The Effect of Vacuum Quenching on Corrosion and Hardness of the Surface of SKD61 Steel

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Abstract. JIS SKD61 Steel is chromium-molybdenum hot work steel which has composition 5.58 wt% Cr – 2.51 wt% Mo. This steel is a well-known material used as a die material to get good mechanical and hardening properties. Besides this material should have good corrosion resistance. Samples made of SKD61 steel were subjected to vacuum heat treatment at 1030°C for 3 hours with step quenching in different rates of quenching. The difference in the cooling speed of each sample causes properties on the surface to vary. Micro Hardness Vickers is a tool used to determine micro-scale hardness. While to test corrosion resistance, electrochemical testing was carried out using NaCl solution with variations in concentrations of 1%, 3.5%, and 7%. X-Ray Diffraction was used to characterize the crystal structure and the crystallite size. The results show that the speed of quenching affects the hardness of the surface of the material, structure and corrosion resistance.

1. Introduction

JIS SKD61 steel is a chromium-based material that is included in three main groups in hot tool steel. This type of tool steel with medium carbon content, which can be used for hot work applications, such as aluminum extrusion die, forging molds, plastics, and nonferrous injection mold cavities and all types of molds for heat work applications, involving shocks.[1] JIS SKD61 steel is widely used in industry because it has good hardenability, high toughness, and excellent heat resistance. In addition, SKD61 steel has moderate wear resistance, so it can be carburated or nitrided with the aim of increasing surface hardness. However has the effect of decreasing heat resistance. [1,2]

SKD61 steel is a type of steel that is combined with a number of chromium as a substitute for iron to form carbides, which can prevent decarburization by establishing carbon as carbide. Research shows the addition of 1% chromium produces 10.5 MPa stronger than normal steel with the same carbon content with a slight increase in toughness.[1] Although chromium content increases decarburization resistance, significant decarburization can still be found on heated SKD61 steel if the heat treatment is not well controlled.[3]

One way to increase material hardness is by vacuum heat treatment. Vacuum heat treatment is the process of heating material in a vacuum furnace. When in this condition, the material is heated in a furnace surrounded by a vacuum. This is done to prevent oxidation in the material because when heating at high temperatures allows oxidation quickly. After the heat treatment steps are completed,
the next process that is carried out is vacuum quenching. In the vacuum quenching process, there are 2 main factors that influence it, namely gas pressure and gas velocity. The gas that is usually used for cooling is nitrogen, argon, helium, or hydrogen.[4] At present, the vacuum heat treatment has been used widely, one of which is widely used in the automotive industry. Vacuum heat treatment is widely used in industry because it has due to its dimensional stability and low induced stresses in treated parts due to uniform heating and cooling.[5]

Steel SKD61, which belongs to the medium carbon steel, has fairly good corrosion resistance, but the resistance to corrosion is still lower than stainless steel. This is because the medium carbon steel only has about 5 wt% Chromium which does not have enough chromium to form the chromium oxide layer. This allows oxygen to bind with iron to produce iron oxide or rust. However, increased resistance to corrosion can be done by means of heat treatment. Therefore, it is very important to pay attention to the various stages of heat treatment carried out.[6]

2. Experimental Procedure

2.1. Material
This experiment uses medium carbon steel type SKD61 Steel, whis is divided into four different sized samples and each sample is given a different treatment. The first sample is Steel A LQ (Low Quenching) measures 5 cm x 2.6 cm x 0.6 cm, the second sample is Steel B MQ (Medium Quenching) measures 5 cm x 3.5 cm x 1 cm, the third sample is Steel C OQ (Optimum Quenching) measures 5 cm x 3 cm x 1 cm in length, width, and their thickness. Table.1 lists the chemical composition of the specimens, analyzed by a X – Ray Fluorescence (XRF).

