Normalising of 316L Stainless Steel using Temperature and Holding Time Variations

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Abstract. Materials for some component must be through machining processes, for example, is a lathe. Lathe causes internal stress. One of many ways to avoid the internal stress is normalising. Normalising of the heat treatment is heating materials above austenite temperature, holding for a period for transformation and air cooling. This research was conducted to analyse the effect of holding time and temperature normalising of 316L stainless steel toward hardness and microstructure. Hardness testing was measured by microhardness Vickers tester, and the characterisation was used optical microscope and XRD. The highest hardness was obtained 193.2 HVN from normalising with a temperature of 800ºC and holding time of 30 minutes, while the lowest was 166.3 HV using 950ºC and 90 minutes. The microstructure was obtained austenite and ferrite phase. The normalised specimen had a wider peak profile than the unnormalised specimen, can be observed at 50.9º of the 2θ angle which had bigger FWHM.

1. Introduction
The use of metal as the main material of production has an increasing value for industrial. Steel of raw materials was used as tool steel and automotive components. Kinds of steel can be applied as industrial components such as stainless steel. Stainless steel is widely used as engine components such as gears, tableware, shafts and other components [1]. Stainless steels certainly cannot be separated from the machining process, especially the lathe process. The lathe process is done by slicing the specimen by the chisel edge to form a product using a rotating process. In the process of the lathe, the chisel edge often damaged because abrasive resistance decreased. The shifts between chisel edge and workpiece cause the decreasing of abrasive resistance. As a result of the continuous shift, the temperature of the material is highly increased. The temperature differences on the material surface and the depth of the material causes residual stress. Residual stress effect to mechanical properties such as hardness properties [2].

One way to improve the mechanical properties is heat treatment such as normalising. Normalising is a process that is done by heating the steel until it reaches the austenite temperature. The temperature is held for a while and then cooled slowly by using air cooling medium. The purpose of normalising treatment is to increase uniformity and to eliminate residual stress. The heating temperature of normalising is about 50º C above the critical temperature Ac₃ for hypereutectoid steels to obtain homogeneous Austenite [3]. Some factors during heat treatments contribute to the mechanical properties of the treated material. Carbide phases were produced after heat treatment. Some aspects of carbides including size, type, volume percentage, microstructures have important roles in mechanical properties [4]. Therefore, it should be controlled to avoid decreasing of...
mechanical properties. This study aims to investigate the temperature and to hold time effect to hardness and microstructure for 316L SS using normalising treatment.

2. Materials and Method

2.1. Materials

The materials used in this research are a rounded bar of Stainless steel type 316L. The compositions of 316L stainless steel material can be seen in Table 1. Sample preparation was done by cutting stainless steel that had been in the lathe using a hacksaw with a length of 10 mm, and diameter 25 mm for nine specimens. The dimension for the sample without normalising is 10 mm long, and 10 mm diameter of 1 specimen. The schematic of the sample dimension was shown by figure 1.

| Elements percentage for 316L SS (%) | C  | Si   | Mn  | P   | S   | Cr  | Mo  | Ni  | N  |
|-----------------------------------|----|------|-----|-----|-----|-----|-----|-----|----|
| C                                 | 0.020 | 0.49 | 1.79 | 0.040 | 0.010 | 16.88 | 2.06 | 10.6 | 0.0655 |

2.2. Method

The sample was prepared by the desired dimension. The following step is heat treatment. The sample was heated by muffle furnace at different temperatures which were 800°C, 950°C, and 1100°C. Those temperatures formed single phase which improved the mechanical properties. Holding time of heat treatment was also used as a variation for 30, 60, and 90 minutes. Then, the sample was cooled by air as normalisation media. Prepared samples were characterized by hardness test using microhardness test, microstructure analysis, and X-Ray Diffraction (XRD) analysis. For hardness test, samples should be smoothed by grinding and polishing step. The mechanism of testing materials based on ASTM E 384. Vickers’s microhardness can be calculated from the diagonal of the indentation area. Polished samples were etched to appear grain boundaries. The microstructure was observed by a microscope optic, and XRD identified the sample component.

