Influence of Different Chemical Composition on Resistance of TRIP 800 Steel to Hydrogen Embrittlement

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Abstract

Resistance to hydrogen embrittlement was studied for 3 variants of TRIP 800 steel, based on C-Mn-Si; C-Mn-Si-Al and C-Mn-Si-P, by performing tensile tests on electrolytically hydrogenated specimens.

The specimens were prepared from laboratory heats, ingot weight was 150 kg. The ingots were forged into slabs, which were rolled into sheets 3 mm thick and subsequently cold-rolled to a thickness of 1.5 mm. The sheets were then subjected to two-stage annealing: the sheets were first annealed at the intercritical region, cooled rapidly, and annealed at the bainitic transformation region. All the variants examined met the requirements for the TRIP 800 steel. The yield point laid within the range of 420-490 MPa, tensile strength laid within the range of 880-950 MPa. The residual austenite content was 9-13% in dependence on the variants.

Specimens for the tensile tests serving for examination of their resistance to hydrogen embrittlement were obtained by hydrogenation in dilute sulphuric acid for 4 hours with application of a current density of 1 mA.cm\(^{-2}\). The elongation rate during the tensile test was approx. 10\(^{-3}\) s\(^{-1}\).

The increased hydrogen content in the steel brought about, in particular, decrease in ductility and, furthermore, change in the micro-mechanism of failure. Reduction of ductility was the biggest in the C-Mn-Si-Al variant and the smallest for the C-Mn-Si variant. The fracture morphology was basically comparable in all 3 variants. In addition to ductile failure regions, trans-granular cleavage fracture regions were observed following the electrolytic hydrogenation process. Fish-eyes were observed to a minor extent. Inter-granular brittle failure was not observed even in the variant with increased phosphorus content.

When comparing the 3 TRIP 800 steel variants quantitatively, the classical C-Mn-Si variant appears to be the most resistant to hydrogen embrittlement.

1. INTRODUCTION

TRIP steels (Transformed Induced Plasticity) belong to a group of high strength steels, which are known for their outstanding combination of mechanical properties, particularly high tensile strength \(R_m\) (around 850 MPa) and excellent ductility (up to 30%). This exceptionally advantageous specific combination of mechanical properties can be achieved by alloying and appropriate thermo-mechanical treatment. [1]

TRIP steels find extensive usage namely in automotive industry, where their main role is to absorb deformation energies at impact. Thanks to their lower mass they at the same time contribute also to reduction of weight of car body load bearing structure [2].

To this day low carbon TRIP steels with multi-phase micro-structure are formed mainly by matrix of polygonal ferrite (around 50%), bainite (30 – 35%) and residual austenite (15 – 20%), and they are most often alloyed by manganese 1.5% and silicon 1.5%. Some car components are exposed to adverse external environmental conditions, which causes corrosion of these parts. For this reason coating is an important operation in industrial production, especially galvanising of sheets and of some parts used in automotive
industry. The present research was focused on TRIP steels, in which silicon was replaced by other alloying elements, particularly by aluminium, which does not form on surface of TRIP steel oxides, which may hinder galvanising. Aluminium has, however, a tendency to form in the matrix non-metallic inclusions, which may deteriorate mechanical properties. Another possible solution for improvement of ability to galvanising consists in addition of phosphorus. It is questionable, whether this is an ideal solution, considering influence of phosphorus on mechanical properties of steel. During operations accompanying galvanising the so called hydrogen embrittlement of TRIP steels may develop. For safe production and assurance of high service properties it is important to know resistance of TRIP steels to hydrogen embrittlement [3 - 4].

Three different variants of TRIP 800 steel were used in the presented work: C-Mn-Si; C-Mn-Si-Al and C-Mn-Si-P. Their resistance to hydrogen embrittlement was investigated by tensile test on the test bars, which were preliminarily electrolytically hydrogenated. In this manner resistance to hydrogen embrittlement was described by parameters of chemical composition and by influence of individual alloying elements [5].

2. EXPERIMENT

Experiment was performed on three types of heats of TRIP 800 steel, which differed by their chemical composition. Individual heats were marked as 1, 2 and 3. Heat 1 was the type of steel with chemical composition C–Mn–Si, heat 2 was C–Mn–Si–Al, and heat 3 was steel C–Mn–Si–P.