2.2 Heat Treatment.
Heat treatment atmospheric conditions are vacuum heat treatments. At the stage of vacuum quenching using nitrogen gas and fan to accelerate cooling. To learn how each process parameter that is applied in influencing the hardness produced and corrosion resistance, the experiment is carried out in Table 2 and Table 3.

| Table 1. The compositions of medium carbon steel type SKD61 Steel |
|-----------------|---|---|---|---|---|---|---|---|---|
| Elements | Fe | C | Si | Mn | P | S | Cr | Mo | V |
| Weight % | 89.411 | 0.353 | 0.32 | 1.17 | 0.004 | 0.003 | 5.58 | 2.62 | 0.539 |

| Table 2. Heating Condition Stage Experimental Planning |
|---------------------------------------------|-----------------|-----------------|-----------------|-----------------|-----------|
| Heating Condition | Preheat 1 | Preheat 2 | Austinitizing |
| Temperature | Time | Temperature | Time | Temperature | Time |
| 650°C | 2 H | 850°C | 2H | 1030°C | 3H |

| Table 3. Cooling Condition Stage Experimental Planning |
|-------------------------------------|-----------------|-----------------|
| Pressure | Fan Speed | Time |
| Initial | 2 bar | 15 Hz | 20 minute |
| 2nd | 6 bar | 50 Hz | Steel A LQ 30 minute |
| | | | Steel B MQ 40 minute |
| | | | Steel C OQ 50 minute |
| 3rd | 4 bar | 35 Hz | 20 minute |
2.3 EDM
After the vacuum heat treatment process an EDM (Electrical Discharge Machining) process is performed. The samples that were carried out by the EDM process were Steel B MQ and Steel C OQ, whereas for the A LQ samples there was no EDM process. This process was carried out in this research because the EDM process is a machining process that is often used in making dies and molds. Electro spark on EDM produces high heat on the surface of the material so that it can potentially change the nature of the material on the surface.

2.4 Microhardness Test
The microhardness testing method used in this experiment is Vicker's Hardness Test. The load applied to this test is 200 gf. Hardness testing of micro vickers is carried out in order to determine the depth of hardening softening. As for testing the surface hardness using Rockwell Hardness with a load of 150 KPressure. Hardness testing of micro vickers is carried out in order to determine the depth of hardening softening.

2.5 Corrosion Test
Corrosion test using a potentiostat. Potentiostat uses 3 electrodes namely, working electrode, auxiliary electrode, and a reference electrode. The reference electrode used in this experiment is Ag / AgCl.[4] The reference electrode serves to keep the voltage applied to the electrode stable, therefore the reference electrode is only used as a conductor of voltage without current. The electrode is connected to a wire that has been glued to the sample, then the four types of sample are dipped in 1%, 3.5% and 7% NaCl solution. Data obtained from the potentiostat are electrochemical potential and currents. From these data, the corrosion rate can be calculated at material using a formula obtained from Faraday's Law in Determining the Corrosion Rate of Metals and Alloys. [7,8]

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

where

M = atomic weight (g.mol\(^{-1}\))

i = current density (A/cm\(^2\))

n = number of electron involved

\(\rho\) = density (g/cm\(^2\))

C = constant (0,129 in mpy; 3,27 in mm/year; 0,00327 in mm\(^3\)/year)

3. Results and Discussion

3.1. XRD Analysis
The results of x-ray diffraction patterns in the sample used in this study can be seen in Figure 1, and data collected from the characterization of this sample are attached in Table 4. From Figure 1 it appears that each sample tested, there are 3 peaks identified. For the three peaks of Steel A LQ has an angle position 43,98°, 64,47°, 81,71°. For the three peaks of Steel B MQ has an angle position 44,56°, 64,83°, 821,80°. For the three peaks of Steel C OQ has an angle position 44,1973°, 64,7334°, 81,82°. In the second peak and third peak, each peak has 2 different phases, namely, iron-alpha and ferchromide. This is because the two phases have diffraction reflections with almost the same d-spacing. The highest peak phase can also be obtained from the XRD test results, as shown in Table 4. Steel A LQ has the highest peak of the ferchromide phase 58,92 cps, Steel B QM 82,69 cps, Steel C OQ 80,44 cps.
Table 4. Crystallographic parameters of the Untreated Steel, Steel A QL No EDM, Steel B QM EDM, and Steel C QO EDM

| Sample          | First peak’s height (cps) | First peak’s d (Å) | Lattice Parameter (Å) | Volume (Å³) | Density (g/cm³) | Concentration (%) |
|-----------------|---------------------------|-------------------|-----------------------|-------------|----------------|------------------|
|                 | Iron-Alpha                | Ferchromide       | Iron-Alpha            | Ferchromide |                 | Ferchromide      |
|                 | 43.2                      | 58.92             | a = 2.8852            | a = 2.8683  | 23.0198        | 41.7             |
|                 | 59.21                     | 82.69             | b = 2.8852            | b = 2.8683  | 23.4876        | 43.7             |
|                 |                            | 80.44             | c = 2.8852            | c = 2.8683  | 23.5126        | 45.6             |
|                 |                            |                   | Iron-Alpha            | Ferchromide |                 | Ferchromide      |
|                 |                            |                   | a = 2.8683            | a = 2.8830  | 23.5988        | 58.3             |
|                 |                            |                   | b = 2.8683            | b = 2.8830  | 23.9641        | 56.3             |
|                 |                            |                   | c = 2.8683            | c = 2.8830  | 23.0522        | 54.4             |
|                 |                            |                   |                       |             |                |                  |

