MECHANICAL TESTING OF A HYDROGEN EMBRITTLED STEEL

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ABSTRACT

The paper presents a series of mechanical tests on specimens made of X 65 PSL 2 steel. Specimens were taken from a slab, obtained at a continuous casting machine. In addition to the metallurgical hydrogen (already in steel following the processes of the steelmaking and casting) a part of specimens have been hydrogenated by keeping them different time periods into hydrogen sulfide.

It is known that the presence of hydrogen in steel leads to its embrittlement, and thus its physical-mechanical properties become smaller. Impact and hardness tests tried to highlight this aspect in connection with hydrogenation time. Results obtained confirm that the mechanical properties of the tested steel were degraded with the increase of the hydrogenation time.

Keywords: Mechanical testing, Hydrogen embrittlement

1. INTRODUCTION

The embrittlement action of the hydrogen [1, 2] can be appreciated by same methods of investigation which give us information on the behavior of steel in the processes of deformation or breaking and lead to same criteria values [3].

The treatment of the specimens into an environment that contains hydrogen sulfide has been done under test HIC (NACE TM 0284 Standard [4]) using an acidified saline solution, where H₂S was bubbling.

Hydrogen sulphide is prepared in a Kipp dish, from sodium sulphide and sulphuric acid, according to the reaction:

\[ Na_2S + H_2SO_4 = Na_2SO_4 + H_2S \]

For HIC test, usually treating into hydrogen sulfide environment is made for 96 hours. For the tests performed were prepared more sets of specimens collected from the same areas of the slab, some untreated, some treated for 96 hours. To highlight the influence of hydrogenation on embrittlement some specimens were treated a double time that the one prescribed by the standard: 192 hours.

Till their tests, after hydrogenation the specimens were kept at low temperature (-10°C), in order to reduce the loss of hydrogen through diffusion.

2. EXPERIMENTAL RESULTS

From an intermediate slab of the charge parallelepipeded shaped block (200mm x 250 mm x 1550 mm) was cut (fig. 1).

![Fig. 1. The position of the samples block on the slab](image)

From the block thus obtained were debited blocks that were used for samples for traction,
hardness, impact tests and others that were not presented in this paper [5] (fig. 2).

2.1. The tensile test

For the mechanical characterization of slab steel was performed a tensile test. The cutting of samples was done so that the fragments from the edges that are a heat affected zone, to be removed. The sampling areas of the samples, denoted 1, 2, 3 are shown in fig. 3.

Traction test specimens will be coded T-1, T-2, T-3. It must be notice that the sample T-3 is taken from the surface of the slab, and T-1 from the middle.

The specimens have circular section, diameter 10 mm, length (Lo) 80 mm. Grip heads are rectangular, measuring 30 X 35 X 110 mm (fig. 4).

Tensile tests were performed according to SR EN 10002-1:2002 [6], on a tensile test machine (USA) S:5594 K1180. There were tested three samples (from each region of the sample block for tensile test).

The ultimate strength and elongation values are shown in table 1.

Table 1. Ultimate strength ($R_m$) and ultimate elongation values ($A$)

<table>
<thead>
<tr>
<th>Samples</th>
<th>$R_m$ (N/mm$^2$)</th>
<th>$A$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T-1</td>
<td>533</td>
<td>18</td>
</tr>
<tr>
<td>T-2</td>
<td>515</td>
<td>6</td>
</tr>
<tr>
<td>T-3</td>
<td>539</td>
<td>24</td>
</tr>
</tbody>
</table>

The representation of the values from table 4.16 is shown in fig. 5.

From the charts it appears that both resistances to breaking and breaking elongation are higher for the sample taken from the surface of the slab than to those from inside. The difference has been explained by samples microstructure. [7]. It results that that the
samples taken from the slab surface have a ferritepearlite structure with small and inhomogeneous grains. The ferrite is lamellar and globular. The samples taken from middle of the slab have a ferritepearlite casting structure with bigger and inhomogeneous grains. Borders between grains are not always obvious. It is noticed many gas holes specific to casting and chemical inhomogeneity.

2.2. Impact bending test on hydrogenated and nonhydrogenated specimens

From the initial block was cut another block for impact test samples as shown in fig. 2. From this block were obtained then 9 samples according to fig. 6. The dimensions of the samples are $L \times a \times b = 55 \times 10 \times 10$ mm, notch in V, $\alpha = 45^\circ$, $h = 2$, notch radius $= 0.25$ mm.

In fig. 7 it can see pieces before breaking. The two specimens presented are: nonhydrogenated, the glossy one and hydrogenated (mate).

The samples were treated like this:
- samples 1, 5 and 10 are nonhydrogenated, being encoded: I-1, I-5, I-10;
- samples 2, 6 and 11 were treated in a hydrogen sulfide environment for 96 hours, being encoded: I-2(96), I-6(96), I-11(96);
- samples 3, 7 and 12 were treated in a hydrogen sulfide environment for 192 hours, being encoded: I-3(192), I-7(192), I-12(192).