3. Results and Discussion

3.1. Hardness Properties

Figure 2 shows the hardness number of each specimen treated by temperature variations. From the figure was known that high temperatures treatment decrease the hardness. Phase transformation at
higher temperatures is faster than the phase transfers at low temperatures, so the phase formed on the specimen effects the hardness value. The austenite phase dominates the phase, and the characteristic of the austenite phase is soft. The hardness of Normalised sample at 1100°C and held for 30 minutes achieved 181.4 HV. The raw material of 316L SS was tested by a hardness test to compare the effect of heat treatment, and it has 189.5 HV. Temperature treatment caused decreasing of hardness at 950°C but increased at 1100°C [5]. Increasing temperatures promote the formation of carbide precipitate and lead to decrease the hardness. Otherwise, decreasing of hardness may due to grain size. Fine grain size has more grain boundaries and has a large number of carbides. Coarsening grain above 950°C of temperature treatment promote lower precipitation carbides to increase the hardness of the normalised material. For further reasons, increasing lath size, precipitate size, subgrain in dislocation have an important role to decrease in hardness while increasing normalising temperature [6]. It has mentioned that increasing dislocation density at higher normalising temperature can decrease in hardness.

Figure 2. The hardness number with normalising temperature.

Figure 3. The hardness number with holding time variations of normalising.

Figure 3 shows that longer time to hold the samples during normalising promotes gradually decrease of hardness. The normalised sample reaches the highest hardness at 800°C held for 30 minutes which is 193.2 HV. The temperature of 950°C achieves the maximum hardness value of 180 HV with holding time 30 minutes and decreased while increasing the holding time. Depending from hardness testing, holding time of normalising decreases in hardness but significantly increase during 1100°C of normalising temperature. Accumulations of nitrogen content increase on the surface area. High amounts of nitrogen cause differences of concentration inside and outside materials. It promotes diffusion from the surface to inside materials. Increasing of nitrogen content enhance the hardness. Detail information about the hardness of normalised samples is presented by Table 2.

| Temperature | Holding time | Repetition | d1     | d2     | HV     | Average ± Dev |
|-------------|--------------|------------|--------|--------|--------|---------------|
| Raw Material|              | 1          | 54.29  | 54.31  | 188.6  | 189 ± 2.14    |
|             |              | 2          | 53.83  | 53.83  | 191.9  |               |
|             |              | 3          | 54.4   | 54.4   | 187.9  |               |
| 30 minutes  |              | 1          | 53.48  | 53.46  | 194.5  | 193.2 ± 2.73  |
|             |              | 2          | 54.09  | 54.09  | 190.1  |               |
|             |              | 3          | 53.38  | 53.4   | 195.1  |               |
| 800°C       | 60 minutes   | 1          | 54.56  | 54.56  | 186.8  |               |
|             |              | 2          | 53.56  | 53.6   | 193.7  | 190.1 ± 3.46  |
|             |              | 3          | 54.13  | 54.15  | 189.7  |               |
| 90 Minutes  |              | 1          | 53.77  | 53.79  | 192.3  |               |
|             |              | 2          | 54.4   | 54.4   | 187.9  | 189.8 ± 2.25  |
|             |              | 3          | 54.2   | 54.2   | 189.3  |               |
| Temperature | Holding time | Repetition | d1   | d2   | HV | Average ± Dev |
|-------------|--------------|------------|------|------|----|---------------|
| 950°C       | 30 minutes   | 1          | 56.1 | 56.1 | 176.7 | 180 ± 2.88   |
|             |              | 2          | 55.37 | 55.39 | 181.3 |
|             |              | 3          | 55.26 | 55.3  | 182   |
|             | 60 minutes   | 1          | 56.18 | 56.18 | 176.2 | 177.3 ± 2.48 |
|             |              | 2          | 56.26 | 56.32 | 175.5 |
|             |              | 3          | 55.57 | 55.57 | 180.1 |
|             | 90 minutes   | 1          | 57.66 | 57.64 | 167.3 |
|             |              | 2          | 57.85 | 57.85 | 166.2 |
|             |              | 3          | 57.97 | 57.97 | 165.5 |
| 1100°C      | 30 minutes   | 1          | 55.25 | 55.25 | 182.2 | 181.4 ± 1.74 |
|             |              | 2          | 55.67 | 55.69 | 179.4 |
|             |              | 3          | 55.19 | 55.19 | 182.6 |
|             | 60 minutes   | 1          | 55.86 | 55.86 | 178.2 |
|             |              | 2          | 55.01 | 55.01 | 183.3 |
|             |              | 3          | 54.55 | 54.57 | 186.8 |
|             | 90 minutes   | 1          | 54.4  | 54.4  | 187.9 |
|             |              | 2          | 55.37 | 55.39 | 181.3 |
|             |              | 3          | 54.75 | 54.79 | 185.4 |

3.2. Morphological Analysis

From the metallography test results obtained that microstructure of specimens contains austenite (white colour), ferrite (dark) and chrome carbide (black spots). In the non-normalising specimen shown in figure 4 has a microstructure of austenite and ferrite phases but is dominated by the ferrite phase.

