Effects of Cooling Rate in an Innovative Heat Treatment Route for High-Strength Steels

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The requirement for high strength and ductility is usually associated with martensitic microstructure with a certain amount of retained austenite. One of the innovative heat treatment processes that can lead to such microstructure is the Q&P process (Quenching and Partitioning). It can produce microstructures consisting of martensite and a certain amount of retained austenite, which exhibit strengths above 2000 MPa and elongation levels of more than 10%. The objective of this research was to explore the effects of the cooling rate in the Q&P process and evaluate the effects of various microstructure constituents on mechanical properties of high-strength steels. Three newly-created experimental steels, which contained 0.43% carbon and had reduced Ms temperatures thanks to manganese addition, were subjected to several heat treatment routes which involved various cooling rates. The cooling rates were chosen on the basis of calculations using the JMatPro software and earlier results. It was found that by varying the cooling rate one can obtain various mixed microstructures and a wide range of mechanical properties. The strengths were in the range of 1200-2300 MPa and $A_{5mm}$ elongation levels were up to 18%. Because the amount of retained austenite has a considerable impact on the resulting mechanical properties, it was measured by means of X-ray diffraction.

Keywords: Q-P process, retained austenite, AHSS, X-ray diffraction

1 Introduction

Today’s advanced steels are required to possess high strength and ductility. High strength is guaranteed in martensitic steels [Fig. 1]. On the other hand, it tends to be at the cost of ductility in these steels. This deficiency can be overcome by producing additional phases in the microstructure. In advanced high-strength steels, such a phase is retained austenite which is present as foil-like particles along the boundaries between martensite laths. To keep retained austenite stable, carbon is needed to migrate from super-saturated martensite to retained austenite, instead of forming pearlite or carbides and leaving retained austenite depleted. Carbide formation can be prevented in steels which contain appropriate levels of manganese and silicon. Pearlite formation can be suppressed in steels with an appropriate chemistry and/or by suitable treatment parameters, particularly the cooling rate. For this reason, it is beneficial to use material-technological modelling in a thermomechanical simulator [Fig. 2]. With this method, small amounts of material can processed under laboratory conditions approaching the real-world process.

Fig. 1 Typical martensitic-bainitic microstructure with retained austenite after processing; the cooling rate was 1°C/s

Fig. 2 Specimen in a thermomechanical simulator during processing

One of the routes which lead to martensitic microstructures with retained austenite is the Quenching and Partitioning process (Q&P) [1, 2]. This method allows strengths of more than 2000 MPa to be achieved, together with elongation levels of about 10% [3, 4]. It is a complex process, in which a number of parameters must be optimized.
In high-strength steels, the microstructure and mechanical properties upon heat treatment are dictated by the cooling rate and, to a great extent, by their alloying concept. The elements that play a role in the stabilization of retained austenite include not only carbon but also manganese, silicon and nickel [5]. To simplify the processing routes, it is desirable to depress the \( M_s \) and \( M_f \) temperatures by manganese and molybdenum additions. If additions are made, cooling rate and, to a great extent, by their alloying concept. The elements that play a role in the stabilization of manganese, silicon and nickel [5]. To simplify the processing routes, it is desirable to depress the \( M_s \) and \( M_f \) temperatures by manganese and molybdenum additions. If additional solid solution strengthening is to be obtained and mechanical properties improved, chromium content is important [6].

2 Experimental programme

The use of the Q&P process in real-world treatments depends on the ability to interrupt quenching between the \( M_s \) and \( M_f \) temperatures. As this is a crucial aspect, four new experimental steels have been proposed. Their special chemistries were designed to depress the \( M_s \) and \( M_f \) temperatures (Tab. 1). The aim was to explore whether the process could be simplified and whether other cooling media or quenching routes might be used. In all these steels, the \( M_s \) and \( M_f \) temperatures were depressed predominantly through the addition of manganese, silicon, chromium, and molybdenum. Besides carbon, the main alloying element was manganese, as it considerably reduces the \( M_s \) and \( M_f \) temperatures and shifts the start of ferritic and pearlitic transformations towards lower cooling rates.

Silicon was chosen in order to prevent carbide formation to ensure sufficient super-saturation of martensite with carbon. Nickel was added in small amounts to stabilize austenite during cooling, to enhance hardenability, and to provide solid solution strengthening. The purpose of molybdenum was to depress the martensitic transformation temperature even lower and to retard the growth of austenite grains at high processing temperatures. The carbon content was the same in all steels: between 0.42 and 0.43%.