Chemical composition of individual heats of steel is given in Table 1.

<table>
<thead>
<tr>
<th>Heat</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Cu</th>
<th>Mo</th>
<th>V</th>
<th>Ti</th>
<th>Al</th>
<th>Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.20</td>
<td>1.48</td>
<td>1.48</td>
<td>0.014</td>
<td>0.004</td>
<td>0.17</td>
<td>0.15</td>
<td>0.06</td>
<td>0.006</td>
<td>0.02</td>
<td>0.002</td>
<td>0.006</td>
<td>0.02</td>
</tr>
<tr>
<td>2</td>
<td>0.21</td>
<td>1.57</td>
<td>1.05</td>
<td>0.013</td>
<td>0.005</td>
<td>0.16</td>
<td>0.15</td>
<td>0.07</td>
<td>0.006</td>
<td>0.02</td>
<td>0.002</td>
<td>0.54</td>
<td>0.02</td>
</tr>
<tr>
<td>3</td>
<td>0.20</td>
<td>1.50</td>
<td>1.50</td>
<td>0.05</td>
<td>0.005</td>
<td>0.16</td>
<td>0.15</td>
<td>0.06</td>
<td>0.006</td>
<td>0.02</td>
<td>0.002</td>
<td>0.006</td>
<td>0.02</td>
</tr>
</tbody>
</table>

Initial material for all three types of steel was made as laboratory heats with ingot mass of 150 kg. The ingots were then re-forged into slabs and hot rolled to sheets with thickness of 3 mm, and then cold rolled to a thickness of 1.5 mm. The sheets were subjected to two-stage annealing – first at inter-critical temperature of 810°C with dwell of 4 minutes, followed by rapid cooling down to the temperature of isothermal bainitic transformation within the interval of 410°C - 425°C, where they remained for approx. 5 minutes, with subsequent free air cooling. Standard samples for tensile test were made from these sheets. Total length of the samples was L = 100 mm, width b = 10 mm, thickness a = 1.50 mm.

Initial mechanical properties of individual heats are given in Table 2.

<table>
<thead>
<tr>
<th>Heat</th>
<th>R_p0.2 [MPa]</th>
<th>R_m [MPa]</th>
<th>A_50 [%]</th>
<th>Content of residual austenite [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>460</td>
<td>950</td>
<td>29</td>
<td>11</td>
</tr>
<tr>
<td>2</td>
<td>415</td>
<td>913</td>
<td>28</td>
<td>14</td>
</tr>
<tr>
<td>3</td>
<td>417</td>
<td>944</td>
<td>21</td>
<td>9</td>
</tr>
</tbody>
</table>

Quantity of residual austenite varied within the interval of 9 – 14 %, measured by X-ray analysis.
Resistance to hydrogen embrittlement was tested by tensile test on the electrolytically hydrogenated samples. Hydrogenation took place in a solution of 0.05M H$_2$SO$_4$, at current density $i = 1$ mA·cm$^{-2}$ for 4 hours. Test samples were connected as cathode and platinum grid placed around the sample served as anode. Tensile test was performed immediately after termination of hydrogenation. Strain rate at tensile test corresponded to $\dot{\varepsilon} = 10^{-3}$ s$^{-1}$.

Embrittlement of the matrix is manifested during tensile test by drop of ductility. Index of hydrogen embrittlement, as one of indicators of hydrogen embrittlement, can be calculated from the following relation: (equation 1) [6]

$$F = \frac{A_0 - A_H}{A_0} \cdot 100 \% \quad (1)$$

where $A_0$ is ductility in non-hydrogenated state and $A_H$ is ductility in hydrogenated state.

The higher is the value of the index $F$, the higher is the level of hydrogen embrittlement of investigated material.

3. RESULTS OF EXPERIMENT

Within this experiment special attention was paid particularly to assessment of influence of additions of silicon, aluminium and phosphorus on mechanical properties of TRIP 800 steel by tensile tests and by evaluation of micro-mechanism of failure. Final values of mechanical properties after electrolytic hydrogenation and tensile test are given in Table 3.

