the oxygen present in the solution.
- iii. A review from the previous research stated that when the temperature is increase, the time of the metal to crack or fail is also increase. This happens because of high temperature condition accelerate the electrochemical reaction and the rate of reaction.
- iv. By comparing both of the experimental immersion test and the review electrochemical test, the data shows that both of the corrosion rate is having the same trend which is the corrosion rate increase as the length of immersion is increase.
- v. The correlation between the temperature and the oxygen level is that when the temperature increase, it accelerates the diffusion of the oxygen and as well as as the formation of the corrosion product thus increases the corrosion rate.

Nowadays, the used of the austenitic stainless steels have a higher demand as most of the petrochemical industry and construction are using it at large. Thus, to have a better understanding to the cause and behaviour of the austenitic stainless steel to stress corrosion cracking, suitable corrosion test should be conducted. A further investigation such as electrochemical test should be performed in order to get quicker result on how the material will behave and analyse its microstructures to identify how the crack propagates.

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