From Figure 1 the XRD plot results show that the first peak of each sample has a different FWHM. From the peak width can find out the relationship between FWHM and crystallite size. In Steel A LQ has FWHM 0.5995 °2Th and crystallite size 129.961700 Å. In Steel B MQ has FWHM 0.6246 °2Th and crystallite size 122.119800 Å. In Steel C QO has 0.6176 °2Th FWHM and crystallite size 124.914100 Å. From these data, it can be concluded that the wider the FWHM, the smaller the crystallite size. The smallest crystallite size is owned by Steel B MQ. The ferchromide crystal system is cubic, so the lattice parameter values a, b, and c are the same. However, because each sample has a different peak position, the lattice parameter value is different for each sample.

3.2. Microhardness Analysis

After the austenitization temperature, the sample quenched using nitrogen to accelerate cooling. The cooling rate used at the core is at least 3°C / minute and more than 5°C / minute is recommended. This is done with the aim of obtaining high toughness.[9] In this experiment, the cooling rate used was 8.5 °C / minute and used the step quenching process. The reason for using step quenching is to minimize the occurrence of induced cooling deformation and simplify controlling the cooling process in accordance with the CCT diagram to achieve the desired quality. In this process, the process is controlled by varying 2 parameters, gas pressure and fan speed.[10]

In the first step, the cooling process initially has a low gas pressure. In the second step, the gas pressure is increased significantly to prevent cooling from approaching the nasal passages of the CCT chart. This is done with the aim to avoid the formation of the pearlite phase and the bainite phase as much as possible. Then in the third stage, the gas pressure is lowered when it enters the martensite phase formation phase.[10]
Figure 1. XRD’s plot of the Steel A LQ, Steel B MQ and Steel C OQ.

From the results of vickers micro hardness test showed in Table 5 and Figure 2, the sample that has the highest hardness is Steel A LQ. This is because Steel A LQ does not accept the EDM process, so there is no decrease in hardness obtained from the vacuum heat treatment process. Then the sample that has the second highest hardness is Steel C OQ, but the core has decreased violence. This is because the time taken to cool the surface to the core is different. So that the core allows there are still residual phases of austenite, bainite, and pearlite in amounts greater than the martensite phase. The sample that has the third highest hardness is Steel B MQ. The results of the hardness obtained in Steel B MQ and Steel C OQ can be seen that there is a softening of hardness due to the EDM process of around 0.02 mm. Results of Rockwell hardness test showed in Table 6 and Figure 3. Whereas the highest hardness rockwell hardness testing is Steel A LQ, which is 43 HRC, followed by Steel B MQ 42.1 HRC, and Steel C OQ 41.5 HRC. From the data obtained reflecting the surface after EDM corrected an average decrease of 1 HRC.

| SKD61 Steel | Depth | Steel A LQ No EDM | Steel B MQ EDTM | Steel C OQ EDTM |
|-------------|-------|-------------------|-----------------|-----------------|
|             | 0.01  | 372.5             | 352.8           | 371.7           |
|             | 0.02  | 420.3             | 380.1           | 411.5           |
|             | 0.03  | 429.8             | 406.6           | 414.7           |
|             | 0.04  | 425.9             | 419.7           | 426.6           |
|             | core  | 431.7             | 426             | 423.6           |
3.3. Potentiodynamic Polarization Curves Analysis

Corrosion testing is carried out to produce a potentiodynamic polarization curve that describes the electrochemical behavior of SKD61 steel material. The electrochemical behavior of this sample can be seen in Figure 4 and the data obtained in Table 7. The process of corrosion can occur if there are 4 factors[11]:

- **Anode**
  At the anode, there is a process of oxygen binding and electron release which causes corrosion. Anodic reaction is:
  \[
  M^+ + Cl^- + H_2O \rightarrow MOH + H^+ + Cl^- \tag{2}
  \]

- **Cathode**
  At the cathode, there is a process of releasing oxygen and binding of electrons. Cathodic reaction is:
  \[
  2H_2O \rightarrow O_2 + 4H^+ + 4e^- \tag{3}
  \]

- **Metallic Pathway**
  A conductor that connects the anode and the cathode.

