Intercritical Heat Treatment Temperature Dependence of Mechanical Properties and Corrosion Resistance of Dual Phase Steel

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This study investigated the effect of intercritical heat treatment temperature on the tensile properties, work hardening and corrosion resistance of dual phase steel. Ferrite-martensite dual phase steel with different martensite volume fractions were obtained after heat treatment at different intercritical temperatures. Microstructure, mechanical properties of steel were measured and the corrosion resistance was evaluated via polarization test. Tensile strength of the specimens increased by increasing the martensite volume fraction up to 48.2%. Further increase in martensite volume fraction led to decrease in tensile strength. Work hardening behavior analyzing showed that in DP steel with less than 50% martensite volume fraction, the work hardening took place in one stage and by increasing the martensite volume fraction two-stage work hardening behavior was observed in the Holloman analysis. The results of polarization test showed that, the corrosion resistance of dual phase steel is higher than that of plain carbon steel with ferrite-pearlite microstructure.

Keywords: Dual phase steel, Intercritical heat treatment, Work hardening, Corrosion resistance.

1. Introduction

Dual phase (DP) steel is identified as an important class of high strength low alloy steels (HSLA). These steels have unique properties such as high tensile strength, high elongation, high strength to weight ratio, continuous yielding behavior and favorable ultimate tensile strength (UTS) to yield stress ratio. These properties are related to microstructure of dual phase steels in which soft and ductile ferrite matrix ensures high formability; while, hard martensite phase provides strengthening effects. The use of advanced high strength steels (AHSS), due to the optimal combination of these properties, in the automotive industries has increased, dramatically. The unique combination of mechanical properties, study of corrosion behavior of dual phase steels, to explore the true potential of these steels used in the automotive industries, seems to be necessary. The easiest way to achieve special structure of dual phase steels is to perform heat treatment on low alloy steels (carbon percentages less than 0.2%) in temperature range between AC1 and AC3 and then quenching in cool environment. As a result, ferrite-pearlite microstructure is replaced by ferrite-martensite microstructure. Since last decade, mechanical properties and corrosion behavior of dual phase steels and their relation to microstructure have been considered by many researchers.

Movahed et al. has observed that dual phase steels with approximately equal amounts of ferrite and martensite phases exhibit the optimum mechanical properties in terms of tensile strength, ductility and fracture energy. Yang et al. studied the effect of martensite strength on the tensile strength of dual phase steels. They observed that this variation is ascending and not linear. Bag et al. studied a series of dual phase steels with different volume fraction of martensite (varied from 0.3 to 0.8) by changing the intercritical annealing temperature. They showed that DP steels with finely dispersed microstructures and 55% martensite volume fraction had excellent mechanical properties. It is shown by Davies that the strength of dual phase steels is dependent on the ferrite grain size and the volume fraction of martensite and is independent of the composition and strength of the martensite. Bhagavathi et al. studied the mechanical properties and corrosion behavior of dual-phase steels and found that by increasing the martensite volume fraction (MFV), the UTS of dual-phase steels increased and corrosion rate of DP steel samples marginally decreased compared to that for the low alloy steel sample. Kelestemur et al. showed that the corrosion rate of dual phase steel has increased with increasing the amount of martensite. Sarkar et al. studied the electrochemical behavior of microalloyed dual phase steels and found that an increase in martensite content and structural refinement, decreases the corrosion resistance.

In summary, there is little work on corrosion behavior and mechanical properties of dual phase steels and due to controversies in this field, further investigation is required to explore the effect of martensite volume fraction on these properties. In the present study, the SAE 1015 sheet steel was intercritically heat-treated and the effect of martensite...
volume fraction on mechanical properties and corrosion behavior of the produced DP steel was examined.

2. Experimental Procedure

2.1. Material and heat treatment

The steel used in the present study was 1.5 mm thick SAE 1015 ferrite-pearlite steel sheet. The chemical composition of this steel, determined using quantometric analysis technique, is shown in Table 1. Quantometric analysis was done by using OSE 1000 Skyray Instrument. For the tensile testing, plane carbon steels were cut from the sheet according to the ASTM E8M.