The specimens were tested at $0^\circ$ C, in accordance with the standard [8] on a computerized Tinius Olsen IT 542 E testing machine. For cooling of the samples, they were introduced in a thermostatic controlled electronic. Test results are presented in table 2.

<table>
<thead>
<tr>
<th>Hydrogenation time [hours]</th>
<th>Samples code</th>
<th>Impact bending strength (J)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>I-1</td>
<td>12.46</td>
</tr>
<tr>
<td></td>
<td>I-5</td>
<td>6.01</td>
</tr>
<tr>
<td></td>
<td>I-10</td>
<td>4.99</td>
</tr>
<tr>
<td>96</td>
<td>I-2(96)</td>
<td>12.08</td>
</tr>
<tr>
<td></td>
<td>I-6(96)</td>
<td>4.71</td>
</tr>
<tr>
<td></td>
<td>I-11(96)</td>
<td>4.34</td>
</tr>
<tr>
<td>192</td>
<td>I-3(192)</td>
<td>10.35</td>
</tr>
<tr>
<td></td>
<td>I-7(192)</td>
<td>4.80</td>
</tr>
<tr>
<td></td>
<td>I-12(192)</td>
<td>4.33</td>
</tr>
</tbody>
</table>

The results for impact bending test are shown in fig. 8. From these tests on hydrogenated and nonhydrogenated samples results:
- the impact bending strength decreases for inside slab samples: I-5; I-10; I-6(96); I-11(96); I-7(192); I-12(192) and has a minimum value for the specimens from the middle: I-10; I-11(96); I-12(192);
- for the hydrogenated specimens the impact bending strength are smaller than for nonhydrogenated ones, because hydrogen embrittlement;
- when the duration of hydrogenation is higher, 192 hours compared to 96 hours, the impact bending strength is less, due to the higher embrittlement aspect connected to the hydrogenation time.

As a result of the test is was observed that for non-hydrogenated samples, the breaking occurred without separate fragments, in addition, analyzing the breaking zone, it is found that it has a semi-ductile aspect, opposed to the hydrogenated samples, where the breaking occurred with the separation of fragments, and the zone has a fragile appearance.

2.3. Hardness tests

It was determined the Brinell hardness on specimens used to impact bending test denoted by 1, 2 and 3 in fig. 6.
The sample number 1 was nonhydrogenated and was encoded H-1, the sample number 2 was hydrogenated 96 hours and was encoded H-2(96), the sample number 3 was hydrogenated 192 hours and was encoded H-3(192).

The hardness was measured according to [9] on an optical projection apparatus WPM-MP-O-Leipzig, with a 2.5 mm penetration ball. The test load was 62.5 daN applied for a period of 15 seconds. For each sample were carried out two determinations. The average values obtained are shown in table 3.

In fig. 9 are shown the average values obtained for the Brinell hardness.

### Table 3. The average values for hardness test

<table>
<thead>
<tr>
<th>Hydrogenation time [hours]</th>
<th>Samples code</th>
<th>HBW hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>H-1</td>
<td>166.5</td>
</tr>
<tr>
<td>96</td>
<td>H-2(96)</td>
<td>144</td>
</tr>
<tr>
<td>192</td>
<td>H-3(192)</td>
<td>138.5</td>
</tr>
</tbody>
</table>

3. CONCLUSIONS

During the hydrogenation of the X 65 steel specimens, the hydrogen meats: non-metallic inclusions, lattice defects, gas holes etc., which may consist of traps, especially those in the middle of the slab. The pressure created by trapped hydrogen, plus the increased level of internal tensions could lead to the emergence of microcracks. These internal tensions and microcracks decrease the mechanical properties of hydrogenated samples.

The tensile test shows that both ultimate strength and ultimate elongation are higher for the samples from the surface of the slab than for those from inside. The differences are justified by the slab structure. On the surface is a ferrite-pearlite structure with small and inhomogeneous grains, with few non-metallic inclusions and gas holes. Inside the structure have the big grains and many non-metallic inclusions and gas holes.

From the impact bending tests results:
- the values of the impact bending strength decreases for specimens from inside of the slab and has a minimum value for those from the middle;
- for the hydrogenated specimens the values of the impact bending strength are smaller than for nonhydrogenated ones, due to hydrogen embrittlement;
- when the duration of hydrogenation increases, the values of the impact bending strength decreases, due to a higher hydrogen embrittlement;
- it was found that for nonhydrogenated samples, the breaking occurred without separate
fragments, in addition, analyzing the breaking zone, it is found that it has a semi-ductile aspect, opposed to the hydrogenated samples, where the breaking occurred with the separation of fragments and the zone has a fragile appearance.

From the hardness test, it appears that this decreases for a long duration of hydrogenation.

REFERENCES

7. *** SR ENV 10247:2000, Micrographic examination of the nonmetallic inclusion content of steels using standard pictures