Analysis of Strength Improvement and Crystallographic Transformations In an Isothermal Oil Quenched on Martempering AISI 4140

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Abstract. This study aims to maintain the phase change process of 4140 steel at the starting point of martensite (Ms) by adjusting the speed and restraint of the transformation process to prevent the formation of the final martensite (Mf) phase. The use of oil with a constant temperature is able to stabilize the austenite phase into retained austenite (RA). The isothermal cooling (IQ) process uses a cooling chamber controlled by a microcontroller. The partitioning process is isothermal quenching deformation (IQDP) by heating the specimen at 850°C for 20 minutes which is rapidly cooled in oil quenching medium at constant temperature. Oil quenching media is used to hold the cooling rate so that the austenite phase transformation (FCC crystal) to martensite phase (BCT crystal) occurs. The effect of IQDP treatment on phase change, crystal orientation and mechanical properties were analyzed by hardness test, tensile test and SEM. The test results show that IQ treatment on SAE 40 oil is able to increase its tensile strength and modulus of elasticity. The application of isothermal quenching using SAE 40 oil with a constant temperature of 40°C has been proven to increase the ductility of raw materials from 1226.94 MPa to 1361.77 MPa.

Keywords. Isotermal, Oil Quenching, Tensile Strength, Scanning electron microscopy.

1. Introduction

Steel heat treatment aims to change the phase into a new crystalline form with better mechanical properties. The shift of metal atoms and crystals was affected by the temperature level and the cooling rate. The quenching process with oil was used to prevent a phase change that was too fast so that crystals with smaller grains are formed which will have an impact on increasing the ductility of the metal, [1]. The development of heat treatment by selecting the type of media with certain conditions continues to be carried out to achieve the desired mechanical and phase properties. The development of heat treatment by selecting the type of media, temperature treatment and process control continues to be carried out for the desired mechanical and phase properties.

The process detection method by photographing phase changes and atomic partitioning was carried out using the quenching deformation partitioning (QDP) method, [2]. The quenching deforming (QP) method was designed to detect every phase change due to treatment by adjusting the speed and time of phase transformation. The QP stage was carried out with a change limit referring to
the change of austenite into the phase that was the target of treatment. The use of oil with a constant
temperature was used to prevent the formation of coarse crystals of martensite and stabilize the austenite
phase change to pearlite and ferrite phases. The holding temperature stabilizes the shifted carbon
elements to find the position of the crystal space, and was not trapped in an imperfect arrangement in
the formed martensite phase. QP treatment was able to increase the tensile strength with an elongation
of 25% before reaching the fracture point. [3]. The QP process was a method used to improve the phase
balance to achieve high mechanical properties, especially ductility according to the function of the
material in special functions. [4]. Rapid cooling results in a less homogeneous final phase distribution,
uneven crystal dimensions and decreased ductility [5].

Medium carbon steel with carbon content between (0.4-0.6%), phase change refers to the Fe-
Fe3C diagram and the time-temperature diagram can be designed so that the temperature, type of cooling
medium and cooling speed are adjusted to produce phase/equilibrium with a uniform crystal shape.
Carbon content plays an important and effective role in the process of atomic bonding in the crystal
lattice. The shift of carbon that was able to fill the crystal lattice space by diffusing into a fine martensite
phase will affect the final hardness level of the material, [6].

The effect of temperature was very important to study to determine the best conditions for the
transformation of austenite to fine martensite, and homogeneous phase of pearlite by maintaining a
constant oil temperature. The type of quenching media with variations in viscosity and treatment will
produce a final phase that affects the mechanical properties, especially the ductile properties of AISI
4140 steel, with a special function as a waste sorting blade. Metal properties will be analyzed based on
the results of hardness, tensile and Scanning Electron Microscopy (SEM) tests. The crystal structure
was needed to determine the specific changes of the metal phase, the composition and volume of the
phase as well as its mechanical properties. The concept of isothermal cooling with an oil medium was
set at a temperature of 40 oC, with a viscosity index of 40 and 90 SAE. The temperature treatment above
room temperature provides sufficient time for the phase transformation process, so that a phase with
fine grains was formed. The temperature was kept constant, thus giving the holding time for the austenite
phase to gradually change to the fine martensite, ferrite and pearlite predominant phases with finer and
uniform crystal dimensions.