Table 3

<table>
<thead>
<tr>
<th>heats</th>
<th>$R_{0.2}$ [MPa]</th>
<th>$R_m$ [MPa]</th>
<th>$A_{50}$ [%]</th>
<th>$F$ [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>456</td>
<td>898</td>
<td>14</td>
<td>51</td>
</tr>
<tr>
<td>T2</td>
<td>415</td>
<td>724</td>
<td>6</td>
<td>79</td>
</tr>
<tr>
<td>T3</td>
<td>454</td>
<td>1033</td>
<td>10</td>
<td>53</td>
</tr>
</tbody>
</table>

Effect of hydrogen was manifested by lower values of ductility $A_{50}$ and of ultimate strength in the heats 1 and 2, see Table 3, but also by change of micro-mechanism of failure. Micro-mechanism of failure of heats after tensile test was analysed by scanning electron microscope JEOL JSM 6490LV. Heat 1 in initial state, with no effect of hydrogen, had a mixed character of fracture area, as it can be seen in Fig. 1. Trans-granular ductile failure prevailed, but areas of cleavage fracture were also locally observed. After hydrogenation character of failure has changed in favour of trans-granular cleavage fracture, see Fig. 2. Initial state of the heat 2, Fig. 3, shows trans-granular ductile failure with occurrence of non-metallic inclusions of aluminium nitride type. After electrolytic hydrogenation mechanical properties have been considerably degraded, see Table 3. Change of character of failure is also evident, when trans-granular cleavage fracture prevails. Calculated index of hydrogen embrittlement $F$ in Table 3 and Fig. 4 confirm this.

The third heat with an increased content of phosphorus shows already in initial state lower values of ductility in comparison with previous heats. Trans-granular cleavage fracture was observed in greater extent than in the heat 1 already in initial state, see Fig. 5. After hydrogenation and tensile test the fracture surface consists exclusively of cleavage fracture, see Fig. 6. Inter-granular brittle failure was not observed.
3.1. Discussion of the obtained results

Results obtained by this experiment are valuable particularly from the viewpoint of usability of TRIP steels with different chemical composition. It follows from the results obtained in investigated variants that all 3 variants show comparatively high susceptibility to hydrogen embrittlement. This is surprising, especially with respect to comparatively high content of residual austenite, which supposedly should act as favourable and deep hydrogen trap. The best results gives the classical variant of TRIP steel based on Mn–Si. On the contrary to that the worst results, or the lowest resistance to hydrogen embrittlement, were observed in the variant of TRIP steel with an increased content of aluminium. This may be related to the fact that in this
variant higher occurrence of non-metallic inclusions rich in aluminium was observed, namely aluminium oxides and nitrides. Especially coarser particles of aluminium nitrides may act as the initiation sites of brittle (cleavage) fracture. [7] Presence of aluminium nitrides may be related to production of steel in electric arc furnace. The variant of TRIP steel with an increased content of phosphorus showed lower plastic properties already in initial state. Surprisingly though, its resistance to hydrogen embrittlement was not the worst one, as it might have been expected at the content of phosphorus of 0.05%. Significant degradation of steel by hydrogen is usually observed already at the contents of phosphorus above 0.01%. No inter-granular brittle failure was observed either, which is typical for hydrogen embrittlement of steels with increased content of phosphorus.

4. CONCLUSIONS

The presented paper evaluated resistance to hydrogen embrittlement of three different variants of TRIP 800 steel, namely: C-Mn-Si; C-Mn-Si-Al and C-Mn-Si-P. The obtained results may be summarised in the following manner:

- All investigated variants of TRIP 800 steel had comparatively low resistance to hydrogen embrittlement.
- Effect of hydrogen was manifested particularly by reduced ductility at tensile test, as well as by presence of trans-granular cleavage fracture on fracture surfaces.
- Steel variant C-Mn-Si had the highest resistance to hydrogen embrittlement.
- On the other hand the variant C-Mn-Si-Al had the lowest resistance to hydrogen embrittlement, which may be connected particularly with occurrence of coarser particles of aluminium nitrides.
- The variant of TRIP steel with an increased content of phosphorus showed resistance to hydrogen embrittlement comparable to that of the variant C-Mn-Si. This variant had, however, lower plastic properties in initial state.

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