ULTRA-FAST ANNEALING OF HIGH STRENGTH STEEL

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Abstract: The mechanical properties of a cold-rolled HSLA steel are studied after intercritical annealing with conventional and ultra-high reheating rate followed by subsequent water quenching without isothermal soaking. By monitoring the hardness and the microstructure, it was shown that the increase of the reheating rate from 140 to 1500°C/s causes refinement of the ferrite grains from 4 µm to 1 µm in diameter. The final ferrite grain size depends significantly on the reheating temperature and reheating rate. It was observed that after an extreme reheating rate of 1500°C/s the α-to-γ phase transformation starts before the completion of the recrystallization in a recovered matrix. It was found that the ultra-fast reheating results in a very fine non-equilibrium ferrite-martensite structure with an excellent ultimate tensile strength of more than 1200 MPa and an acceptable elongation at fracture. The observed data are very promising from industrial-application point of view and open up possibilities for further microstructural refinement and alternative texture control.

KEYWORDS: ULTRA-FAST HEATING, GRAIN REFINEMENT, HIGH STRENGTH, RECRYSTALLIZATION, PHASE TRANSFORMATION, TEXTURE

1. Introduction

The development of advanced high strength steel grades (AHSS) is one of the enduring priorities of the steel manufacturing industry today and requires often non-conventional approaches in development of stronger and tougher steel grades. One reason is that the use of high strength steels in automotive industry appears to be an effective way to decrease the weight of vehicles, to increase the safety of the passengers and to decrease the fuel consumption. Such steels are known as 3rd generation advanced high strength steels and they are still under development by steel producers. The classical approach for the development of AHSS grades is two-fold (i) the use of special combinations of alloying elements and (ii) control of cooling rates, which determine microstructure formation. This approach appears to be very successful and gave rise to well-known steel grades like dual phase (DP) steels, transformation induced plasticity (TRIP) steels, multiphase or complex phase steels (steels with BCC dislocation rich microstructures such as martensite and bainite) [1] and recently twinned induced plasticity (TWIP) steels. The relation between the main engineering properties strength and ductility for the most popular steel grades is shown schematically in Fig.1.

![Fig. 1: Distribution of the existing steel grades according to their ultimate tensile strength (UTS) and tensile elongation (TE). The zone marked with a dashed oval is still without good representatives. (Adapted from ref. [1] with the data for TWIP steels)](attachment:image1.png)

It is clear that there are no representatives at the moment which can cover the gap between the heavy alloyed TWIP steels and low alloyed TRIP steels. Adding of alloying elements is an easy solution but the problems with metal scarcity will restrict their extensive use more and more in the future because of high prices limitations. The control of cooling rates during production is already very well studied; i.e., the limits for the use of this parameter are known and significant new improvements in this direction cannot be expected, although new heat treatment approaches like quenching and partitioning (Q&P) heat treatments are more and more investigated for industrial applications in order to marked with question mark region in Fig. 1 [2].

A non-conventional approach in this case is to explore the limits of the heat treatment as a tool for microstructure-properties control by using well known standard compositions and to design and produce fine grained steel by employing very high heating rates. This approach leads to extreme grain refinement [4-7] and to a corresponding increase of the strength of the material above the “normal” well known limits. In previous works [3-7] numerous microstructural phenomena like remarkable grain refining and overlapping of the recrystallization with the α-γ phase transformation were reported for the reheating rates of 3000°C/s and higher (up to 7000°C/s). Although the results were very promising, it is very difficult to reproduce such heating rates in the industrial lines. Therefore the goal of the current work is to study the microstructure and properties formation of cold rolled high strength low alloyed steel after heating and quenching with heating rates more affordable for industrial processing, between 100°C/s and 1500°C/s.

2. Experimental

Cold rolled steel with a composition shown in Table 1 is an object of study in this work.

<table>
<thead>
<tr>
<th>%C</th>
<th>%Si</th>
<th>%Mn</th>
<th>%P</th>
<th>%S</th>
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<tbody>
<tr>
<td>0.114</td>
<td>1.263</td>
<td>2.072</td>
<td>0.0149</td>
<td>&lt;0.001</td>
</tr>
</tbody>
</table>

Hot rolled plates were heat treated to obtain microstructures of ferrite and tempered at 550°C martensite (samples A) and microstructure of pearlite and ferrite (samples B). The steel sheets were next 75% cold rolled to a final thickness of 1 mm and heat treated according the temperatures and times shown in table 2. Samples of size 80x30x1 mm³ were cut parallel to the rolling direction of the cold rolled sheet. They were reheated with heating rates of 140°C/s, ~400°C/s and ~1500°C/s and subsequently water quenched from different temperatures in the temperature interval 500 to 1000 °C without isothermal soaking.
The reheating of the samples with ~140°C/s was implemented in a salt bath whilst the temperature was measured and controlled by a K1 type thermocouple welded to the surface of the sample. The reheating with average reheating rate of 400 °C/s and 1500°C/s was carried out by passing a high intensity electrical current through the strip specimen. The temperature measurement and control were done using an infrared pyrometer (IMPAC IW5) with an operating range between 400 and 1200°C and a response time of 1 ms. With such equipment it is possible to produce thermal cycles at heating rates between 200 and 7000 °/s with satisfactory temperature control and possibility of subsequent water quenching [6]. The detailed parameters of the annealing treatment are shown in Table 2 for samples with initial microstructure of ferrite and tempered martensite (samples A) and in Table 3 for samples with initial microstructure of ferrite and pearlite (samples B). A record of the real “reheating – quenching” cycles are shown in Fig.2 including the samples heat treated in a salt bath.

