Effect of tempering temperature on isothermal decomposition product formed below \( M_s \)

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ABSTRACT

Recent papers have shown that bainitic ferrite plates can be produced by transformation at low temperatures, resulting in enhanced mechanical properties of martensitic steels and increased resistance to tempering. To that end, experimental investigations were performed on the formation of the microstructure in SAE 9254 steel during isothermal heat-treatment below the martensite start \( (M_s) \) temperature. After the isothermal heat-treatment, the same steel was subjected to tempering at various temperatures. Vickers microhardness measurements were performed on the isothermally heat-treated and tempered samples, and microhardness values for the samples were compared with those for quenched and tempered samples of the same steel. All the isothermally treated samples contained the martensite \( (\alpha) \), bainitic ferrite \( (\alpha_b) \), and retained austenite \( (\gamma_r) \) phases. The retained austenite blocks \( (\gamma_r) \) decomposed in the samples tempered at 400 °C. A significant decrease in the microhardness value for the sample heat-treated at 270 °C can only be observed after the sample was tempered at 450 °C, because bainite was the predominant phase in the microstructure, which self-tempers as it forms.

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1. Introduction

The development of high-strength steels usually involves a balance between strength and toughness. It was previously found that the bainitic microstructure reaches an ultimate tensile strength 2.3 GPa and a toughness of approximately 30 MPa m\(^{1/2}\). This condition can be obtained in high-carbon, silicon-rich steels by transformation at low temperatures [1–3]. The bainitic microstructure in high-silicon steel consists of bainitic ferrite \( (\alpha_b) \) sheaves separated by carbon-enriched retained austenite \( (\gamma_r) \) [4,5]. The formation of carbides is prevented by the high silicon content [6]. The microstructure may also contain some martensite \( (\alpha_m) \), obtained from the residual austenite. The martensite \( (\alpha_m) \) is formed during the final cooling from isothermal heat treatment when the temperature decreases below the martensite start \( (M_s) \) temperature [7]. Although the bainitic microstructure can be formed by continuous cooling, it is usually formed by isothermal heat treatment in a process known as austempering [8]. The formation of bainite during isothermal heat treatment below \( M_s \) was first studied by Radcliffe and Rollason [9], who observed that the formation of bainite below \( M_s \) was associated with the presence of martensite plates that accelerate the...
nucleation of bainite in the region adjacent to untransformed austenite.

Van Bohemen et al. performed dilatometry measurements and microstructural observation to clarify the characteristics of the isothermal decomposition of austenite in a temperature range below M_s [10] and observed an increase in the dilation of the sample during isothermal heat treatment below M_s. They also observed that the magnitude of dilation of the sample during isothermal treatment below M_s was consistent with that of the dilation of the sample during isothermal treatment above M_s, which is evidence that the remaining austenite had decomposed into bainite. The main factor responsible for the strength of martensite is the carbon content in solid solution. Thus, the strength of the martensite microstructure is strongly affected by tempering [8]. Unlike martensite, bainite is the least sensitive to tempering because it autotemper during its formation [11]. The tempering resistance of bainitic steel is different from that of the equivalent martensitic steel. The tempering resistance is associated with intense precipitation of fine carbides when the retained austenite decomposes. Furthermore, the fine carbides precipitate between bainitic ferrite (α_b) plates; thus, preventing the coarsening of the fine carbides. Since the thickness of the sheaves plays a key role in determining the strength of steel, restricting the coarsening of carbides by precipitation explains the tempering resistance of bainite [12,13]. Thus, the present study aims to investigate the effect of tempering temperature on the microstructure and hardness of SAE 9254 steel isothermally heat treated below M_s.

2. Experimental procedure

SAE 9254 steel is used to manufacture axles and coil springs. Its chemical composition and transformation temperatures, A_c1 and A_c3, were estimated using the empirical formulas of Andrews [14] and are listed in Table 1. The high Si content is responsible for preventing carbide precipitation [15–17] while the presence of Mn and Cr promote the hardenability and inhibit the formation of undesirable constituent phases such as ferrite and pearlite in quenched steels.

