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304 Stainless Steel Corrosion Rate in HNO_3 Solution

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ABSTRACT

This research purpose to find out the corrosion rate of Stainless Steel 304 in sulfuric acid. Immerse process do in 700 C temperature for 6 hours and than in 290C temperature for 18 hours every day. This condition aim to get closer to the actual use in the manufacture of tubes of reactor SAMOP (Sub Critical Assembly for Mo99 Prad Action). Specimen atfer TIG welding and electric arc welding cleaned from crust and than measured, drawing balanced, and record the first weight. And than specimen innerse to sulfuric acid pH 0.2 and 0.5 in 700C temperature for 6 hours and than in 290C temperature for 18 hours every day for 3 month. Weight recording has been done every week. The survey findings show heavy shrinkage sample, it is well visible on the corrosion pH 0.5 in electric arc welding of 0.105 gram/dm2/month for corrosion 0.026 gram/dm2/month without welding and pH 0.2, arc welding of 0.045 gram/dm2/ month, pH 0.2 no Las 0.02 gram/dm2/month. Specimens that have been welded have significant differences in corrosion rate compare to specimens that have not welded.

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1. INTRODUCTION

The development of time and technology affects the diversity of human needs(Octavia, 2020). The application of technology in industrialized countries is developing rapidly(Coal, 2020). Almost all industrial equipment and machinery and their components are designed in such a way(Wijayanto et al., 2015)so that the maximum strength and service life can be known. This requires research time and high accuracy(Huda, 2018). A series of chemical processes are required to obtain steel with the desired mechanical properties(Sukarno, nd), for example: ductility, toughness, hardness, corrosion resistance and others.

In this final project the author conducted research on the effect of HNO3 solution with a pH of 0.2 and 0.5 on the corrosion rate of 304 . Stainless Steel(Siahaan, 2021). This type of stainless steel is used for the SAMOP reactor tube (Sub Critical Assembly for Mo99 Prad Action)(STEEL, nd). Furthermore, the parameters of temperature, time and acidity are all made to resemble the actual operating conditions of the SAMOP reactor(Goddess, 2007). The purpose of this study was to determine the corrosion rate of Stainless Steel 304 which has been welded in an HNO3 environment with a pH of 0.2 and 0.5 .(Sumarji, 2011).

2. RESEARCH METHOD

2.1 Research Flowchart

The research flow chart is shown in Figure 1:

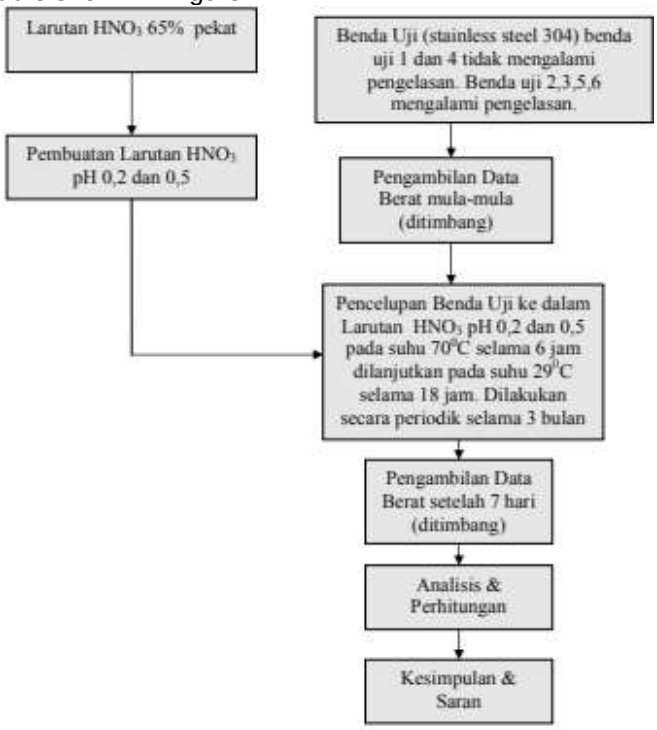

Figure 1. Research Flowchart

2.2 Materials And Equipment

2.2.1 Ingredient

1. Specimen / Test Item

The material used as the specimen is stainless steel 304, with the composition: Cr = 18.358%, Ni = 8.408%, C = 0.047%, Fe = 70.47%(Setiawan, 2012).

2. 65% HNO3 solution.

This 65% HNO3 solution was purchased from the pharmacy department laboratory, Sanata Dharma University Yogyakarta(Sinaga & Rina, 2018).