### Table 2: Heat treatment parameters and hardness of samples with microstructure 75% cold rolled F+TM

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Heating temperature Ta, °C</th>
<th>Heating rate, Vh, °C/s</th>
<th>Quench rate, Vq, °C/s</th>
<th>HV3</th>
</tr>
</thead>
<tbody>
<tr>
<td>A0</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>440</td>
</tr>
<tr>
<td>A1</td>
<td>674</td>
<td>430.2</td>
<td>-1281.7</td>
<td>405</td>
</tr>
<tr>
<td>A2</td>
<td>723</td>
<td>397.8</td>
<td>-821.9</td>
<td>360</td>
</tr>
<tr>
<td>A3</td>
<td>756</td>
<td>397.8</td>
<td>-1069.3</td>
<td>359</td>
</tr>
<tr>
<td>A4</td>
<td>780</td>
<td>328.7</td>
<td>-568.2</td>
<td>341</td>
</tr>
<tr>
<td>A6</td>
<td>807</td>
<td>357.5</td>
<td>-735.8</td>
<td>354</td>
</tr>
<tr>
<td>A7</td>
<td>846</td>
<td>323.4</td>
<td>-836.0</td>
<td>402</td>
</tr>
<tr>
<td>A8</td>
<td>879</td>
<td>317.3</td>
<td>-854.2</td>
<td>510</td>
</tr>
<tr>
<td>A9</td>
<td>949</td>
<td>352.9</td>
<td>-1064.4</td>
<td>561</td>
</tr>
<tr>
<td>A10</td>
<td>968</td>
<td>359.9</td>
<td>-1068.9</td>
<td>512</td>
</tr>
<tr>
<td><strong>AVERAGE</strong></td>
<td><strong>362.8</strong></td>
<td><strong>-922.3</strong></td>
<td><strong>440</strong></td>
<td></td>
</tr>
</tbody>
</table>

### Table 3: Heat treatment parameters and hardness of samples with microstructure 75% cold rolled F+P

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Heating temperature Ta, °C</th>
<th>Heating rate, Vh, °C/s</th>
<th>Quench rate, Vq, °C/s</th>
<th>HV3</th>
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</thead>
<tbody>
<tr>
<td>B0</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>329</td>
</tr>
<tr>
<td>B1</td>
<td>749</td>
<td>1567.6</td>
<td>-803.2</td>
<td>329</td>
</tr>
<tr>
<td>B2</td>
<td>841</td>
<td>1445.5</td>
<td>-1390.2</td>
<td>442</td>
</tr>
<tr>
<td>B3</td>
<td>862</td>
<td>1327.5</td>
<td>-1539.7</td>
<td>421</td>
</tr>
<tr>
<td>B4</td>
<td>1000</td>
<td>1419.0</td>
<td>-966.9</td>
<td>549</td>
</tr>
<tr>
<td>B5</td>
<td>1053</td>
<td>1538.1</td>
<td>-1058.7</td>
<td>545</td>
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<td>B6</td>
<td>1200</td>
<td>1532.2</td>
<td>-685.4</td>
<td>507</td>
</tr>
<tr>
<td><strong>AVERAGE</strong></td>
<td><strong>1471.7</strong></td>
<td><strong>-1074.0</strong></td>
<td><strong>329</strong></td>
<td></td>
</tr>
</tbody>
</table>

Samples for tensile test, hardness tests and microstructure characterisation were taken from the middle part of the heat treated sheets from the zones with warranted homogeneous temperature distribution. Sub-size tensile test samples with a gauge length of 4 mm, width of 1mm, thickness of 1mm and total length of 10 mm were tested with a cross head velocity of 0.5 mm/min at room temperature in micro-tensile test machine “Deben MICROTEST 5000N Tensile stage”. The sample elongation was detected by displacement measurement of the machine grips. Tensile test on sub-size samples always overestimates the strength and elongation in comparison to the standard size (A80 and A50) tensile test samples.

Similar tensile tests were applied to specimens which were water quenched from different temperatures after reheating with conventional heating rates of 140°C/s in a salt bath, in order to compare the results with the obtained from ultrafast heating, and to correctly evaluate the contribution of ultrafast heating to the mechanical properties. The microstructural characterisation was carried out after classical metallographic sample preparation which included mechanical grinding and polishing up to 1μm diamond suspension and etching with 2% HNO₃ in ethanol (Nital 2%). Samples for EBSD characterization were additionally electrolytically polished using Struers® Lectropol 5 device with A2 type electrolyte at 22°C, 40V and electrolyte flow rate of 7. FEI-Nova 600 scanning electron microscope was employed for the SEM and EBSD data acquisition from selected samples. The EBSD data were acquired at 15kV, working distance 7 mm, sample tilt 70° in square scan grid with a step size of 30nm. The data were post-processed with EDAX-TSL OIM® 5.1 data analysis software.