2. Material and Method
2.1. Materials
a. Materials : Steel AISI 4140, (0,4% C)
b. Quenching media : Machine oil 40 dan 90 SAE
c. Specimen : Standard JIS [JIS –Z- 2241]
d. Specimen dimention

Fig. 1. Specimen dimention JIS- Z- 2241, A Microstructural and Tensile test
Table 1. Composition test data AISI 4140

<table>
<thead>
<tr>
<th>Testing Materials</th>
<th>Content AISI 4140</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grade</td>
<td>AISI 4140</td>
</tr>
<tr>
<td>C%</td>
<td>0.413</td>
</tr>
<tr>
<td>Si%</td>
<td>0.342</td>
</tr>
<tr>
<td>Mn%</td>
<td>0.82</td>
</tr>
<tr>
<td>P%</td>
<td>18.01</td>
</tr>
<tr>
<td>S%</td>
<td>12</td>
</tr>
<tr>
<td>Cu%</td>
<td>11</td>
</tr>
<tr>
<td>Ni%</td>
<td>8</td>
</tr>
<tr>
<td>Cr%</td>
<td>1.07</td>
</tr>
<tr>
<td>Mo%</td>
<td>0.19</td>
</tr>
</tbody>
</table>

2.2. Isothermal-Quenching Room Temperature Control System

The oil medium was heated at 40 oC and held at a constant temperature using an electric heater with a thermocouple temperature sensor controlled using the Arduino UNO. The media temperature regulation system is equipped with processing time settings until the equilibrium point was reached, namely the specimen temperature was the same as the cooling medium. The isothermal quenching parts are shown in Fig 2.

Fig 2. The heat treatment process was carried out.

The stage of isothermal quenching/cooling with constant temperature shown in Fig 4, was a heat treatment process on specimens using oil media with controlled viscosity and temperature. The QDP technique was able to identify the microstructural phase of each stage. The quenching (isothermal) stage was one stage of the Quencing Deformations Partitioning (QDP) process, namely the heat treatment stage with phase detection for each stage of (1) heating process, (2) rapid immersion cooling with constant temperature (Isothermal Quenching), (3) which was followed by a reheating process (tempering) to form a martempering phase with an even distribution of carbon and ended with (4) air cooling (Annealing) at room temperature.

Table 2 Specimen standart test

<table>
<thead>
<tr>
<th>No</th>
<th>Specimen</th>
<th>Specification</th>
<th>Number of Specimens (Factor/Level)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Tensile test standard [JIS Z 2241]</td>
<td>Steel AISI 4140, Tensile test specimens and metallographic tests each QP stage</td>
<td>12 piecies tensile tes &amp; 6 piecies (SEM) test rincian : 3 piecies untreatment, 3 piecies tensile test by quenching untreated temperature (oil SAE 40). 3 piecies tensile test by quenching constant temperature treatment (oil SAE 40 temp. 40oC) 3 piecies tensile test by quenching untreated temperature (oil SAE 90)</td>
</tr>
</tbody>
</table>
Table 3. Factor and Level

<table>
<thead>
<tr>
<th>Nu</th>
<th>Factor</th>
<th>Level 1</th>
<th>Level 2</th>
<th>Level 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Quenching media</td>
<td>Oil viscosity SAE 40</td>
<td>Oil viscosity SAE 40°C</td>
<td>Oil viscosity SAE 90</td>
</tr>
</tbody>
</table>

2.3. Tensile Strength Test

Tensile strength test according to ASTM E8-15a Standard:[7] Method testing the tensile strength of AISI 4140 metal with the Untimate machine strength of Merck TN 20 MD made in France at the Mechanical Engineering Laboratory, Gajah Mada University, Yogyakarta Indonesia.