2.2.2 Equipment

The equipment used in this study are: (1) Test Tubes, belonging to the Analysis Laboratory, Department of Pharmacy, Sanata Dharma University, Yogyakarta(Utomo, nd). (2) Digital Electric pH meter, owned by the Analysis Laboratory, Department of Pharmacy, Sanata Dharma University, Yogyakarta(Rust & Has, nd). (3) Digital Electric Scales, owned by the Laboratory of Analysis and Instruments, Department of Pharmacy, Sanata Dharma University, Yogyakarta, with an accuracy of up to grams$\frac{1}{1000}$(Siahaan, 2021). (4) Water Bath and Thermometer, belonging to the Analysis Laboratory, Department of Pharmacy, Sanata Dharma University, Yogyakarta(Julianus & Luckyvano, 2014).

2.2.3 The Process of Making HNO3 Solutions pH 0.2 and 0.5 And Immersion Process

The process of making a solution of HNO3 pH 0.2 and 0.5 from a concentrated HNO3 solution of 65% was mixed with distilled water(Tarakanita, 2011). The steps in the process: (1) Equipment and materials prepared: a. test tube, b. 65% HNO3 solution, c. Pipette, d. Digital Electric pH Meter, e. Aquades. (2) Under normal conditions, the level of 1 normality (N) of HNO3 = 63 g/ltr of pure HNO3. So to obtain a solution of HNO3 with a pH of 0.2 from a 65% HNO3 solution with a specific gravity of 1.39, 34.23 ml was taken. This is obtained from:
pH 0.2 = 10-0, 2 N.
= 0.631 N

$$\text{Maka, } 0{,}631 \times \frac{100}{65} \times \frac{1}{1{,}39} \times 63 = 43{,}9 \text{ ml/ltr}$$

To obtain a solution of HNO3 pH 0.5 from a 65% HNO3 solution with a specific gravity of 1.39, 22.05 ml was taken. It is obtained from(Amalulia, 2016):
pH 0.5 = 10 0.5 N
= 0.3162 N

$$\text{Maka, } 0{,}3162 \times \frac{100}{65} \times \frac{1}{1{,}39} \times 63 = 22{,}05 \text{ ml/ltr}$$

then HNO3 65% is put into a test tube and aquades are added little by little until the volume is 1 liter. Then the pH was measured using a pH meter. (3) Specimens are put into each test tube which already contains HNO3 solution pH 0.2 and 0.5. (4) The test tube is put into a water bath whose temperature has been adjusted. (5) The dyeing process is carried out at a solution temperature in a tube of 700 C for 6 hours and a temperature of 29 0 C for 18 hours. This is to get closer to real use in practice. (6) Within 1 week the specimen is taken, dried and weighed. After that the specimen is immersed in the same solution with the same volume, namely 1 liter. Due to evaporation, it is necessary to add a solution every day to maintain pH and saturation(Siti, 2021).

2.2.4 Results Analysis

In general, this study aims to obtain the corrosion rate of Stainless Steel 304 which has undergone TIG welding & electric arc in HNO3 solution pH 0.2 and 0.5 at a temperature of 700C for 6 hours followed by a temperature of 290C for 18 hours.

3. RESULTS AND DISCUSSIONS

3.1. HNO3 solution pH 0.5

3.1.1 Stainless Steel Without TIG Welding (Test Object I)

The test object I is a stainless steel plate without experiencing. The test object I is a stainless steel plate without welding, immersed in a solution of HNO3 pH 0.5. Specimen Data I: (1) Thickness of test object = 3 mm, (2) Initial weight = 14.608 grams, (3) Length of test object = 34 mm (4) Width of test object = 18 mm. Calculation Analysis, Corrosion rate formula:

$$Laju\ korosi = \frac{\Delta y}{A.t} \quad (1)$$

With:
y = magnitude of change (m, mm, kg, gram)

t = Time (Hour, Month, Year)
A = Surface area (mm2, dm2)

Data on changes in weight of test object I in HNO3 solution pH 0.5
Corrosion rate data of test object II in HNO3 pH 0.5 solution.

