Electrochemical Behavior of Full Annealed Austenitic Stainless Steel

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Abstract. Austenitic Stainless Steel has been being used in many fields. Austenitic Stainless Steel which has composition 19 wt%Cr–7wt%Ni has been subjected to heat treatment. One of the many heat treatments applicable to improve the corrosion resistance is the full annealing method. This material subject to full annealing method with rapid quenching in various annealing temperatures; 1010°C, 1065°C, and 1120°C. Potentiodynamic curves before and after heat treatment were acquired to elaborate the electrochemical behavior of this austenitic stainless steel. Besides the corrosion rate, the structure of the austenitic stainless steel has been characterized using x-ray diffraction (XRD) to study the structure. The results showed the full annealing modified the structure and the electrochemical behavior of the austenitic stainless steel.

1. Introduction
Stainless steel is one type of ferrous-alloy that contains a minimum 10.5% Cr and can be divided into four major groups, as one of them is austenitic stainless steel. This type of stainless steel is the most produced stainless steel and also the most common type of stainless steel used in the world. Some good properties of the austenitic stainless steel are weldable, formable, high strength, ductility, and toughness, and also resistant to corrosion in many conditions. These excellent characteristics beneficially used in manufacturing industries, chemical plants, and other corrosive and severe environment [1]–[5].

The good corrosion resistance of austenitic stainless steel is due to the spontaneous appearance of the chromium oxide layer of its surface when the austenitic stainless steel exposed by the corrosive solution or environment [2]. Austenitic stainless steel is said to have the highest corrosion resistance caused by the higher amount of chromium and the addition of nickel, so the chromium is the foremost element of the austenitic stainless steel [6].

The type of austenitic stainless steel in this experiment is type 302 that is categorized in the normal unstabilized composition. By the American Society of Metals, one in three heat treatment which applicable for the austenitic stainless steel is full annealing with rapid cooling to make sure the dissolved carbides reside in the water. The full annealing temperature range for the type 302 is from 1010°C - 1120°C [3]. This experiment intends to see the sample’s corrosion resistance when different temperatures applied with full annealing method because this still being unclear.
2. Experimental Procedure
The austenitic stainless steel type 302 has been cut into approximately 3 cm x 2 cm x 0.2 cm in length, width, and thickness. Three samples used in this experiment were fully annealed with different temperatures for an hour and a half. The first sample has been through full annealing treatment with temperature 1010°C (hereinafter referred as 1010ASS), the second one was fully annealed with temperature 1065°C (hereinafter referred as 1065ASS), and the last sample was fully annealed with temperature 1120°C (hereinafter referred as 1120ASS). The full annealing method for austenitic stainless steel is heat treatment with rapid quenching, which is different when it applied to the low-alloy or carbon steels [7].

To see the characterization before and after heat treatment, these were analyzed at UPP IPD Research Laboratory, Faculty of Mathematics and Natural Sciences, Universitas Indonesia, and the compositions of the austenitic stainless steel type 302 shown in TABLE 1. For the corrosion tests, all of the samples were dipped into 3.5 wt% NaCl solution and the data were collected using Potentiostat. The corrosion rate values were obtained from Faraday’s Law in Determination Corrosion Rates of Metals and Alloys shown below [8].

\[ r = C \frac{M_i n}{n \rho} \]  

(1)

where
\begin{itemize}
  \item M = atomic weight (g.mol\(^{-1}\))
  \item i = current density (A/cm\(^2\))
  \item n = number of electron involved
  \item \(\rho\) = density (g/cm\(^2\))
  \item C = constant (0,129 in mpy; 3,27 in mm/year; 0,00327 in mm\(^3\)/year)
\end{itemize}

The value of \(i\) has SI unit A/cm\(^2\), where the cm\(^2\) means the cross-sectional area of the samples which will be tested for the corrosion resistance. And for the experiment is 0,785 cm\(^2\) which obtained from the calculation of area of a circle with a diameter of 1 cm.

3. Results and Discussion

3.1. XRD Analysis
In Figure 1 we can see the XRD plot and the data of characterization shown in TABLE 2. For the 1010ASS, angle positions of the six peaks are 43,680°, 44,543°, 50,754°, 64,559°, 74,669°, and 82,072°. For the 1065ASS, angle positions of the six peaks are 43,776°, 44,594°, 50,755°, 64,636°, 74,767°, and 81,997°. For the 1120ASS, angle positions of the six peaks are 43,778°, 44,599°, 50,671°, 64,983°, 74,862°, and 81,928°. The highest first iron-alpha’s peak is on the 1010ASS which value is 116,92 count per second, then followed by the 1065ASS which value is 105,67 count per second and 97,99 count per second for the 1120ASS.

Table 1. The compositions of austenitic stainless steels type 302 identified by XRF

|   | Fe   | Cr   | Ni   | Mn   |
|---|------|------|------|------|
|   | 71 wt% | 19 wt% | 7 wt% | 2 wt% |
The largest d value for the first iron-alpha’s peak is on the 1010ASS which value is 2.03424 Å, then followed by the 1120ASS which value is 2.03311 Å for the 1065ASS. The lattice parameters have the same value for a, b, and c because the crystal system of these samples is cubic. For the iron-alpha, the lattice parameter change in each sample, where the biggest value is on the 1120ASS which value is 2.90586 Å, then followed by the 1010ASS which value is 2.89876 Å, and 2.89262 Å for the 1065ASS. For the iron-gamma, the biggest value of the lattice parameter is on the 1120ASS which value is 3.61667 Å and 3.61 Å for the 1010ASS and 1065ASS. The volume of the iron-alpha and iron-gamma are also
Table 2. Crystallographic parameters of the 1010ASS, 1065ASS, and 1120ASS.

