Identification of Inhomogeneous Temperature on Stainless Steel using Statistical Analysis

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Corrosion in stainless steel, abbreviated as SS, is still an exciting topic to study. Even though SS is a corrosion resistance material, this property will be degraded when exposed to high temperatures for a long time because of σ phase, such as a Fe-Cr compound, formation. The presence of this phase can be observed using a special chemical etchant solution that will give five specific colours to this phase: light brown, dark brown, bluish brown, light blue, and dark blue. In this study, the specimen sample is from ASTM A297. Furthermore, the metallography process is carried out to obtain microstructure images that describe the σ phase. Here, two grains were taken as objects to discretize with one of them was around the specimen sample center and the other was close to the boundary with the environment. The discretization resulted in a 2 x 5 frequency table, called contingency table, that is analysed by the independence $\chi^2$-test. The contingency table is also represented geometrically in cartesian. The study shows that two grains were not independent. The grain which was around the specimen sample center contained many σ phases dominated by light blue colour (43%). In other words, the prolonged heating did not give homogeneous corrosion level.

Keywords: σ phase, $\chi^2$-test, corrosion, stainless steel, statistical analysis

1. Introduction

In human life, there are various types of materials that are often used such as stainless steel, abbreviated as SS, that is a high-level corrosion resistant. The factory industries such as chemical, oil, gas, pipelines, and material storage facilities highly depend on SS [1]. SS contains Iron (Fe), Chromium (Cr), Nickel (Ni), Manganese (Mn), Silicon (Si), and Carbon (C) which were homogeneously distributed. A chemical element that protects this steel from oxide is Cr [2]. Over time, Cr will form an intermetallic phase Fe-Cr compound in SS when heated above 550°C for a long time (greater than 100 hours) [3].

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The intermetallic phase Fe-Cr compound in SS is σ phase. It will appear and decrease the mechanical properties and corrosion resistance of this steel [4][5]. The decrease in corrosion resistance is proportional to the increase in the number of σ phases that appear [6]. Less corrosion resistance also occurs in the Cr-depleted regions [7]. The characteristic of σ is tetragonal, not cubic, easily separated from the other phases [8] and is formed because of eutectoid reaction, \( \delta \rightarrow \sigma + \gamma \). Where \( \delta \) is delta-ferrite and \( \gamma \) is austenite [9]. Hwang and Park, 2009 [10] explained that the amount of ferrite that could be transformed to intermetallic such as σ phase increases when the temperature of solution heat treatment increased. Hau and Seijas 2006 [11] show that the σ phase exposure is at 560-980°C. There is a correlation between precipitation and temperature history in the heat-affected zone [12] and is indicated by the different colour occurrences. Based on the colours, the local temperature and corrosion level can be predicted.

Several studies have discussed corrosion with statistical approaches. Hamzat et al. [13] calculated descriptive statistics (number, mean, standard error of mean, standard deviation, coefficient of variation, minimum, and maximum) for corrosion rate of mild steel in fruit juice environment and modeled it through regression analysis. Yan et al. [14] also used regression analysis to model the corrosion rate prediction. In addition to statistical modeling, fitting distribution also had an important role in the study of corrosion. Morales et al. [15] fitted lognormal distribution to experimental corrosion depth for each tendon cross-section. In this study, corrosion is studied especially for the σ phase both in terms of SS type ASTM A297 material and its statistical data analysis through the discretization process. Furthermore, the discretization result was tabulated in a contingency table of two grains, A and B. The position of A was around the center and B was close to the boundary with the environment, see Figure 3(b). Moreover, their independence was tested using \( \chi^2 \)-test. The σ phase had a level of corrosion that started from low to high, as light brown (CM), dark brown (CT), bluish brown (BKc), light blue (BM), and dark blue (BT) through linkert observation. There is no previous article that analyzes σ phase with a statistical approach. This study has an important role since one of SS quality parameter depends on corrosion caused by σ phase. Therefore, this study will discuss this as a novelty.

2. Methodology

The methodology consists of nine steps. First of all is the material preparation and then investigation of the specimen sample to the microscope. The third step is to determine grain A and B based on the specimen image. Furthermore, the two grains were discretized through circle grid, the colour grid frequency were counted and were then tabulated in a contingency table. The three last steps were analyzed using descriptive statistics, visualized through column vector diagram, and tested their independency hypothesis (Figure 1).
2.1 Material

An object of observation is the SS type ASTM A297 grade HH. The age of this object is 24 years and the material specimen is described in figure 2 (a). The first step in the experiment is to cut the specimen to get a small sample of our material; this step needs time (+/- 5 minutes) and the cutting process of it was using a jigsaw, then the specimen with a 50% thickness was used here.