2.4. Rockweel Hardness Test

Hardness measurement based on the net increment of depth as a defined load. Hardness does not have a unit number and is given on a scale of R, L, M, E, and K. The material hardness test is carried out at the Mechanical Engineering Laboratory, Gajah Mada University, Yogyakarta Indonesia.

2.5. Observation Scanning electron Microscopy

Scanning electron microscope observations using the JSM-610 PLUS/LV model instrument from JEOL to be able to capture two-dimensional images of the metal surface as a result of the tensile test results and the treatment was carried out at Diponegoro University, Semarang, Indonesia. The AISI 4140 steel fracture was mounted on a piece of aluminum-coated platinum and observed for 1 minute at a pressure of 2 bar.

2.6. AISI 4140 . Tempering Temperature Determination

AISI 4140 steel, was a medium carbon steel, (C: 0.41%), to obtain ductile properties based on tempering treatment was enhanced by strain strengthening through the accumulation of dislocations. The QDP Isothermal Quenching diagram was shown in Fig 5.

![Fig. 3. Stages of Quenching Deformations Partitioning.](image)

The heating process was carried out at a temperature of 850°C, for 1200 seconds, followed by oil quenching with temperature control, according to the stages of heating, quenching, tempering and annealing. The phases of the phase change were detected with microstructure test specimens in each treatment. Changes in the orientation and size of the crystal grains and the chemical binding process of the elements will be tested by SEM test. The effect of quenching with constant temperature aims to improve the ductility of the material. Identification of changes in mechanical properties tested for hardness, tensile test and morphology test (SEM) to describe the crystal lattice, carbon diffusion process.
to determine the position in the structure. The oil temperature holding effect was expected to be able to keep the austenite phase limited to the martensite limit, in order to achieve an increase in ductile properties. The specific austenite phase changes were analyzed using crystal changes and the type of carbon bonds formed in each treatment.

3. RESULTS AND DISCUSSION
3.1. Tensile and Modulus elasticity

The results of the tensile test of the quenching treatment as shown in Fig 4.

Fig 4 shows that the average tensile strength of each variation of the quenching media is SAE 90 oil quenching media of 1226.94 MPa, SAE 40 oil media with a strength value of 1279.97 MPa. In the isothermal quenching treatment of SAE 40 oil at a temperature of 40ºC, 1361.77. In graphical form, it is shown in Figure 6. Of the three variations of the viscosity of the quenching media, the one with the highest tensile strength is the SAE 40 oil quenching medium at a temperature of 40ºC with an average value of 1361.77 Mpa. [8]. Quenching with 40 oC oil shows excellent ductility compared to others. The high ductility of quenched specimens may be associated with the inability of the specimens to form martensite. Ferrite and pearlite always exhibit poor ductility. [9]The average tensile strain value for each variation of the quenching media was 13.42% SAE 90 oil, 12.97% SAE 40 oil, and 12.01% SAE 40 oil temperature 40ºC. According to Fig.17.c, Based on the data above, it can be concluded that to produce high strain, appropriate heat treatment and holding time processes are required and in determining the cooling medium it was necessary to pay attention to the level of viscosity.

Elasticity modulus using the calculation of the modulus of elasticity with a variety of Quenching media. Based on Figure 12, the average Elasticity Modulus is shown according to the Quenching media used. SAE 90 Oil Quenching Media has an Elasticity Modulus of 91.73 Mpa, SAE 40 Oil Quenching 99.7 Mpa, and SAE 40 Oil Quenching Media at 40ºC 113.07 MPa. Analysis of tensile test data, analysis of strain and modulus of elasticity found that the quenching medium of SAE 40 oil at...
40°C had a higher value of the modulus of elasticity. Grafik elastisiti modulus sebanding dengan grafik tensile strength [10]. Based on the data above, it can be concluded that to produce high tensile strength, appropriate heat treatment and holding time processes are required and in determining the cooling medium it was necessary to pay attention to the level of viscosity.