| Sample       | 1010ASS     | 1065ASS     | 1120ASS     |
|--------------|-------------|-------------|-------------|
| First peak’s height (cps) |             |             |             |
| Iron-Alpha   | 116.92      | 105.67      | 97.99       |
| Iron-Gamma   | 20.94       | 31.71       | 16.30       |
| First peak’s d (Å) |             |             |             |
| Iron-Alpha   | 2.0342      | 2.0328      | 2.0331      |
| Iron-Gamma   | 2.0681      | 2.0711      | 2.0656      |
| Lattice Parameter (Å) |             |             |             |
| Iron-Alpha   | a = 2.8987  | a = 2.8926  | a = 2.9058  |
|              | b = 2.8987  | b = 2.8926  | b = 2.9058  |
|              | c = 2.8987  | c = 2.8926  | c = 2.9058  |
| Iron-Gamma   | a = 3.6100  | a = 3.6100  | a = 3.6166  |
|              | b = 3.6100  | b = 3.6100  | b = 3.6166  |
|              | c = 3.6100  | c = 3.6100  | c = 3.6166  |
| Volume (Å³) |             |             |             |
| Iron-Alpha   | 24,3576     | 24,2033     | 24,5371     |
| Iron-Gamma   | 47,0458     | 47,0458     | 47,3072     |
| Density (g/cm³) |             |             |             |
| Iron-Alpha   | 7.61        | 7.66        | 7.56        |
| Iron-Gamma   | 7.88        | 7.88        | 7.84        |
| Concentration (%) |             |             |             |
| Iron-Alpha   | 77.8        | 91.9        | 72.8        |
| Iron-Gamma   | 22.2        | 8.1         | 27.2        |
| Microstrain (%) |             |             |             |
| Iron-Alpha   | 0.157       | 0.000       | 0.187       |
| Iron-Gamma   | 0.189       | 0.000       | 0.315       |
| Crystallite Size (Å) |             |             |             |
| Iron-Alpha   | 208.1       | 27913.4     | 175.4       |
| Iron-Gamma   | 173.2       | 27913.4     | 103.8       |

changed following their lattice parameter’s value. The highest concentration of iron-alpha phase is on the 1065ASS which value is 91.9%, followed by 1010ASS which value is 77.8%, and 72.8% for the 1120ASS.

From the XRD’s plot in FIGURE 1(a), the only changes can be seen slightly is the peak’s height of iron-alpha phase, where the highest is on the 1010ASS, then followed by the 1065ASS, and the 1120ASS. This explains that increase in annealing temperature will decrease the peak’s height of iron-alpha phase.

It can be seen in FIGURE 1(b) that the highest crystallite size value is on the 1065ASS, while the other two annealing temperature has the lower value. However, 1065ASS has the lowest microstrain value compared by the other two samples. This explains that crystallite size value inversely proportional to the microstrain value [9].

Table 3. Data from the corrosion test of the 1010ASS, 1065ASS, 1120ASS in the NaCl solution 7wt%

| Wt % NaCl | Sample   | R (ohm)     | $E_{\text{corrosion}}$ (V) | $I_{\text{corrosion}}$ (A) | Corrosion Rate (mm/year) |
|-----------|----------|-------------|-----------------------------|-----------------------------|--------------------------|
| 7 wt%     | 1010ASS  | $3.566 \times 10^2$ | -1.099 | $7.206 \times 10^{-5}$ | 0.0587 |
|           | 1065ASS  | $2.789 \times 10^2$ | -1.080 | $9.211 \times 10^{-5}$ | 0.0751 |
|           | 1120ASS  | $3.894 \times 10^2$ | -1.091 | $6.599 \times 10^{-5}$ | 0.0538 |
Figure 2. (a) Potentiodynamic polarization curves of the samples in 7 wt% NaCl; (b) Relation between crystallite size and corrosion rate of the 1010ASS, 1065ASS, 1120ASS

3.2. Potentiodynamic Polarization Curves Analysis

The mechanism of corrosion of these materials is the occurrence of anodic dissolution on their surfaces and then followed by ionization and hydrolysis of the samples [2,10]. These phenomena are called the anodic and cathodic reaction, which its potentiodynamic curves shown in FIGURE 2(a).

Figure 2(b) summarizes the data from TABLE 2 and TABLE 3, which is display the relation between corrosion rate and crystallite size into a plot. The results obtained are convinced by another experiment which explains that an increase in grain size (or crystallite size) is proportional to the increase of the corrosion rate value (i.e corrosion resistance reduced) [11].

From Table 3, it can be seen that 1120ASS has the lowest corrosion rate, which its value is 0,0538 mm/year. It means the 1120°C annealing temperature is better for improving corrosion resistance from the other annealing temperatures used in this experiment. This is due to the nearness to the ideal temperature which is limit the grain growth is 1095°C [3]. The 1010ASS has the next best annealing
temperature for improving corrosion resistance, which its value is 0.0587, even though the corrosion rate difference between 1120ASS and 1010ASS is not significant. This temperature (1010°C) is close to 1030°C, which is used to some manufacturing industry to annealing their material. And the last annealing temperature which has the highest corrosion rate value is 1065°C which its value is 0.0751. This explains that the increase in annealing temperature is not directly proportional to the corrosion rate value. It might be related to the carbide precipitation and need a further experiment.

4. Conclusion
The three different annealing temperatures used in this experiment affect the crystallographic parameters of the samples, and also affect the corrosion rate value, in which the best annealing temperature to improve corrosion resistance is 1120°C, followed by the 1010°C, and then 1065°C. The corrosion rate also depends on the grain or crystallite size, in which the decrease of grain size is directly proportional to the corrosion rate reduction.

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