![Figure 1](image1.png)

**Figure 1.** The methodology of this research

![Figure 2](image2.png)

**Figure 2.** The material (a), the cutting process (b), and the specimen used in this study (c-d)

![Figure 3](image3.png)

**Figure 3.** An image of the specimen with a 50% thickness (a) and two selected grains to be observed (yellow contour) (b)
2.2 Procedure

The material preparation process is already described in figure 2. Specimens were prepared by cutting, resin mounting, and surface preparation which included grinding and polishing. To reveal the microstructure, specimens were etched using modified Murakami reagent for 5 seconds at a temperature of 25°C. The reagent consists of 30 g K3Fe(CN)6, 30g KOH, and 60 mL water. To obtain the detailed grain image, the specimen was investigated using an optical microscope. For this purpose, the highest lens magnification was taken (100x objective). From the image, two grains were selected and named A and B, respectively (see Figure 3b), which were analyzed in the following steps.

To calculate each colour, the image of each grain has to be partitioned as grid. Here, the circle is the most suitable one among rectangular, triangle, and trapezium. Manually, the circle grid was made in each grain, A and B, see Figure 4(c) and (d).

![Figure 4](image)

Figure 4. Observed optical micrographs for grain A (a), observed optical micrographs for grain B (b), result of the circle grid discretization for grain A (c), and result of the circle grid discretization for grain B (d)

The radius of circle is constant for each grain. Then, each colour is counted for A and B. See Table 1 below.

| Colour Level | A (30) | B (48) |
|--------------|--------|--------|
| CM           | 1 1 1 1 | 1 1 1 1 1 1 1 1 1 1 |
| CT           | 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 | 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 |
| BKc          | 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 | 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 |
| BM           | 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 | 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 |
| BT           | 5 5     |        |

*Level 1, 2, 3, 4, and 5 in colour category is linkert data
Furthermore, A and B denoted in rows and colours represent columns in matrix or contingency table, see table 2 with black colour as the observed value.

| Grain | CM | CT | BKc | BM | BT | Total |
|-------|----|----|-----|----|----|-------|
| A     | 2 (4.62) | 5 (8.85) | 10 (8.85) | 13 (6.92) | 0 (0.77) | 30 (30.01) |
| B     | 10 (7.38) | 18 (14.15) | 13 (14.15) | 5 (11.08) | 2 (1.23) | 48 (47.99) |
| Total | 12 | 23 | 23 | 18 | 2 | 78 |

Therefore, the data in the contingency table can be analyzed by descriptive statistics, and displayed in a column vector diagram. Then, the independence test using $\chi^2$ was applied to identify the dependency between two grains. It shows that the frequency of BKc was almost the same in both grains, while other colours were pretty different, predominantly the CT. In addition, based on the total count in a row, grain A (30) and B (48) were not independent. The next step was analyzing the contingency table using descriptive statistics and testing the independence of grain A and B. The observed frequencies used in this test are presented in table 2. The total in row and column in table 2 are called marginal frequencies [16].

### 3. Results and discussion

The result consists of descriptive and inference statistics. The summary statistics and the boxplot of each grain is represented in table 3 and figure 5, respectively. Table 3 describes the descriptive statistical result and it is presented graphically in figure 5. The bar chart uses the frequency (number) of occurrences and the data categories of interest to summarize the data [17].

| Grain | N | Min | Max | Median | Q1 | Q3 | Skewness | Kurtosis |
|-------|---|-----|-----|--------|----|----|----------|----------|
| A     | 30 | 1   | 4   | 3      | 2.75 | 4  | 0.6      | 0.3125   |
| B     | 48 | 1   | 5   | 2      | 2   | 3  | 1        | 0.1667   |

*Skewness and Kurtosis calculated by order statistic measure

According to table above, the colour frequencies for both grains were quite different. From the median column, A was 3 and B was 2, respectively, which means grain A tends to be blue and grain B tends to be brown. Furthermore, A and B were moderately skewed between 0.5 and 1, and their kurtosis was less than 3 (platykurtic). Surprisingly, the boxplot of grain B had 2 outliers (dark blue). Given the 2 outliers, grain B was positive skewed.