The JMatPro software was used for calculating approximate transformation temperatures. In the AHSS-1 steel, the manganese level was 2.5%, and the silicon level was 2.03%. The calculated \( M_s \) temperature was 218 °C and the \( M_f \) was 88 °C. In order to find whether molybdenum affects mechanical properties and transformation temperatures, its content in the AHSS-2 steel was set to 0.16%. However, this molybdenum content has not altered the \( M_s \) and \( M_f \) temperatures in any substantial way. The \( M_s \) temperature was 214 °C and the \( M_f \) was 83°C. In AHSS-3, the nickel level was 0.56% in order to provide the desired hardenability and to depress martensitic transformation temperatures. The \( M_s \) temperature was 209 °C and the \( M_f \) was 78°C. The only difference between AHSS-4 and AHSS-3 was in the molybdenum level. Thanks to its composition, AHSS-4 had the lowest transformation temperatures: the \( M_s \) was 204°C, and the \( M_f \) was 73°C.

\[ M_s [°C] = 723 - 414 \times C - 36.5 \times Mn - 36.5 \times Si - 36.5 \times Cr - 36.5 \times Ni - 36.5 \times Mo - 36.5 \times Nb \]

\[ M_f [°C] = 539 - 273 \times C - 54 \times Mn - 54 \times Si - 54 \times Cr - 54 \times Ni - 54 \times Mo - 54 \times Nb \]

Q&P process

In order to correctly evaluate the effects of individual parameters of the Q&P process on microstructural evolution – and therefore on mechanical properties – the prescribed parameter profiles must be followed, i.e. not only the quenching and partitioning temperatures but also the cooling rate. For these reasons, the experiments were carried out in a thermomechanical simulator. The simulator provides close and accurate control of temperature profiles and cooling curves, including those involving high rates of cooling [7-9].

Three different cooling rates from the austenitizing temperature (TA) to the quenching temperature (QT) and partitioning temperature (PT) were used in this experimental programme (Tab. 2). The purpose was to determine at what cooling rate the undesirable pearlite formation begins. The cooling rates were chosen on the basis of CCT diagrams for individual heats of the experimental materials [Fig. 3 – Fig. 5].

The austenitizing temperature of 850°C and the soaking time of 100 s, as well as the quenching temperature of 150°C and the partitioning temperature of 200°C were the result of an optimization carried out in earlier experiments.

\[ \text{Rate of cooling} = \frac{\text{Temperature difference}}{\text{Time}} \]

\[ \text{Cooling rate} = \frac{\Delta T}{\Delta t} \]

Tab. 1 Chemical compositions of experimental steels AHSS [wt. %]

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cu</th>
<th>Cr</th>
<th>Ni</th>
<th>Al</th>
<th>Mo</th>
<th>Nb</th>
<th>( M_s [°C] )</th>
<th>( M_f [°C] )</th>
</tr>
</thead>
<tbody>
<tr>
<td>AHSS1</td>
<td>0.430</td>
<td>2.5</td>
<td>2.03</td>
<td>0.006</td>
<td>0.003</td>
<td>0.07</td>
<td>1.33</td>
<td>0.07</td>
<td>0.008</td>
<td>0.03</td>
<td>0.03</td>
<td>218</td>
</tr>
<tr>
<td>AHSS2</td>
<td>0.428</td>
<td>2.48</td>
<td>2.03</td>
<td>0.005</td>
<td>0.003</td>
<td>0.07</td>
<td>1.46</td>
<td>0.08</td>
<td>0.004</td>
<td>0.16</td>
<td>0.03</td>
<td>214</td>
</tr>
<tr>
<td>AHSS3</td>
<td>0.419</td>
<td>2.45</td>
<td>2.09</td>
<td>0.005</td>
<td>0.002</td>
<td>0.06</td>
<td>1.34</td>
<td>0.56</td>
<td>0.005</td>
<td>0.04</td>
<td>0.03</td>
<td>209</td>
</tr>
</tbody>
</table>

Tab. 2 Heat treatment routes and results of mechanical testing

<table>
<thead>
<tr>
<th>Route number/steel type</th>
<th>( TA[°C]/TA[s] )</th>
<th>Rate of cooling from ( TA ) to ( QT[°C/\text{s}] )</th>
<th>QT ( [°C] )</th>
<th>PT ( [°C/s]) /( \text{time}[\text{s}] )</th>
<th>HV10 [-]</th>
<th>( R_m [\text{MPa}] )</th>
<th>( A5\text{mm} [%] )</th>
<th>RA [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1/AHSS1</td>
<td>850/100</td>
<td>0.5</td>
<td>150</td>
<td>200/600</td>
<td>592</td>
<td>2308</td>
<td>11</td>
<td>8</td>
</tr>
<tr>
<td>1/AHSS2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>521</td>
<td>2025</td>
<td>10</td>
<td>8</td>
</tr>
<tr>
<td>1/AHSS3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>507</td>
<td>1864</td>
<td>8</td>
<td>12</td>
</tr>
<tr>
<td>2/AHSS1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>634</td>
<td>2303</td>
<td>9</td>
<td>9</td>
</tr>
<tr>
<td>2/AHSS2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>345</td>
<td>1192</td>
<td>18</td>
<td>0</td>
</tr>
<tr>
<td>2/AHSS3</td>
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<td></td>
<td></td>
<td>613</td>
<td>2108</td>
<td>7</td>
<td>8</td>
</tr>
<tr>
<td>3/AHSS1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>638</td>
<td>2269</td>
<td>8</td>
<td>10</td>
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<tr>
<td>3/AHSS2</td>
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<td></td>
<td>664</td>
<td>2287</td>
<td>10</td>
<td>9</td>
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<tr>
<td>3/AHSS3</td>
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<td></td>
<td>596</td>
<td>2151</td>
<td>8</td>
<td>7</td>
</tr>
</tbody>
</table>

