Microstructure and Mechanical Properties of a Microalloyed Steel After Thermal Treatments

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The properties of a microalloyed steel, with Nb and V in its composition, were studied, after different intercritical thermal treatments and at different austenitizing and tempering temperatures. The mechanical properties of the specimens were measured in a Vickers hardness tester, and their microstructures were analyzed by optical microscopy, with the aid of a digital image processor. After austenitizing at 1100 °C and tempering at 625 °C, the samples showed significantly higher tempering resistance, reflected by their retention of high hardness, which may be associated with a secondary hardening precipitation of Nb carbon nitrides. In the sample with dual-phase microstructure, the martensite volume fraction varied from 18.2 to 26.3% and the ferrite grain size remained unchanged, upon the variation of the time length of the intercritical treatments. Tempered samples showed Vickers hardness (HVN) varying from 327 to 399, and dual-phase samples showed HVN from 362 to 429.

Keywords: thermal treatment, dual-phase steel, tempered martensite

1. Introduction

The high strength and high toughness of tempered steels promote their widespread use in machine structural parts. Dual-phase steels, presenting a microstructure composed of martensite and/or bainite in a ferrite matrix, are basically employed by the automobile industry.

Commercial dual-phase steels present a volume fraction of martensite ranging from 10 to 30%, yield strength between 380 and 550 MPa and ultimate tensile strength from 620 and 850 MPa. The mechanical properties of dual-phase steels are function of the composite morphology, the volume fraction of martensite, the carbon content and the alloying elements.

Direct-quench through fast cooling after controlled or hot rolling is employed to produce tempered steel plates. Mintz, Foley and Weiss compared the mechanical properties and the microstructures of direct-quenched and conventionally tempered steels, to determine the influences of V, B and Ti and of the process variables.

This paper presents the effect of the austenitizing and tempering temperatures, as well as the effect of the intercritical thermal treatment parameters, on the microstructure and mechanical properties of a microalloyed steel containing Nb and V. The influence of the austenitizing temperature over tempering resistance was also evaluated.

2. Experimental

The steel composition (wt%) is given in Table 1. The samples were provided by the industry after hot rolling.

The austenitizing temperatures Ac1 and Ac3, which define the ferrite+austenite region, were calculated by Andrew’s empirical equations as 719.5 °C and 838.7 °C, respectively. The intercritical thermal treatments were performed at 750 °C for 5, 10 and 30 min, followed by quenching in a ice-cold water mixture. The tempered samples were austenitized at 900, 1000 and 1100 °C, for 15 min, quenched in ice-cold water, and tempered at 525 and 625 °C for 1 h, according to ASTM A514 procedure.

The microstructures were analyzed by optical microscopy, after etching with Nital 2%. Volume fractions...
and grain sizes were determined with the aid of a digital image processor program, hooked to the microscope. The Vickers hardness tests were performed with a load of 50 N, 20 measurements per sample.

3. Results and Discussion

3.1 Microstructure

Figure 1 shows the influence of the periods of time of the intercritical thermal treatments on the martensite volume fraction. The martensite volume fraction varies from 18.2 to 26.3%, between 300 and 1800 s of treatment.

Figure 2 shows the micrographs of samples submitted to the normalizing and intercritical thermal treatments. Figure 2a shows that the microstructure of the normalized sample consists of ferrite and pearlite. The hardness of this sample was measured as HV = 205 ± 1, for a pearlite volume fraction of 24.4%, and ferrite average grain size of 6.9 µm. The effect of the intercritical treatments on the microstructure is shown in Fig. 2b-2d. Micrographs 2b-2d reveal martensite thin layers thickening on the ferrite grain boundaries with time, until it forms a continuous network around the ferrite grains. This is a result of the increase in the martensite volume fraction with intercritical treatment time. No variation of the ferrite grain size was observed, and its average value stabilized at about 4.5 µm.

Figure 3 shows martensite structures in samples austenitized at different temperatures prior to quenching. The structure shows clusters of needles typical of low carbon martensite. Figure 4 shows hardness and austenite grain size as functions of the austenitizing temperature.