For the present investigation, the lower and upper critical temperatures (AC$_1$ and AC$_3$) were estimated as 742°C and 858°C, respectively, by using Eqs. (1) and (2)$^{12}$:

\begin{align}
AC_1(°C) &= 751 - 16.3C - 27.5Mn - 5.5Cu - \\
&\quad 5.9Ni + 34.9Si + 12.7 + 3.4Mo
\end{align}

\begin{align}
AC_3(°C) &= 881 - 20.6C - 15Mn - 26.5Cu - \\
&\quad 20.1Ni + 53.1Si + 12.7Cr + 41.7V
\end{align}

All the samples were heated in intercritical temperature range (between AC$_1$ and AC$_3$) and were held for 20 min in a muffle furnace followed by water quenching to produce dual phase microstructure. The heat treatment procedure which is used in this study is shown in Fig. 1. The heat treated samples are designated by capital letter, as shown in this figure.

2.2. Microstructure and mechanical properties

4 specimens of each samples were prepared to investigate the microstructure and mechanical properties of as-received and DP steel. Microstructures of the as-received and heat treated samples were observed under a light microscope following the standard metallographic polishing and etching with 2% nital solution. The volume fraction of the martensite was measured by point count technique according to ASTM E 562. Vickers hardness of the specimens was measured using 5 kgf load with a loading duration of 10s in all cases. Tensile testing was performed using an Instron 8502 machine with a cross head speed of 1mm/min at room temperature in accordance to ASTM E8M.

2.3. Corrosion test

Galvanostatic polarization technique (EG&G 237A instrument) was used to investigate the corrosion resistance of the samples in 3.5% NaCl solution at 25°C using a scan rate of 1×10$^{-3}$ V s$^{-1}$. Before performing the corrosion test, 3 specimens of each samples were prepared and one of the surfaces of each sample was polished up to 4/0 grade emery paper and then cleaned by acetone. The other surfaces of the samples were coated with an insulating lacquer. The polished surface played the role of the working electrode when immersed in the solution of the electrochemical cell comprising the platinum as counter electrode and Ag/AgCl as reference electrode. After the corrosion testing, the specimens were observed under XL300 SEM for surface degradation by corrosion.

3. Results and Discussion

Microstructures of the heat-treated samples are shown in Fig. 2. This figure shows the optical micrographs of A, B, C and D steels, respectively. From the micrographs shown in Fig. 2, all microstructures consisting of proeutectoid ferrite phase (light matrix) and martensite (dark phase). Fig. 3 shows the variation of martensite volume fraction (MVF) with intercritical temperature. This figure shows that, due to increase of the austenite volume fraction with increasing the intercritical temperature, the amount of martensite increases up to 74% after quenching in water.

As it is shown in Fig. 4, the hardness of heat-treated samples is influenced by variation in the intercritical temperature. It should be noted that the hardness of the as-received sample is 120HV. By increasing temperature from 770°C to 840°C, the hardness of the specimens increases from 185HV to 427 HV. It can be seen that there is an increase in hardness of DP steels compared to as-received steel which is due to change in microstructure from ferrite-pearlite to ferrite-martensite.

Table 1. Chemical composition of the investigated steel (wt%).

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>S</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.1</td>
<td>0.44</td>
<td>0.13</td>
<td>0.04</td>
<td>0.08</td>
<td>0.02</td>
<td>0.14</td>
</tr>
</tbody>
</table>

Figure 1. Schematic illustration of heat treatment cycle used in this investigation.
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Variation of martensite volume fraction and hardness of specimens with temperature are summarized in Table 2.

3.1. Tensile properties of DP steels

Fig. 5a shows that the stress-strain curve of the as-received sample has yield-point elongation and exhibits well-defined yield point while it is absent in the stress-strain curves of dual phase steels in Fig. 5b. The presence of yield point phenomenon in as-received sample is related to the formation of Cottrell atmospheres. While in DP steels, presence of martensite in the matrix of ferrite eliminates the yield point phenomenon in their stress-strain curve. Formation of martensite from austenite, during water quenching, is associated with approximately 4% increase in

**Figure 2.** Optical micrographs of the heat-treated samples at various intercritical temperatures. showing light ferrite areas and dark pearlite/martensite areas.

**Figure 3.** The variation of martensite volume fraction (MVF) as a function of intercritical temperature.

**Figure 4.** The variation of hardness as a function of intercritical temperature.
volume. This leads to plastic deformation in the ferrite matrix and increases the density of dislocations. The high density of dislocations prevents the formation of Cottrell atmospheres, which are responsible for yield point phenomenon. The absence of yield point phenomenon in stress-strain curves of heat treated samples confirms that a dual phase structure is successfully developed.