The steel was received in the hot-rolled condition (8-mm diameter wire rod). The microstructure of the steel consisted of fine pearlite and some islands of pro-eutectoid ferrite. Specimens were austenitized at 900 °C for 5 min prior to isothermal heat treatment at 200 °C, 220 °C, and 270 °C for 0.5, 2, 24, and 48 h in order to obtain a multiphase microstructure consisting of bainite, martensite, and retained austenite. The heat treatment was performed in an isothermal bath consisting of an alloy of Sn_50–Pb_50, which has a melting point below 200 °C. A type K thermocouple was inserted into the bath to control the temperature throughout heat treatment. After the isothermal heat treatment, the specimens were air-cooled to room temperature. Finally, the specimens were tempered at 350 °C, 400 °C, and 450 °C for 1 h (Fig. 1).

| Table 1 – Chemical composition of C-Si steel in wt%.
<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Cr</th>
<th>A_c1 (°C)</th>
<th>A_c3 (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-Si</td>
<td>0.56</td>
<td>0.58</td>
<td>1.43</td>
<td>0.47</td>
<td>766.00</td>
<td>820.00</td>
</tr>
</tbody>
</table>

The transformation start temperatures for martensite (M_s) and bainite (B_s) were calculated as 594 °C and 278 °C, respectively, according to Eqs. (1) and (2) [18]. Samples of the same steel were quenched in oil (cooling rate ≈ 22 °C/s) and tempered at 200 °C, 350 °C, 400 °C, and 450 °C for 1 h in order to compare their microhardnesses with those of isothermally heat-treated samples.

\[
B_s (°C) = 830 – 270C – 90Ni – 70Cr – 83Mo \quad (1)
\]

\[
M_s (°C) = 539 – 423C – 30.4Mo – 17.7Ni – 12.1Cr – 7.5Mo \quad (2)
\]

C, Mn, Cr, etc. are steel alloying elements (wt%). The samples were prepared using conventional metallographic techniques. The samples were then etched using LePera etchant [19]. This enables the identification of the constituent phases because they appear as different colors: bainite and martensite appear blue and brown, respectively [20]. Microstructural analysis was performed using scanning electron microscopy (SEM, FEI, model Inspct S50). Microhardness tests were performed in a Future Tech hardness machine, using the Vickers indenter with a load of 0.3 kgf and a loading time of 20 s; twenty measurements were performed along the diameter of the samples.

The volume fraction of the retained austenite (γ_r) was quantified using X-ray diffraction and the direct comparison method. This method considers the integration of the most intensive austenite peaks characterized by the (1 1 1), (2 0 0), (3 1 1), and (2 2 0) planes. The analysis was performed on a Philips PW 1710 diffractometer with Cu Kα radiation, at a rate of 0.02 °C/min, a delay time of 1 s, a voltage of 40 kV, and a current of 20 mA. X-ray diffraction data were also used to determine the carbon content in the retained austenite according to Eq. (3):

\[
a_v = 3.578 + 0.0033%C_r.
\]

where C_r is the carbon content (in wt%) in the retained austenite, and a_v is the lattice parameter (in Å) of the retained austenite [21,22].

From Fig. 1, a certain amount of martensite (V_m) must be formed for the isothermal heat treatments performed below
$M_s, V'_s$ can be estimated by applying the Koistinen–Marburger equation (4) \[7\]:

$$1 - V'_s = \exp[-0.011(M_s - T_r)].$$

(4)

where $M_s$ and $T_r = 25 \, ^\circ\text{C}$ represent the martensite-start and room temperatures, respectively. The martensite was tempered ($\alpha_{\mu}$) during the isothermal treatment. The maximum fraction of bainite ($V_b$) formed at a given temperature is determined using Eq. (5):

$$V_b = \frac{C_r - x}{C_r - x_b},$$

(5)

where $C_r$ is the carbon content in the retained austenite, $x$ is the average carbon content in the alloy, and $x_b$ is either the carbon content in the bainitic ferrite or in the solid solution ($x_b = 0.03 \, \text{wt.\%}$) \[23\]. The volume fraction of the martensite ($\alpha_{\mu}$) formed after the final cooling was calculated based on the difference in the volume fraction of the previously formed remaining constituents.