The variations in mechanical properties as a function of quench temperature were documented by Vickers hardness measurements with a load of 3kgf (HV3).

### Results and discussions

Variations in the hardness of the cold rolled and water quenched samples with different initial microstructures after heating to different temperatures with various heating rates are shown in Fig. 3. The initial hardness after cold rolling depends on the microstructure and it is 329HV3 for the samples from group B with ferrite-pearlite microstructure, whereas the hardness of the cold rolled microstructure of ferrite and tempered martensite is 440HV3. It does not change significantly after quenching from temperature up to 500°C (Fig. 3, zone I). An increase of the temperature with heating rates of 140°C/s (heating in a salt bath) triggers initially recovery and recrystallization in the cold rolled steel which is associated with a decrease in the hardness (cf. Fig. 3, zone II). After quenching from temperatures from the dual phase ferrite-austenite to the single phase austenite regions, the hardness increases due to the martensite formation from the parent austenite phase (cf. Fig. 3, zone III). When the austenitisation temperature is very high the hardness after quenching slightly decreases, which can be associated with the grain growth of the parent austenite which forms coarse and low carbon martensite (Fig. 3 zone IV).
Two main tendencies are clearly demonstrated in the hardness variations – apart from the heating temperature the hardness variations are dependent on: (i) the initial microstructure and (ii) the heating rate. The increase of the heating rate from 140°C/s to ~360°C/s and 1500°C/s shows a clear tendency to increase the hardness after quenching from both the intercritical (α+γ) and the single phase γ regions. The hardness decrease in the temperature interval 500-750°C (zone II in Fig. 3), which is associated with recovery and recrystallization, becomes smaller when the heating rate increase, which indicates that recovery and recrystallization are suppressed. No changes in the hardness are observed in samples B which were heated to the austenitic region with ~1500°C/s (cf. zone II in Fig. 3- the grey line for heating rate of 1500°C/s). This is an indication that both recovery and recrystallization are completely suppressed and the α-to-γ phase transformation starts before the onset of recrystallization (i.e. in unrecrystallized matrix). This effect was previously observed after heating and quenching of cold rolled HSLA steels with heating rates of ~300°C/s and ~700°C/s [3, 5, 6, and 7]. The results of tensile test for selected samples are shown in Fig. 4. The samples were water quenched from the temperatures of the two-phase (α+γ) region in order to create microstructures of ferrite and martensite, which is typical for the DP steels [1,2]. It is clearly demonstrated that the increase of the heating rate for intercritical annealing from 140°C/s to 400°C/s and 1500°C/s causes a significant increase in the ultimate tensile strength (Rm). This phenomenon could be associated with the significant grain refining effect observed in the fast heated samples in comparison to the samples heated in the salt bath. A second important observation is that the short soaking times, practically ~1-2 s in both cases, do not allow homogenization of the carbon content of the intercritical austenite before quenching. This means that the martensite “islands” are structurally heterogeneous- i.e. they may contain zones with high carbon martensite, low carbon martensite or even retained austenite with high carbon content and correspondingly with different hardness.

Another relevant observation is that the initial microstructure has significant effect on the mechanical properties (type of the tensile test curve), which is more clearly pronounced at low heating rates. The tensile test curves of samples with ferrite and pearlite microstructures quenched after heating with 140°C/s have well-defined yield point, and display very low work hardening, which is missing in the samples with initial microstructure of ferrite and tempered martensite. It is also important to mention that the
strain-stress curves reveal identical characters after ultrafast heating (compare the black thick and dashed lines in Fig. 4).

The evolution of the average ferrite grain size as a function of the heating rate was investigated on optical and SEM micrographs by means of linear interception method and a cross check of the result was made by EBSD measurement on samples A3 (cf. Fig. 7-black triangle). The results of the measurement, which are shown in Fig. 7, display that the average grain size drops from 5 µm to 1 µm with an increase of the heating rate from 140°C/s to 1500°C/s reaching a steady state. The last point in the figure represents data adopted from ref. [7] for steel with the same chemical composition heated to the same temperatures with 3000°C/s.

4. Conclusions

Results of the current study undoubtedly show that the ultrafast reheating of HSLA steels is a potential way for significant strengthening with a minimum decrease in elongation. The observed effects of increase in strength were associated with the strong grain refining effect caused by the overlapping of the α-to-γ phase transformation and recrystallization. The grain refining effects and the associated strengthening are observed even in a range of heating rates as high as 400°C/s, which could be obtained in the industrial conditions. Results also showed that the control of the initial microstructures before cold rolling together with the heating rates are powerful tools for designing HSLA steels with improved mechanical properties and to effectively fulfill the gap of the materials with strengths above 1200MPa and elongations of ~20% without expensive alloying additions.

5. References