Variation of UTS and %Elongation of the DP steels with martensite volume fraction has been characterized as shown in Table 3 and Fig. 6a. The strength values of DP steels are higher than that of the as-received steel. Presence of martensite as a harder second phase in dual phase steels led to increase in the strength compared to as-received sample. As can be seen in Fig. 6a, there is no proportional relationship between martensite volume fraction and ultimate tensile strength of the DP steels. By increasing the martensite volume fraction from 17% to 48.2%, the UTS of DP steels increased from 643 to 1071 MPa. However, further increase in martensite volume fraction did not significantly affect UTS of DP steels. Indeed, in addition to martensite volume fraction, the hardness of the martensite phase also plays an important role in deformation behavior of the DP steels. The hardness of the martensite phase is mainly controlled by its carbon content. This parameter can be calculated by using the rule of mixtures (Eq. (3)):

\[ C_0 = C_r V_f + C_m V_m \]  

Where \( C_0 \) is the steel mean carbon content, \( C_r \) is the carbon content of ferrite and \( C_m \) is the carbon content of martensite. \( V_f \) and \( V_m \) are also the ferrite and martensite volume fractions, respectively. In this equation the carbon content is assumed to be 0.015. It is observed from Fig. 6b that as a consequence of the increase of the martensite volume fraction, the amount of carbon content of martensite decreases. Therefore, the breakdown of a proportional relationship between martensite volume fraction and tensile strength of DP steels at high martensite volume fraction can be explained by considering the combined effect of martensite volume fraction and martensite hardness. Firstly, increasing the martensite volume fraction led to increase the tensile strength of DP steels due to increasing volume fraction of harder phase. Secondly, by increasing volume fraction of martensite, the carbon content of martensite phase decreases which in turn decreases the strength of martensite.

Fig. 6a shows the correlation between martensite volume fraction and uniform elongation of the DP steels. It should be noted that the uniform elongation of the base material is 32%. In sample A, where there is 17% martensite volume fraction, the uniform elongation decreased to 16%. The reduction in ductility of the DP steels compared to as-received ferrite-pearlite steel is related to the presence of hard martensite phase. Increasing in martensite volume fraction from 17% to 23% has led to further decrease in uniform elongation from 16% to 8%. The presence of higher martensite volume fraction induces higher restriction on the plastic deformation of the soft ferritic matrix.

It is expected that increasing martensite volume fraction monotonically decreases the ductility of the DP steels. However, as can be seen in Fig. 6a, increasing martensite volume fraction of the heat-treated samples from 23 to 74% did not affect uniform elongation, significantly. This can be explained by reduction of carbon content of the martensite by increasing martensite volume fraction. On one hand, increasing martensite volume fraction increases the volume.

Table 2. Martensite volume fraction and hardness of specimens with temperature.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Martensite Volume Fraction</th>
<th>Hardness(HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A (770˚C)</td>
<td>17.35</td>
<td>185</td>
</tr>
<tr>
<td>B (800˚C)</td>
<td>23.3</td>
<td>212</td>
</tr>
<tr>
<td>C (820˚C)</td>
<td>48.2</td>
<td>382</td>
</tr>
<tr>
<td>D (840˚C)</td>
<td>74.4</td>
<td>427</td>
</tr>
</tbody>
</table>

Table 3. UTS and %Elongation of the DP steels.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>UTS (MPa)</th>
<th>%Elongation</th>
</tr>
</thead>
<tbody>
<tr>
<td>A (770˚C)</td>
<td>643.6</td>
<td>15.57</td>
</tr>
<tr>
<td>B (800˚C)</td>
<td>829.5</td>
<td>8.93</td>
</tr>
<tr>
<td>C (820˚C)</td>
<td>1071.8</td>
<td>9.33</td>
</tr>
<tr>
<td>D (840˚C)</td>
<td>1033.5</td>
<td>10.26</td>
</tr>
</tbody>
</table>

Figure 5. Engineering stress-strain curves of (a) as-received and (b) heat-treated samples (a) A, (b) B, (c) C, (d) D.
fraction of the hard phase in the steel and on the other hand, increasing martensite volume fraction decreases the carbon content of the martensite phase which in turn enhances the ductility of the martensite (i.e. the effect of higher martensite volume fraction on the ductility is overridden by the softness of the martensite at higher martensite volume fractions)\(^{22,23}\).