Table 1. Corrosion rate of test object I in HNO3 solution pH 0.5

No	Week -	Initial Weight(grams)	Final Weight(grams)	Weight Loss (grams)	Large(mm2)	Corrosion Rate(grams/dm2/hour)
1	1	14.608	14.608	-	0.1536	0
2	2	14.608	14.607	0.001	0.1536	0.013
3	3	14.607	14.606	0.001	0.1536	0.019
4	4	14.606	14.606	-	0.1536	0
5	5	14.606	14,603	0.003	0.1536	0.097
6	6	14,603	14.601	0.002	0.1536	0.078
7	7	14.601	14.601	-	0.1536	0
8	8	14.601	14,598	0.003	0.1536	0.156
9	9	14,598	14,597	0.001	0.1536	0.058
10	10	14,597	14,597	-	0.1536	0
11	11	14,597	14.596	0.001	0.1536	0.0716
12	12	14.596	14.596	-	0.1536	0

3.1.2 TIG Welded Stainless Steel (Test Object II)

Data on changes in weight of Stainless Steel 304 which has been welded in a solution of HNO3 pH 0.5:

Table 2. Data on changes in weight of Stainless Steel 304 which has been welded in HNO3 solution pH 0.5

No	Sunday to-	Heavyobject test (grams)
1	0	13,795
2	I	13,795
3	II	13,793
4	III	13,789
5	IV	13,786
6	V	13,783
7	VI	13,780
8	VII	13,778
9	VIII	13,776
10	IX	13,772
11	X	13.771
12	XI	13,770
13	XII	13,770

From the data obtained the corrosion rate:

Table 3. Corrosion rate of SS 304 after welding in HNO3 pH 0.5

No	Week -	Initial Weight(grams)	Final Weight(grams)	Weight Loss (grams)	Large (dm2)	Corrosion Rate(grams/dm2/hour)
1	1	13,795	13,795	-	0.1464	0
2	2	13,795	13,793	0.002	0.1464	0.027

No	Week -	Initial Weight(grams)	Final Weight(grams)	Weight Loss (grams)	Large (dm2)	Corrosion Rate(grams/dm2/hour)
3	3	13,793	13,789	0.004	0.1464	0.081
4	4	13,789	13,786	0.003	0.1464	0.081
5	5	13,786	13,783	0.003	0.1464	0.102
6	6	13,783	13,780	0.003	0.1464	0.122
7	7	13,780	13,778	0.002	0.1464	0.095
8	8	13,778	13,776	0.002	0.1464	0.109
9	9	13,776	13,772	0.004	0.1464	0.245
10	10	13,772	13.771	0.001	0.1464	0.068
11	11	13.771	13,770	0.001	0.1464	0.075
12	12	13,770	13,770	-	0.1464	0

3.1.3 Stainlees Steel Subjected to Electric Arc Welding (Test Item III)

The test object III is a 304 stainless steel plate that has undergone electric arc welding. Data on changes in the weight of the test object III in the HNO3 solution pH 0.5:

Table 4. Data on changes in weight of test objects III in HNO3 solution pH 0.5

No	Week-	Weight of test object (grams)
1	0	16.081
2	I	16.081
3	II	16,074
4	III	16.058
5	IV	16.046
6	V	16,041
7	VI	16,041
8	VII	16,039
9	VIII	16.038
10	IX	16.035
11	X	16.033
12	XI	16,032
13	XII	16.031

Table 5. Corrosion rate of test object III in HNO3 solution pH 0.5

No	Week -	Initial Weight(grams)	Final Weight(grams)	Weight Loss (grams)	Large(mm2)	Corrosion Rate(grams/dm2/hour)
1	1	16.081	16.081	-	0.1542	-
2	2	16.081	16,074	0.007	0.1542	0.09
3	3	16,074	16.058	0.006	0.1542	0.116
4	4	16.058	16.046	0.001	0.1542	0.025
5	5	16.046	16,041	0.005	0.1542	0.162

No	Week -	Initial Weight(grams)	Final Weight(grams)	Weight Loss (grams)	Large(mm2)	Corrosion Rate(grams/dm2/hour)
6	6	16,041	16,041	-	0.1542	-
7	7	16,041	16,039	0.002	0.1542	0.09
8	8	16,039	16.038	0.001	0.1542	0.051
9	9	16.038	16.035	0.003	0.1542	0.175
10	10	16.035	16.033	0.002	0.1542	0.129
11	11	16.033	16,032	0.001	0.1542	0.071
12	12	16,032	16.031	0.001	0.1542	0.077

3.1.4 TIG Welded Stainless Steel (V Test Item)

Table 6. Corrosion rate of test object V in HNO3 solution pH 0.2.