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Discussion of results

After route 1, which involved a cooling rate of 0.8°C/s, the AHSS-1 steel, which was alloyed with manganese, silicon and chromium, had a martensitic-bainitic microstructure with 10% retained austenite. No pearlite was found. The ultimate strength was 2269 MPa, the $A_5$ elongation reached 8% and the hardness was 638 HV10. After the cooling rate was reduced to 0.5 and 0.2°C/s, no appreciable changes in the microstructure were observed. Pearlite was not found even upon the route with the slowest cooling rate (Fig. 6, Fig. 7, Fig. 10, Fig. 11).

In the AHSS-2 steel, which, besides the same alloy additions as the other steels, contained molybdenum, pearlite was found upon the route with the cooling rate as high as 0.8°C/s. Pearlite was present along boundaries of prior austenite grains. (Fig. 8, Fig. 9, Fig. 12, Fig. 13, Fig. 14, Fig. 15). The ultimate strength was high as well: 2287 MPa. Elongation was 10%. The presence of pearlite did cause the amount of retained austenite to decrease. 9% of retained austenite was found in the microstructure by X-ray diffraction. After the slowest cooling rate, 0.2°C/s, the microstructure consisted of pearlite and martensite (Fig. 12, Fig. 13). No retained austenite was detected. The ultimate strength decreased considerably to 1190 MPa. The value of $A_5$ elongation was 18%. (Tab. 2).

The purpose of the AHSS-3 experimental steel, which was the last one to be experimentally treated, was to study the effect of nickel addition. Upon the route with the cooling rate of 0.8°C/s, pearlite was only found in small islands along prior austenite grain boundaries (Fig. 16, Fig. 17). Due to higher nickel level, this steel contained up to 12 vol. % of retained austenite (Tab. 2). The ultimate strength reached 2151 MPa and the elongation was up to 8%. As the cooling rate slowed, the amount of pearlite along grain boundaries increased.
Fig. 7 Route 1; AHSS-1; martensitic-bainitic structure, detail scanning electron micrograph

Fig. 8 Route 1; AHSS-2; martensitic microstructure with pearlite – optical micrograph

Fig. 9 Route 1; AHSS-2; martensitic microstructure with pearlite – detail scanning electron micrograph

Fig. 10 Route 2; AHSS-1; martensitic-bainitic structure, optical micrograph

Fig. 11 Route 2; AHSS-1; martensitic-bainitic structure, detail scanning electron micrograph

Fig. 12 Route 2; AHSS-2; martensitic microstructure with a higher amount of pearlite – optical micrograph
Fig. 13 Route 2; AHSS-2: martensitic microstructure with a higher amount of pearlite – detail scanning electron micrograph

Fig. 14 Route 3; AHSS-2: martensitic microstructure with a small amount of pearlite – optical micrograph

Fig. 15 Route 3; AHSS-2: martensitic structure with a small amount of pearlite – detail scanning electron micrograph

Fig. 16 Route 3; AHSS-3: martensitic-bainitic microstructure with a small amount of fine pearlite along prior grain boundaries – optical micrograph

Fig. 17 Route 3; AHSS-3: martensitic-bainitic microstructure with a small amount of fine pearlite along prior grain boundaries – detail scanning electron micrograph

4 Conclusion

Q&P process with various cooling rates was experimentally tested on newly-created AHSS-type steels alloyed with manganese, silicon, chromium, molybdenum and nickel. The evaluation focused on the effects of the cooling rate on pearlite formation and on mechanical properties in individual steels. After processing, the AHSS1 steel contained martensite, bainite and a certain amount of retained austenite. None of the cooling rates (0.2, 0.5 and 0.8°C/s) led to formation of pearlite. Very good mechanical properties have been achieved. The ultimate strength reached 2308 MPa and the elongation was 11%. The AHSS-2 steel, which contained a higher level of molybdenum, developed pearlite at a cooling rate as high as...
0.8°C/s. Its amount decreased with decreasing cooling rate. The largest fraction of pearlite was obtained with the cooling rate of 0.2°C/s. Due to a large fraction of pearlite, the ultimate strength dropped from 2025 MPa to 1192 MPa and hardness dropped from 521 HV10 to 348 HV10. In AHSS3 pearlite was only found in small amounts along prior austenite grain boundaries. In this steel, which had an elevated nickel content, the largest amount of retained austenite, 12% by volume, was found by X-ray diffraction. Due to its pearlite morphology, this steel does not show good mechanical properties.

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References


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