### 3.2. Work hardening behavior of DP steels

The flow behavior of the most metals described by the following Eq. (4). This equation is named as Hollomon equation, where \(K\) and \(n\) are constant and normally called as the strength coefficient and strain hardening exponent, respectively \(^{24}\).

\[
\sigma = Ke^n
\]  

Work hardening of the metals can be good indicated by the stain hardening exponent (n). The higher value of \(n\) shows the higher rate of materials work hardening. The material with a high value of \(n\) is preferred for processes because of the more plastic deformation before necking starts. For this purpose, stress-strain curve in logarithmic scale should be drawn and fitting a line to these data. the slope of this line indicates the value of stain hardening exponent (n). \(^{25}\).

Fig. 7 shows the lnσ-lnε diagrams for samples A, B, C and D. As it can be seen in this figure the variation of lnσ-lnε diagrams for DP steels with martensite volume fraction less than 50% is linear. In fact, dual phase steels with \(V_{\text{m}}\) <50% have one stage work hardening behavior while the dual phase steel with 74.4% martensite volume fraction has two stage work hardening mechanism which is due to the activation of different work hardening mechanism (Table 4). The first stage can be related to ferrite plastic deformation, while both ferrite and martensite plastic deformation can lead to second stage \(^{27}\).

Unlike Cribb et al.\(^{26}\) the value of stain hardening exponent (n) increases by increasing the volume fraction of martensite. As the maximum plastic deformation is equal to work hardening power, by increasing the value of stain hardening exponent, the plastic deformation increased. So we can consider \(n\) as a measure of ductility. Therefore, it is expected that by increasing the amount of martensite volume fraction, the amount of ductility and thus stain hardening exponent decrease and it is true in the case of samples A and B. But this is not true in the case of samples C and D dual phase steels examined in this research. Indeed, the trend of the variation of ‘\(n\)’ is similar to the trend of the variation of uniform elongation with martensite volume fraction. The reason for this can be due to the difference in the strength of ferrite and martensite by increasing the martensite volume fraction \(^{1}\). Therefore, we can conclude that sample C with 48.2% martensite volume fraction has optimum mechanical properties in terms of tensile strength and ductility.

### 3.3. Corrosion behavior of DP steels

It should be noted that when the low carbon steels corrode in neutral 3.5% NaCl solution, the following reactions occur on the steel surface \(^{6}\):

**Anodic reaction:** \(\text{Fe} \rightarrow \text{Fe}^{2+} + 2e\)

**Cathodic reaction:** \(\text{O}_2 + 2\text{H}_2\text{O} + 4e \rightarrow 4\text{OH}^-\)

Fig. 8 indicates the potentiodynamic polarization behavior for as-received and DP samples in 3.5% NaCl solution. The results of polarization studies shown in Table 5 are derived from the experimentally obtained cathodic and anodic polarization (E vs. log i) curves after using Tafel’s linear extrapolation method. As it can be seen in Table 5, corrosion current density (\(i_{\text{corr}}\)) of all the DP steels is lower than that for as-received steel. As a consequence, to change in microstructure from ferrite-pearlite to ferrite-martensite, the corrosion current density (\(i_{\text{corr}}\)) and therefore, the corrosion rate (CR) decrease. In DP steels the galvanic couple is formed between ferrite and martensite. The structure of
Figure 7. lnσ-lnε curves for investigated steel with different martensite volume fraction.

![Graphs showing lnσ-lnε curves for different martensite volume fractions.](image)

Table 4. Work hardening exponent for different stages for heat-treated samples.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Vm%</th>
<th>Stage 1</th>
<th>Stage 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>17.35</td>
<td>0.22</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>23.3</td>
<td>0.354</td>
<td>-</td>
</tr>
<tr>
<td>C</td>
<td>48.2</td>
<td>0.367</td>
<td>-</td>
</tr>
<tr>
<td>D</td>
<td>74.4</td>
<td>0.391</td>
<td>0.164</td>
</tr>
</tbody>
</table>