No	Week -	Initial Weigh t (grams)	Heavy End (grams)	Weight Loss (grams)	Large(mm2)	Corrosion Rate(grams/dm 2/ja m)
1	1	14.364	14.364	-	0.1464	-
2	2	14.364	14,362	0.002	0.1464	0.027
3	3	14,362	14,358	0.004	0.1464	0.081
4	4	14,358	14,352	0.006	0.1464	0.163
5	5	14,352	14,359	0.003	0.1464	0.102
6	6	14,359	14.356	0.003	0.1464	0.122
7	7	14.356	14,355	0.001	0.1464	0.047
8	8	14,355	14,352	0.003	0.1464	0.163
9	9	14,352	14.349	0.003	0.1464	0.184
10	10	14.349	14.349	-	0.1464	-
11	11	14.349	14.349	-	0.1464	-
12	12	14.349	14.348	0.001	0.1464	0.081

3.1.5Stainless Steel Subjected to Electric Arc Welding (VI Test Item)

Table 7. Corrosion rate of test specimen VI in HNO3 solution pH 0.5

No	Week -	Initial Weigh t (grams)	Heavy End (grams)	Weight Loss (grams)	Large(mm2)	Corrosion Rate(grams/dm2/hour)
1	1	18,238	18,238	-	0.1542	-
2	2	18,238	18,236	0.002	0.1542	0.09
3	3	18,236	18,233	0.003	0.1542	0.058
4	4	18,233	18,231	0.002	0.1542	0.051
5	5	18,231	18,228	0.003	0.1542	0.097
6	6	18,228	18,225	0.003	0.1542	0.116

7	7	18,225	18,224	0.001	0.1542	0.045
8	8	18,224	18,223	0.001	0.1542	0.051
9	9	18,223	18,219	0.004	0.1542	0.223
10	10	18,219	18,218	0.001	0.1542	0.064
11	11	18,218	18,218	-	0.1542	-
12	12	18,218	18,217	0.001	0.1542	0.077

Table 8. Average corrosion rate for 3 months at pH 0.5

No	Test Object	Initial Weight(grams)	Final Weight(grams)	Large(mm²)	Average Corrosion Rate (grams/dm²/month)
1	I	14.608	14,590	0.1536	0.026
2	II	13,795	13,770	0.1464	0.056
3	III	13,478	13,469	0.1542	0.105

Table 9. Average corrosion rate for 3 months at pH 0.5

No	Test Object	Initial Weight(grams)	Final Weight(grams)	Large(mm²)	Average Corrosion Rate (grams/dm²/month)
1	IV	13,478	13,469	0.1442	0.02
2	V	14.364	14.348	0.1464	0.036
3	VI	13,795	13,770	O,1542	0.045

3.2. Corrosion Rate Chart

3.2.1 HNO3 solution pH 0.5

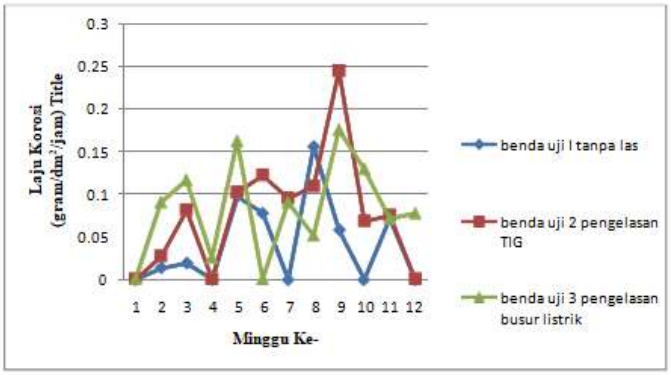

Figure 2. Graph of corrosion rate of stainless steel 304 in HNO3 solution with pH 0.5

3.2.2 No-load Test Results

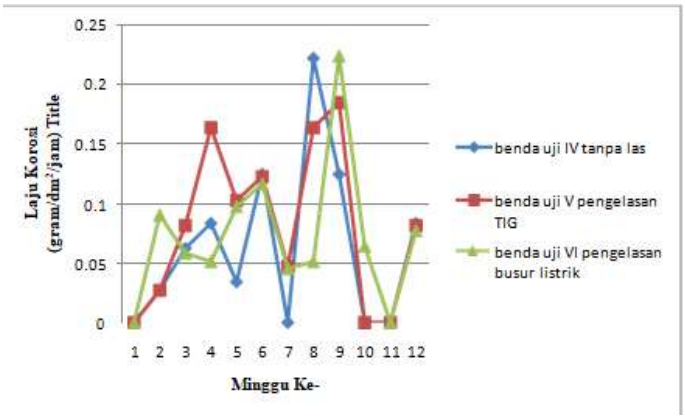

Figure 3. Graph of corrosion rate of stainless steel 304 in HNO3 solution with pH 0.2.