3.2. Hardness Test Results

The results of the hardness test are as shown in table 4

<table>
<thead>
<tr>
<th>Nu</th>
<th>Spesimen</th>
<th>Hardness Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Raw Material</td>
<td>31</td>
</tr>
<tr>
<td>2</td>
<td>Quencing SAE 90</td>
<td>41.67</td>
</tr>
<tr>
<td>3</td>
<td>Quencing SAE 40</td>
<td>38.67</td>
</tr>
<tr>
<td>4</td>
<td>Quencing SAE 40 suhu 40°C</td>
<td>37.33</td>
</tr>
</tbody>
</table>

Based on the graph of the results of the hardness test using the Rockwell method, it shows that there was an increase in the hardness value between the raw material and the specimen that has been quenched. The raw material specimen shows a hardness number of 31 HRC. Quenching treatment with SAE 90 oil media showed an increase of 0.34% when compared to raw material, with an average hardness value of 41.67 HRC. Specimens with SAE 40 Oil quenching media experienced an increase of 0.24%, an average of 38.67 HRC. Specimens with SAE 40 oil quenching treatment at 40°C increased by 0.20%, the average was 37.33 HRC. The treatment and viscosity level affect the hardness value according to research results [11]

3.3. SEM Test Results

The results of the SEM test on AISI AISI 4140 raw material and morphology according to the heat treatment stages according to the QDP for each process are shown in Fig 6.

Figure 6. a. SEM raw materials, b. SEM being heated at 850°C.

In the next stage the morphology and crystal phase of the material/specimen after quenching and tempering was shown in Fig 7.

Fig 7.a. SEM quenching 40 SAE oil, b. Transformation tempering at 550°C.
The results of the APT analysis of the partitioning of elements between phases were shown in Fig. 9. Retained austenite in Fig. 9(a) is a film-like retained austenite with a thickness of about 25 nm situated within the martensite region. The pink color represents the carbon atom and the green represents the iso surface with the carbon atom in the 3.0 di fraction. %. Figure 96(b) shows the distribution of elements along the longitudinal axis of the ROI shown in Fig. 6(a). Figure 6(b) shows that the distribution of carbon across the retained austenite film was non-uniform and the carbon concentration is in the range of 6.075%. The carbon concentration in retained austenite was much higher than in martensite and a clear carbon concentration gradient was up to 1.1 in. %/nm exists across the phase interface [12].

From the observation of the microstructure, it can be seen that the specimen with Quenching oil sae 40 without treatment contained martensite and pearlite phases, the specimen with quenching oil sae 40 at 40ºC contained ferrite and pearlite phases, and the specimen with quenching oil sae 90 contained martensite and pearlite phases. The application of isothermal quenching using SAE 40 oil with a constant temperature of 40ºC has been proven to increase the ductility of raw materials from 1226.94 MPa to 1361.77 MPa. The highest level of ductility compared to the use of SAE 90 and SAE 40 oils without temperature treatment, this indicates that the IQ method improves ductility properties the best compared to other oil media (SAE 90 and 40 without temperature treatment).

4. Conclusion

Specimen testing using oil with a viscosity (40 and 90 SAE) with an isothermal constant temperature setting is aimed at getting a slightly decreased hardness but high ductile properties, from the experimental data and the tests carried out it can be concluded as follows:

Microstructure observations can be seen that the specimens with SAE 40 oil quenched without treatment have martensite and Pearlite phases, Specimens with SAE 40 oil quenching at 40ºC have ferrite and pearlite phases, and Specimens with SAE 90 oil quenching have martensite and pearlite phases. The application of isothermal quenching using SAE 40 oil with a constant temperature of 40ºC has been proven to increase the raw material ductility from 1226.94 MPa to 1361.77 MPa. The highest level of ductility compared to the use of SAE 90 and SAE 40 oils without temperature treatment, this shows that the IQ method increases the ductility properties the best compared to other oil media (SAE 90 and 40 without temperature treatment).

Media Quenching Oil SAE 90 can be applied to surface hardening, the highest average hardness value is 41.67 HRC, this shows that for surface hardening while still having ductility SAE 90 is used; Crystal morphology based on the iq process, the SEM test showed the best transformation using SAE 40 oil with a constant temperature of 40 oC, the impact of using IQ media had an effect on the carbon bonding process in the crystal lattice.

Referensi