martensite is BCT and the structure of ferrite is BCC. Therefore, martensite phase is homogeneous with respect to composition and also structurally is closer to the matrix ferrite phase. But in as-received steel with ferrite-pearlite structure, pearlite is a mixture of ferrite and cementite lamellae, in which cementite has 6.67% carbon and an orthorhombic structure. It is relatively more inhomogeneous compositionally and structurally. In addition to number of galvanic cells across ferrite and pearlite boundaries, a large number of microgalvanic cells will be set up between lamellae of pearlite. Therefore, the galvanic couple between ferrite and martensite is weaker than that between ferrite and pearlite. After potentiodynamic polarization testing, the as-received and heat treated samples were observed under SEM to study the corrosion products. As it is shown in Fig. 9, and based on the above reason, the corroded surface of the as-received sample is larger than that for DP steels. Sarkar et al. attributed the increase in the corrosion rate of dual phase steel as compared to ferrite-pearlite steel. This result is different from what is observed in the present study which can be explained as follows. Firstly, the chemical composition of steel used in this study is quite different. Secondly, island-like morphology of martensite, observed in this study, shows better corrosion resistance as compared to the network of martensite surrounding the ferrite grains in Sarkar et al. study. Thus, lower corrosion rate is observed for dual phase steels as compared to as-received steel. So, dual phase steels obtained by heat treating of the low carbon steel had greatly improved mechanical properties, without compromising the corrosion resistance.

Furthermore, it can be seen in Fig. 9 that the pits in sample A are smaller in size and fewer in number compared to other heat-treated samples. Fig. 10 shows the effect of martensite volume fraction on $i_{corr}$ and corrosion rate of DP steels. Due to relative change in the amount of the phase constituents, a change occurs in the ratio of cathode to anode areas. As indicated by increasing the volume fraction of martensite...
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(cathode), unfavorable area between cathode and anode increases and this leading to the higher corrosion rate of dual phase steels with higher martensite volume fraction (MFV) compared to the ones with lower volume fraction of martensite. As a result, by increasing the amount of martensite volume fraction the severity of pitting is the highest on the surface of D sample with respect to both frequency and depth of pits.

4. Conclusions

In this works, ferrite-martensite dual phase steels were produced by intercritical heat treatment at various intercritical temperatures. The microstructure, tensile properties and corrosion resistance of the produced steels were investigated. The following conclusions can be drawn from this study:

- There is no proportional relationship between martensite volume fraction and ultimate tensile strength of the DP steels. By increasing the martensite
Table 5. The results obtained from corrosion tests performed in 3.5% NaCl solution.

<table>
<thead>
<tr>
<th>Samples Name</th>
<th>Martensite Volume fraction</th>
<th>i_corr (µA/cm²)</th>
<th>Corrosion Rate (mm/year)</th>
<th>E_corr (mV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>16.8</td>
<td>13.74</td>
<td>0.159</td>
<td>-0.441</td>
</tr>
<tr>
<td>A (770°C)</td>
<td>17.35</td>
<td>4.403</td>
<td>0.051</td>
<td>-0.353</td>
</tr>
<tr>
<td>B (800°C)</td>
<td>23.3</td>
<td>5.381</td>
<td>0.062</td>
<td>-0.357</td>
</tr>
<tr>
<td>C (820°C)</td>
<td>48.2</td>
<td>5.381</td>
<td>0.117</td>
<td>-0.359</td>
</tr>
<tr>
<td>D (840°C)</td>
<td>74.4</td>
<td>11.22</td>
<td>0.130</td>
<td>-0.377</td>
</tr>
</tbody>
</table>
volume fraction from 17 to 48.2, the UTS of DP steels increased from 643 to 1071 MPa. However, further increase in martensite volume fraction did not significantly affect the UTS of the DP steel.

- Uniform elongation values dropped from about 16% for sample A to about 8% for sample B by increasing the martensite volume fraction. This drop is attributed to the increase in martensite volume fraction as the harder and less ductile phase. In addition, further increase in martensite volume fraction of the steels from 23 to 74 did not affect the uniform elongation, significantly. This can be explained by reduction of carbon content of the martensite by increasing martensite volume fraction.

- The work hardening behavior of the heat treated samples with $V_m<50\%$ is single stage. By increasing the martensite volume fraction, the work hardening capacity also increases. While for the heat treated sample containing more than 50% martensite, two stage work hardening is observed.

- The corrosion rates obtained from potentiodynamic polarization tests showed that the corrosion rate of dual phase steels is lower than that of as-received steel. This is because of the weaker galvanic couple between ferrite and martensite compared to that between ferrite and pearlite.

- By increasing the volume fraction of martensite (cathode), unfavorable area ratio between cathode and anode increases leading to the higher corrosion rate of dual phase steels with higher volume fraction of martensite compared to the ones with lower volume fraction of martensite.

5. References