3.2.3 Corrosion rate per month at pH 0.5

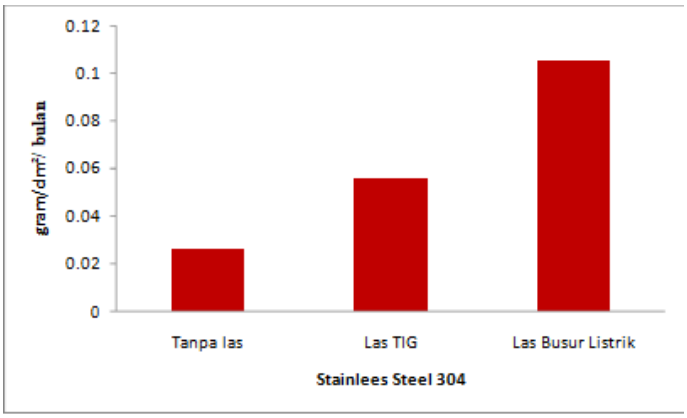

Figure 4. Graph of corrosion rate per month at pH 0.5

3.2.4 Corrosion rate per month at pH 0.2

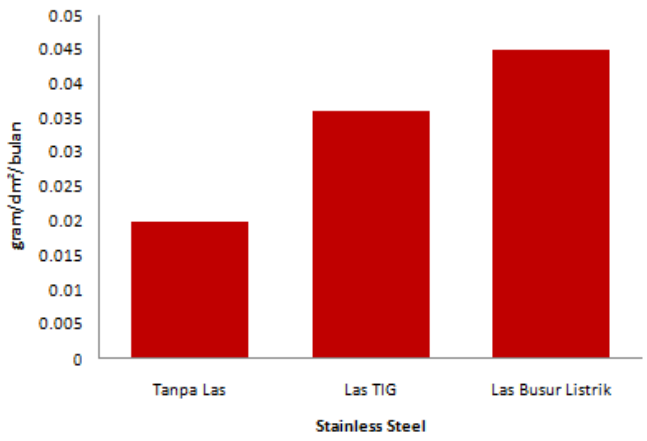

Figure 5. Graph of corrosion rate per month at pH 0.2

3.3. Discussion

Test objects that have undergone welding and those that have not undergone welding can corrode, this can be seen because of the reduction in weight of the test object after being immersed in a pH 0.5 HNO3 solution and also at a pH of 0.2. Corrosion that occurs evenly on the entire surface is due to damage to the protective layer that protects stainless steel from corrosion. There is a significant difference in the corrosion rate between the test specimens that have been welded and those that have not undergone welding. The highest corrosion rate of electric arc welding specimens is due to the poor welding method. In this welding, during cooling a lot of Cr is oxidized so that the protective layer becomes less than perfect. When the weight of the test object is reduced, the test object is corroded because part of the protective layer is damaged. When the test object does not experience a reduction in weight or the weight is stable, it means that the test object is not corroded. It is possible that the damaged protective layer was re-formed after an oxidation reaction occurred between the Cr in stainless steel and oxygen in the free environment when drying in the sun before measuring the weight once a week. From the visual appearance of the test object there was a change in color from before immersion to after immersion. it means the test object is not corroded. It is possible that the damaged protective layer was re-formed after an oxidation reaction occurred between the Cr in stainless steel and oxygen in the free environment when drying in the sun before measuring the weight once a week. From the visual appearance of the test object there was a change in color from before immersion to after immersion. it means the test object is not corroded. It is possible that the damaged protective layer was re-formed after an oxidation reaction occurred between the Cr in stainless steel and oxygen in the free environment when drying in the sun before measuring the weight once a week. From the visual appearance of the test object there was a change in color from before immersion to after immersion.

4. CONCLUSION

From the results of the research for 12 weeks, it can be concluded that there is a significant difference in the corrosion rate between the test objects that have undergone welding and those that have not undergone welding. All of them were equally corroded either by welding or not by welding, but the highest corrosion was found in electric arc welding. Corrosion that occurs evenly on the entire surface of the test object due to partial damage to the protective layer. The highest corrosion rate at pH 0.5 is 0.245 gram/dm2/week, while the highest corrosion rate at pH 0.2 is 0.223 gram/dm2/week.Suggestions in this study are: (1) If there is SS being welded, it is better to use TIG welding because its corrosion resistance is quite good against HNO3 (2) Use an accurate weighing tool for the test object and do not change the scales. (3) Dry the test object before weighing it, so that the moisture content is lost.

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