### 3. Results and discussion

The volume fractions of the constituents are summarized in Table 2. The amount of martensite ($\alpha_{\mu}$) formed during final cooling strongly depends on the carbon content ($C_r$) in the residual austenite. A higher carbon content ($C_r$) was observed for isothermal treatment performed at $270 \, ^\circ\text{C}$, causing $M_s$ to decrease below room temperature so that martensite was not formed during the final cooling, as indicated in Table 2.

![Table 2 – Volume fractions (%) of tempered martensite ($\alpha_{\mu}$), bainitic ferrite ($\alpha_b$), retained austenite ($\gamma_r$), carbon content in retained austenite ($C_r$), and martensite ($\alpha_{\mu}$).](image)

<table>
<thead>
<tr>
<th>Temperature ($^\circ\text{C}$)</th>
<th>Time (h)</th>
<th>$\alpha_{\mu}$</th>
<th>$\alpha_b$</th>
<th>$\gamma_r$</th>
<th>$C_r$</th>
<th>$\alpha_{\mu}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>0.5</td>
<td>57.5</td>
<td>12.6</td>
<td>07.6</td>
<td>0.7</td>
<td>22.3</td>
</tr>
<tr>
<td>220</td>
<td>0.5</td>
<td>47.1</td>
<td>26.5</td>
<td>09.2</td>
<td>1.0</td>
<td>17.2</td>
</tr>
<tr>
<td>270</td>
<td>0.5</td>
<td>08.4</td>
<td>81.3</td>
<td>10.3</td>
<td>1.2</td>
<td>–</td>
</tr>
</tbody>
</table>

Fig. 2a–c shows optical micrographs of the samples that were etched with LePera etchant after isothermal heat treatment at $200 \, ^\circ\text{C}$, $220 \, ^\circ\text{C}$ and $270 \, ^\circ\text{C}$, respectively. Bainitic ferrite ($\alpha_b$), retained austenite films ($\gamma_r$), and martensite ($\alpha_{\mu}$) were observed in transmission electron microscopy (TEM) micrograph of the samples isothermally heat treated at $200 \, ^\circ\text{C}$ and $270 \, ^\circ\text{C}$, according to reference \[24\]. Fig. 2d–f shows the same samples; however, they were tempered at $400 \, ^\circ\text{C}$ for 1 h. Martensite (brown region) was predominantly observed in the samples isothermally heat treated at either $200 \, ^\circ\text{C}$ or $220 \, ^\circ\text{C}$; the bainite is shown by the blue areas. Retained austenite blocks ($\gamma_b$), on the other hand, are shown by white regions.

![Fig. 2 – Optical micrographs of samples isothermally heat-treated at (a) 200 °C, (b) 220 °C, (c) 270 °C for 0.5 h. Figures (d), (e) and (f) represent same samples tempered at 400 °C for 1 h, respectively. Samples were etched with LePera etchant.](image)
Table 3 shows that the decomposition of the retained austenite (γ_r) primarily occurs in blocks (γ_b) because retained austenite films (γ_f) are more thermally stable than blocks (γ_b) since the retained austenite films (γ_f) have higher carbon content than the blocks [25].

The decomposition of retained austenite was previously observed by Podder and Bhadeshia [26]. They observed that a small amount of cementite precipitated from the austenite when the austenite was tempered at 450 °C for less than 1 h. Cementite precipitation led to a decrease in the carbon content of the austenite and consequently to the formation of untempered martensite upon cooling to room temperature.