![Figure 4. Microstructure image of raw material (316L SS)](image)

The 316 L stainless steel microstructure with normalising treatment at 800°C is shown by Figure 5. There are austenite, ferrite and chromium carbide phases. Austenite phase dominates microstructure at normalised 800°C. The appearance of chromium carbide is due to the normalising process of slow cooling. The phases are still dominated by austenite and low amount of Chromium carbides. This sample has the highest hardness because it has low Cr carbides. They appear at grain boundaries as fine carbides. It is considered that the presence of Cr contributes to hardness decreasing [7].

The increasing normalising temperature at 950°C promotes more carbides and ferrite compared to normalised at 800°C. It is presented by figure 6. Austenite grain size is larger than ferrite and shows the obvious grain boundaries. Cr carbides appear as a precipitate. Ferrite and Cr carbides uniformly distributed in the austenite matrix because the diffusion rate of a solute atom is faster at the higher temperature [8]. Precipitation occurred near 950°C. Therefore, the hardness gradually decreased after 950°C. It is proven by Figure 7 that show more Cr carbides for normalised at 1100°C. Coarsening grain was observed for normalised temperature at 1100°C. It composed carbides and austenite matrix. The grain size is increased with increasing normalising temperature and Cr carbides distributed on the austenite matrix. The main carbides distributed inside the grains or at grain boundaries. Uniformly distributions of Cr carbides are dissolved into the matrix after treated at higher temperature [9]. More Cr carbides at high temperature caused by the good stability of Cr at high temperatures [10].
Figure 5. Microstructure image of normalised 316L SS at 800°C for 30 minutes

Figure 6. Microstructure image of normalised 316L SS at 950°C for 30 minutes

Figure 7. Microstructure image of normalised 316L SS at 1100°C for 30 minutes

3.3. XRD Analysis

Figure 8 shows the diffraction patterns of normalised 316L SS using temperature variations for 30 minutes. The strongest peaks of XRD indicate as austenite (γ) peaks. The position of γ peaks at 2θ=43.5°, 50.9°, 74.7°, 90.5° [10]. XRD pattern confirmed the presence of austenite phase from microstructure as mentioned above but XRD can not identify Cr carbides. Low amount of carbides may cause it. The diffraction peak of 2θ= 95° showed the presence of δ-ferrite phase. The lowest intensity explained low amount δ-ferrite phase. XRD cannot detect the phase under 5wt%. During the cooling at air media, phase transformation occurs from δ → γ and it was controlled by diffusion. The existence of δ-ferrite phase shows that transformation to the austenite phase is not completed [11].

The diffraction peak of normalised sample at 2θ = 50.9° has a wider peak shift than a 316 L stainless steel specimen without normalising treatment. The shift and widening peak are indicated by the magnitude of the widening number (FWHM) as shown in table 3. The slight peaks of XRD pattern mean crystalline structure.
Figure 8. XRD Pattern of normalised 316L SS using temperature variations for 30 minutes.

Table 3. XRD data for FWHM left

| Raw Material | Normalised at 800°C | Normalised at 950°C | Normalised at 1100°C |
|--------------|---------------------|----------------------|----------------------|
|              | Pos. [°2θ.]         | FWHM Left [°2θ.]     | Pos. [°2θ.]         | FWHM Left [°2θ.]     | Pos. [°2θ.]         | FWHM Left [°2θ.]     |
|              | 43.5792             | 0.1004               | 43.5789             | 0.1004               | 43.5743             | 0.1338               |
|              | 50.9097             | 0.3346               | 50.9154             | 0.0415               | 50.7653             | 0.0415               |
|              | 74.7015             | 0.4015               | 72.5446             | 0.4015               | 74.6664             | 0.3346               |
|              | 90.5995             | 0.5353               | 74.7500             | 0.5353               | 90.6769             | 0.8029               |
|              | 95.9431             | 0.8029               | 90.8488             | 0.9628               | 95.9333             | 0.6691               |

4. Conclusions
Based on the investigation of normalising treatment for hardness and microstructure analysis, can be concluded that the high normalisation temperature affects the decreasing of hardness. For normalised 316L stainless steel at 1100°C has a higher hardness than normalised at 950°C because of more Cr carbides and ferrite phases. Moreover, holding time also promotes the decreasing of hardness. Longer time for normalised 316L stainless decreases the hardness but increases at a temperature higher than 950°C. Moreover, macrostructures of normalised 316L stainless steel using temperature and holding time variations present austenite, ferrite, and Cr carbides. High Cr Carbides amount occurred while increasing normalisation temperature.

5. References
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