- **Electrolyte**
  A solution containing which can conduct electricity which is in this experiment uses a solution of NaCl electrolytes.
Table 6. Surface Hardness (Hardness Rockwell)

| Item                      | SKD61 Steel |
|---------------------------|-------------|
| Steel A LQ No EDM         | 43 HRC      |
| Steel B MQ EDM            | 42.1 HRC    |
| Steel C OQ EDM            | 41.5 HRC    |

Figure 3. Hardness Rockwell plot of the Steel A LQ No EDM, Steel B MQ EDM and Steel C OQ EDM

From the potentiodynamic polarization curve, the corrosion current density can be determined by extrapolating the Tafel line. From the data on Table 7 it can be seen that in general, the corrosion rate will increase when the concentration is higher. The highest corrosion rate at 7 wt% NaCl solution. NaCl which functions as a strong oxidizing agent has successfully oxidized Fe. The greater the concentration of NaCl, the more atoms released from iron, the faster the corrosion.[11] From the curve, it can be seen as the lower the current density, causing corrosion rates slower. It happens because the lower the current density then the more electrons increase.

Table 7. Data from the corrosion test of the Steel A LQ, Steel B MQ, and Steel C OQ in the 1 wt%, 3.5 wt% and 7 wt% NaCl solution

| Wt % NaCl | Sample     | Sample | R (ohm)  | $E_{corrosion}$ (V) | $I_{corrosion}$ (A) | Corrosion Rate (mm/year) |
|-----------|------------|--------|----------|---------------------|----------------------|--------------------------|
| 1 wt%     | Steel A LQ | 2.844 x 10^2 | -1.088   | 9.036 x 10^{-5}     |                       | 0.0011                   |
|           | Steel B MQ | 3.477 x 10^2 | -1.094   | 7.39 x 10^{-5}      |                       | 0.0009                   |
|           | Steel C OQ | 3.794 x 10^2 | -1.097   | 6.772 x 10^{-5}     |                       | 0.0008                   |
| 3.5 wt%   | Steel A LQ | 1.97 x 10^2  | -1.098   | 1.304 x 10^{-4}     |                       | 0.0016                   |
|           | Steel B MQ | 1.939 x 10^2 | -1.097   | 1.325 x 10^{-4}     |                       | 0.0016                   |
|           | Steel C OQ | 2.316 x 10^2 | -1.089   | 1.109 x 10^{-4}     |                       | 0.0014                   |
| 7 wt%     | Steel A LQ | 2.019 x 10^2 | -1.086   | 1.272 x 10^{-4}     |                       | 0.0016                   |
|           | Steel B MQ | 1.959 x 10^2 | -1.085   | 1.312 x 10^{-4}     |                       | 0.0016                   |
|           | Steel C OQ | 1.627 x 10^2 | -1.109   | 1.579 x 10^{-4}     |                       | 0.0020                   |
Different quenching speeds in each sample turned out to affect the corrosion rate. In general, the low corrosion rate is at the optimum quenching conditions and the highest corrosion rate is at the low quenching conditions. The speed of quenching will affect the volume of carbides produced in the martensite phase. Carbide volume can increase if the number of C atoms in the austenite phase decreases, which causes corrosion resistance to decrease. The low corrosion rate results in the entire sample show that vacuum heat treatment is very helpful to prevent corrosion. Because from the start of the heating process to cooling air conditions in a vacuum, thus preventing oxidation of the material because when heating at high temperatures allows oxidation quickly.

4. Conclusion

One that affects the hardness of SKD61 steel is the cooling method used and the cooling rate. Because these two things will determine the phase that is formed. So it's important to pay attention to these two things so that when the material goes through the EDM process, the reduction in hardness is not drastic.
SKD61 steel corrosion resistance is determined in the entire vacuum heat treatment process. Because the final phase formed and air condition will be the determining factor of how resistant the material is to corrosion. Another factor that determines the corrosion rate other than those mentioned above is the concentration of the electrolyte solution used. The higher the concentration of the solution used, the higher the corrosion rate of the material.

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