The Influence of Holding Time to Mechanical Properties of Steel Processed with Powder Metallurgy

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Abstract. Steel with very small grain has high strength but still have ductility. Heating the steel until reach to austenite temperature can make the grain of the steel will growth and reduce the hardness of the steel, to prevent this it is necessary to know the effect of holding time on steel hardness. The hardness is indicating of grain growth of the steel. In this research, we investigated the effect of holding time to the hardness of very small-grained steel. Heat treatment was done at 1100 °C using an electric furnace. Micro-hardness testing used a micro-Vickers method using 300 gram forces and indentation time 10 minutes. Microstructure observation was done with metallurgical microstructure. The etching solution for microstructure observation was nital solution. The heat treatment process of steel powder metallurgy with a holding time of 60 minutes will cause hardness to decrease from 245 in holding 15 minutes to 207 and grain size grow from 1 µm to 4 µm.

Keywords: Heat Treatment, hardness, Grain Size, Holding Time

1. Introduction

Steel is the most widely used metal in human civilization. The metal consumption industries 70% is steel[1]. Steel has many advantages, these are high strength, low cost and easy to produce and manufacturing[1][2]. Mechanical properties of steel depend on the chemical composition, adding alloying element will changed the mechanical, physical and chemical properties of the steel[3]. The addition of carbon to steel will increase the hardness and tensile strength, the addition of chrome made the steel more resistant to corrosion. Manganese makes steel more shock resistance[4][5].

The most widely material adding to steel is carbon[1]. The steel with the addition of carbon known as carbon steel. Carbon steel has wide tensile strength, low strength, and low carbon steel and very high strength on high carbon steel. Beside amount of the carbon, heat treatment can modify mechanical properties of the carbon steel[5].

Another way to strengthening steel is reduce grain size. Steel with a small grain size will increase its strength but its toughness also increases unlike in carbon steel[6]. Small grain size can be obtained by the thermomechanical process[7][8], dynamic recrystallization [9]and the powder metallurgy process[10][11][12]. powder metallurgy process produce steel with very fine and homogeneous grains also the mechanical properties are more homogeneous compared to another methode[13][14][6].
Heat treatment of steel can be done to certain carbon steel. The carbon steel can be heat treated is steel with minimum carbon 0.2%[15]. The heat treatment process on steel is heating the steel until austenite temperature, holding it at this temperature and quenching in certain media[16]. The media for quenching depend on the steel composition[2]. The heating of steel until austenite temperature can make the grain of the steel changed. This grain size changed can be affected by properties of steel, especially for steel with very small grain[17]. At high-temperature grain will grow, according to hall-petch equation large grain size will reduce the strength of the metal.

Steel with very small grain has high strength but still have ductility [18]. Heating this steel can make the grain of the steel will be growth. In this research, we investigated the effect of holding time to the hardness of very small-grained steel.

2. Methods

2.1. Materials
The metal used is steel was from the powder metallurgical process of with a length of 10 mm, width 10 mm and thickness of 5 mm. The chemical composition is C 1.18 %, Chrome 4.2%, Molybdenum 5.0 %, Wolfram 6.40 %, Vanadium 3.10 %.

2.2. Heat Treatment
Steel produced by the powder metallurgy process is treated heat using an electric furnace. The test specimens used were rectangular with a length of 10 mm, width of 10 mm and height of 4 mm respectively. The heating process is carried out in two stages, the first stage being heated to a temperature of 500 ℃ and held for 15 minutes. After that, heated to a temperature of 975 ℃ and held for 15, 30, 45, 60 and 75 minutes respectively.

2.3. Microharness Test
After heat treatment the specimens were sanded using sand papers from sizes 100, 200, 500, 1000 and 5000. This process is carried out until the test specimen surface free from scratches. Micro hardness measurements require a scratch fee surface. The hardness test were done using the micro Vickers method. The load used is 300 grams force with a loading time of 10 seconds. The indenter was a diamond pyramid, for each test sample was tested 3 times.

![Figure 1. Micro structure Test Preparation](image)
2.4. Microstructure observation

After the hardness test, microstructure is observed using a metallurgical microscope. The microscope used is the metallurgical microscope Olympus GX 41 equipped with Olympus DSLR cameras. Objective lenses used with 40X magnification and camera adapters with a correction factor of 1.2.

Before the microstructure observed the surface of the steel must be etched. The etching solution used were nital solution. The nital composition were 97% methanol and 3% HNO₃. The etching process is done by dipping the surface of the test specimen for 10 seconds in the nital solution. After that the surface of the test specimen was cleaned with flow water to remove etching solution.

3. Results and Discussion

3.1. Micro Hardness

Macro hardness test of the can be seen in Figure 2. The micro hardness test decreasing the steel hardness correspond to the heating time. The lowest hardness occurs on steel with holding time 60 minutes, while the highest hardness occurs in steel with heating for 15 minutes.

![Hardness Vs HoldingTime](image)

**Figure 2.** Effect of Holding Time to Steel Hardness

The steel hardness change indicates a change in the microstructure of steel. The mechanical properties of steel are not only influenced by chemical composition but also by microstructure. This heat treatment process can change the microstructure of steel, in this study the micro structure changes due to the heating time. This change in microstructure will be proven by microstructure

3.2 Microstructure

The microstructure of the steel before heat treatment can be seen on figure 1 and structure micro for steel with holding time 15 minutes in figure 4 and steel with holding time 60 minutes figure 5. The size of the steel grains before the heat treatment process is 1 µm, the size of steel grains after heat treatment with a holding time of 15 minutes is 2 µm (figure 4) and the size of steel grains after heat treatment with a holding time of 15 minutes is 4 µm (figure 4).
Figure 3. Microstructure of Steel Before Heat Treatment

Figure 4. Microstructure of Steel Holding time 15 minutes
The observation of microstructure shows there was a very large grain growth, before the heat treatment the grain size was 1 micrometer and after heat treatment became to 4 micrometers, this shows that the grain growth occurred very large. This will make hardness of the steel decrease, decreasing of the hardness due to holding time austenite temperature [17][20]. To prevent the growth of grains in steel must be carefully calculated so that the grain growth that occurs is not too large[21].

4. Conclusion

The heat treatment process of steel powder metallurgy with a holding time of 60 minutes will cause hardness to decrease from 245 in holding 15 minutes to 207 and grain size from 1 micrometer to 4 micrometers.

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