Fig. 5 clearly shows that the microhardness of the samples decreases with increasing tempering temperature. Note that the lower microhardness value for the untempered sample is due to the bainite predominantly present in the sample, which is consistent with the formation of bainite below Ms [9,10] under all isothermal heat-treatment conditions. Further evidence of the formation of bainite below Ms is the difference in the microhardnesses of the sample quenched and tempered at 200 °C for 1 h and the sample isothermally treated at 200 °C for 0.5 h. Note that if the sample isothermally heat-treated at 200 °C for 0.5 h (715 HV) had only consisted of the martensite phase, the microhardness value of the sample would have been greater than or equal to that of sample quenched and tempered at 200 °C for 1 h (760 HV). However, the lower microhardness value of 715 HV is probably due to the presence of bainite in the sample. Moreover, the tempering temperature had a greater effect on the microhardness values for the quenched samples than it did on those for the isothermally heat-treated samples, which is consistent with the formation of bainite below Ms. A significant decrease in the microhardness value for the sample heat-treated at 270 °C could only be observed after the sample was tempered at 450 °C, because

![Image](71x578 to 282x736)

Fig. 3 – Effect of tempering temperatures on volume fraction of retained austenite.

Retained austenite blocks (γ_b) can easily be identified in the sample treated at 270 °C. Fewer white areas are present in the tempered samples because of austenite decomposition during tempering at 400 °C.

The decomposition of the retained austenite (γ_r) is indicated by the diffraction patterns obtained for the tempered samples. The intensities of the peaks representing the austenite phase decrease as the tempering temperature increases, as shown in Fig. 3.

The effect of the tempering temperature on the decomposition of the retained austenite (γ_r) is clearly observed in the sample isothermally heat-treated at 270 °C for 0.5 h since that sample contains more retained austenite (γ_r). The lower tempering temperature (350 °C) resulted in a decrease in the volume fraction of austenite. The volume fraction of the retained austenite (γ_r) and the carbon content (C_r) were only calculated for the tempering temperature of 350 °C since the peaks for austenite are not defined for higher tempering temperatures (Fig. 3). The results are listed in Table 3.

Table 3 – Volume fraction of retained austenite (γ_r) and carbon content (C_r) (wt.%) in sample tempered at 350 °C.

<table>
<thead>
<tr>
<th>Isothermal heat treatment (°C, h)</th>
<th>200, 0.5</th>
<th>220, 0.5</th>
<th>270, 0.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tempering temperature</td>
<td>γ_r</td>
<td>C_r</td>
<td>γ_r</td>
</tr>
<tr>
<td>350 °C</td>
<td>8.8</td>
<td>1.2</td>
<td>6.7</td>
</tr>
</tbody>
</table>
bainite was the predominant phase in the microstructure, which self-tempered as it formed [13,14]. Because the tensile strength (TS) is approximately equal to 3 times Vickers hardness value (HV) and because the fatigue limit (FL) corresponds to half of the tensile strength [27], the TS and FL values for SAE 9254 steel should be \( \approx 1881 \) and \( \approx 940.5 \) MPa, respectively, for the sample isothermally transformed at 200 °C and tempered at 400 °C. The SAE 9254 steel sample treated under these conditions has higher values for theoretical mechanical properties than the samples treated under the other conditions. The other treatments led to inferior mechanical properties. Moreover, commercial steel must be tempered at 400 °C to achieve the level of toughness appropriate for industrial application.
4. Conclusions

The study on SAE 9254 commercial steel isothermally heat-treated at various temperatures below $M_s$ and subsequently tempered at various temperatures produced the following results:

- The samples isothermally treated at 200°C, 220°C or 270°C contained the martensite ($\alpha_m$), bainitic ferrite ($\alpha_b$) and retained austenite ($\gamma_r$) phases. The samples isothermally heat-treated at 270°C contained more of the bainitic ferrite and retained austenite phases.
- The decomposition of retained austenite starts at the tempering temperature of 350°C.
- The microhardness values for the samples quenched and isothermally heat-treated at 200°C, 220°C or 270°C are associated with the large amount of martensite formed. The amount of martensite formed in the isothermally heat-treated samples significantly decreased with increasing isothermal heat-treatment temperature, thereby decreasing the microhardness values of the tempered samples.
- Unlike conventional quenching and tempering, the isothermal heat-treatment below $M_s$ applied in this study can be used to obtain higher hardness values for steel isothermally transformed at 200°C for any length of time and then tempered at either 400°C or 450°C.

Conflicts of interest

The authors declare no conflicts of interest.

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