The martensite hardness is usually expected to decrease as the austenite grain size increases. However, Fig. 4 shows the opposite tendency, i.e., as the austenitizing temperature is raised, both the austenite grain size and the martensite hardness increase. This is probably due to the formation of small amounts of bainite during quenching of the samples austenitized at 900 and 1000 °C. Therefore, the microstructures in Figs. 3a and 3b consist of martensite and bainite, and the microstructure in Fig. 3c is mainly martensite.

3.2 Mechanical Properties

Figures 5 and 6 show the evolution of the Vickers hardness (HVN) with the time of intercritical treatment and with the martensite volume fraction, respectively. An increase in the intercritical treatment time leads to increase in the martensite volume fraction, which increases the steel hardness.

The values for the ultimate tensile strengths (TS) shown in Fig. 6 where calculated by the empirical Eq. 1:

\[ \text{TS (MPa)} = -32.0 + 3.0 \times (\text{HVN}) \]

Figure 7 shows the hardness of samples austenitized at different temperatures as a function of the tempering temperature. One can observe that the hardness after tempering, increases with the austenitizing temperature and decreases with the tempering temperature, exception made for the sample austenitized at 1100 °C, which showed an increase in hardness when the tempering temperature was raised.

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

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Al</th>
<th>P</th>
<th>S</th>
<th>Nb</th>
<th>V</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.15</td>
<td>1.39</td>
<td>0.39</td>
<td>0.039</td>
<td>0.016</td>
<td>0.009</td>
<td>0.046</td>
<td>0.046</td>
<td>0.0042</td>
</tr>
</tbody>
</table>

### Table 2: Amount of Nb dissolved in the austenite as a function of temperature.

<table>
<thead>
<tr>
<th>T (°C)</th>
<th>Nb (C, N) (wt%)</th>
<th>% Nb dissolved</th>
</tr>
</thead>
<tbody>
<tr>
<td>900</td>
<td>0.0020</td>
<td>4.3</td>
</tr>
<tr>
<td>1000</td>
<td>0.0057</td>
<td>12.4</td>
</tr>
<tr>
<td>1100</td>
<td>0.0139</td>
<td>30.2</td>
</tr>
<tr>
<td>1262</td>
<td>0.046</td>
<td>100</td>
</tr>
</tbody>
</table>
Figure 2. Optical micrographs (1000 ×) of samples after (a) normalization; intercritical at 750 °C for (b) 300 s; (c) 600 s, (d) 1800 s.

Figure 3. Optical micrographs (1000 ×) of samples austenitized at different temperatures prior to quenching: (a) 900 °C; (b) 1000 °C; (c) 1100 °C.
This behavior is related to the dissolution of Nb in the austenite. Table 2 shows the increase in the percentage of Nb dissolved in the austenite with temperature. The niobium atoms dissolved during austenitizing are retained in the martensite, remaining available to produce precipitation hardening during tempering. Therefore, the higher the austenitizing temperature, the higher the retained Nb content in the martensite, leading to a more pronounced precipitation hardening effect. Tempering at higher temperatures usually leads to lower HVN values, however, the samples austenitized at 1100 °C had such a large amount of Nb retained in the martensite, that the treatment at 625 °C for 1 h probably caused the precipitation of niobium carbon nitrides, leading to further hardening. The precipitation of niobium carbon nitrides is reported to occur at temperatures between 525 and 625 °C\textsuperscript{12}, and it should not affect the sample tempered at 525 °C.

4. Conclusion

The increase in the time of intercritical treatment at 750 °C showed little effect on the ferrite grain size, but it raises the martensite volume fraction and, as a result, the

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**Figure 4.** Vickers hardness and austenite grain size as functions of the austenitizing temperature. Standard deviations are below 10%.

**Figure 5.** Evolution of hardness with time of intercritical treatment.

**Figure 6.** Evolution of hardness and ultimate tensile strength with the martensite volume fraction.

**Figure 7.** Hardness of samples austenitized at different temperatures as a function of the tempering temperature.
hardness of the steel.

Lower austenitizing temperatures decrease the austenite grain size, but increase the presence of bainite, leading to lower HVN values of the dual phase steels.

Higher austenitizing temperatures increase the hardness of tempered samples, due to the higher dissolution of Nb in the martensite matrix, which precipitates during tempering. Samples austenitized at 1100 °C and tempered at 625 °C may precipitate niobium carbon nitrides, leading to further hardening.

References