For the colours, the summary statistics is represented in the histogram below:
Figure 6. The colour frequency of grain A and B

For light brown and dark brown, i.e. the lowest and the second lowest corrosion level, happened in grain B. In contrast, the second highest corrosion level happened in grain A at a 13:5 ratio.

In addition, the identification process of heating temperature displayed through a column vector diagram was based on the normalizing data in a contingency table. There were two orthogonal axes and five vectors, but, geometrically, the diagram with five orthogonal axes and two vectors can also be made [18]. This diagram is presented in figure 7, along with a map of the colour positions relative to grains A and B.

Figure 7. Column vector diagram and position map of grain A vs B

In this study, the recessive vectors are CM, and BT. If the observation time is longer, then this result can be different because of the process of changing colour sequence. Then, the position map is made based on the position of each vector in the column vector diagram for grain A and B assuming that they are independent of each other. From these results, grain A tends to be blue while grain B tends to be brown. The blue colour indicates the appearance of the $\sigma$ phase, so grain A contains more than grain B. Thus, the local temperature of grain A is higher than grain B, because grain B’s position is closer to the surface so there is a heat transfer process from grain B to the environment.

The independency of grain A and B was also tested using $\chi^2$-test. The expected frequencies were calculated through equation (1) and displayed in red colour in table 2.

\[
\text{expected frequency} = \frac{\text{(column total) x (row total)}}{\text{total}} \tag{1}
\]
The example of calculation of expected frequency can be described as follows:

\[ E[BT \text{ in Grain A}] = P(BT) \times P(A) \times \text{total} = \left( \frac{2}{78} \right) \left( \frac{30}{78} \right) (78) = 0.77 \]

\[ E[CT \text{ in Grain B}] = P(CT) \times P(B) \times \text{total} = \left( \frac{23}{78} \right) \left( \frac{48}{78} \right) (78) = 14.15 \]

then these values will be used to calculate the \( \chi^2 \)-test through equation (2).

\[ \chi^2 = \sum_i \frac{(o_i-e_i)^2}{e_i} \]  

\[ \chi^2 = \frac{(2 - 4.62)^2}{4.62} + \frac{(5 - 8.85)^2}{8.85} + \frac{(10 - 8.85)^2}{18 - 14.15)^2} + \frac{(13 - 6.92)^2}{14.15} + \frac{(0 - 0.77)^2}{6.92} + \frac{(10 - 7.38)^2}{23 - 11.08} + \frac{(0.77)^2}{11.08} + \frac{(2 - 1.23)^2}{1.23} = 15.31 \]

The degree of freedom of this case is \( v = (r - 1)(c - 1) = (2 - 1)(5 - 1) = 4 \). According to the \( \chi^2 \)-test table, the critical value is \( \chi^2_{0.05} = 9.488 \). Therefore, the hypothesis of grain A and B are independent and was rejected with a significant level less than and equal to 5%. This means that eventhough the grains of A and B are from two separate locations in the specimen sample, they are dependent on each other. In this study, based on figure 2 (b), table 2, and table 3, grain A has a higher local temperature than grain B because grain B’s position was closer to the boundary, see figure 2(a). Since heat is spread around, then the heat was transferred from grain B to the outside environment. Therefore, grain A tends to be blue and grain B tends to be brown, so grain A has a higher corrosion level than grain B.

4. Conclusion

In this study, the corrosion level through discretization process of two grains in the ASTM A297 Grade HH material specimen with age is 24 years and its independency test was studied. The results explain that descriptive statistics and independence test analysis can identify local temperature gradient based on the frequency of each colour occurrence by the discretization process, namely the process of making circles in the continuous observation area on \( R \). The result describes that grains A and B were not independent. Furthermore, grain A tends to be blue, which means that A contained more \( \sigma \) phases during the heating process, so the local temperature of grain A was higher than grain B. It might be due to the position of grain B which was closer to the surface so that there was a heat transfer process from grain B to the outside environment. Accordingly, grain B tends to be brown. The discretization process with a smaller radius and observations with more than two grains will be discussed in further research.

Acknowledgment

This research was supported by Institut Teknologi Bandung through the Penelitian, Pengabdian Masyarakat, dan Inovasi (PPMI) 2021 programme, Faculty of Mathematics and Natural Science. The authors would like to thank the anonymous reviewers for providing constructive comments to improve the manuscript. The first author would like to thank Institut Teknologi Bandung for providing the scholarship.

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