Microstructural Analysis of Simulated Heat Affected Zone in Creep Resisting Steel

Ljubica Milović1,a, Tomaž Vuherer2,b, Milorad Zrilić1,c, Dejan Momčilović3,d, Slaviša Putić1,e
1Faculty of Technology and Metallurgy, Karnegijeva 4, Beograd, Serbia
2Faculty of Mechanical Engineering, Smetanova 17, Maribor, Slovenia
3Institute for Testing of Materials, Bulevar vojvode Mišića 43, Serbia
a acibulj@tmf.bg.ac.yu, b tomaz.vuherer@uni-mb.si, c misa@tmf.bg.ac.yu, d dejanmomcilovic@yahoo.com, e slavisa@tmf.bg.ac.yu

Keywords: P91, microstructure, simulation of the HAZ, HAZ toughness

Abstract. In the last few decades, the steels intended for use in fossil-fired power plants have been further developed worldwide. These materials are required to endure the ultra supercritical steam conditions, increasing their efficiency of power generation. This paper presents the evaluation of relation between the composition and microstructure of ferritic power plant steel. Microstructure zones where type IV failures may occur have been studied in details.

Introduction

In the seventies of the past century steel T91 for tubes and steel P91 for thick walled components (headers and steam lines) were developed. The microstructure of both steels consists of tempered martensite stabilised by M23C6 carbides with further strengthening due to the presence of molybdenum in solid solution and a fine distribution of vanadium/niobium rich carbonitride (MX) precipitates [1]. Experience of using these steels in European power industry (Germany, Denmark) shows that the steam parameters used approached 300 bar and 600 °C, representing net efficiencies in the range of 45-46% for an inland location [2].

This paper presents the evaluation of relation between the composition and microstructure of ferritic power plant steel.

Experimental

Material. For this investigation, a tube of P91 steel with 320 mm diameter, wall thickness t = 14 mm and 140 mm long was used. Chemical composition of steel P91 tested in this work is given in Table 1.

Table 1. Chemical composition of tested material, mas.%

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Cu</th>
<th>Al</th>
<th>Mo</th>
<th>As</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.120</td>
<td>0.289</td>
<td>0.396</td>
<td>0.009</td>
<td>0.002</td>
<td>8.04</td>
<td>0.080</td>
<td>0.082</td>
<td>0.024</td>
<td>0.850</td>
<td>0.007</td>
</tr>
<tr>
<td>W</td>
<td>Sb</td>
<td>Co</td>
<td>N</td>
<td>Ti</td>
<td>B</td>
<td>V</td>
<td>Nb</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.013</td>
<td>0.0091</td>
<td>0.012</td>
<td>0.0400</td>
<td>0.003</td>
<td>0.0002</td>
<td>0.242</td>
<td>0.073</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The structure of parent metal consisted of tempered lath martensite. On some boundaries of prior austenitic grains, the presence of isolated coarser carbides exceeding 1µm was observed, Fig. 1.
Typical temperatures of transformation during the welding cycle were determined from dilatometric curve, and these are $A_{c1}=835°C$ and $A_{c3}=930°C$. Heating rate was 53.8 °C/s. During cooling, formation of martensite started at temperature of 375°C, while martensite transformation completed at temperature of 210°C, Fig. 2. Martensite formation was preceded by separation of a smaller quantity of bainite.

Simulation. Evaluation of the effects of various welding thermal cycles on microstructure and mechanical properties of test material using thermal simulator SMITWELD, the specimens 11x11x70 mm were used. Different heat affected zone (HAZ) microstructures were obtained by simulation of single-pass welding at temperatures of 1386°C, 1300°C, 1250°C up to 950°C, graded by 50°C, 925°C, 900°C and 850°C and with cooling time between 800°C and 500°C, $t_{8/5}$, 40 sec. After welding, the samples were subjected to post-weld heat treatment (PWHT) at 730°C for 1 hour, Fig.3.

All samples were subjected to inspection of microstructure and measurement of hardness. For examination on scanning electron microscope (SEM), the samples heated to maximum temperatures of 1300°C and 925°C were chosen and subjected to PWHT.
Results and Discussion

Grain size was homogeneous up to temperature of 1000°C. There was no abnormal grain growth, as the grain growth most probably was governed by vanadium-nitride (VN) and niobium-nitride (NbN) precipitates [3]. Starting from 1150°C, different grain size caused by incomplete recrystallisation was observed, with the following minimum and maximum grain size, Table 2.

<table>
<thead>
<tr>
<th>Simulated zone of temperature</th>
<th>Minimum grain size</th>
<th>Maximum grain size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tₚ[°C]</td>
<td>Minimum grain size</td>
<td>Maximum grain size</td>
</tr>
<tr>
<td>1150</td>
<td>10.5</td>
<td>8.5</td>
</tr>
<tr>
<td>1200</td>
<td>10</td>
<td>7</td>
</tr>
<tr>
<td>1300</td>
<td>9.5</td>
<td>6.5</td>
</tr>
<tr>
<td>1386</td>
<td>9</td>
<td>5.5</td>
</tr>
</tbody>
</table>

The appearance of microstructures at temperatures of 925°C, 1100°C, 1300°C and 1386°C is shown in Fig. 4.

The microstructure corresponding to austenitisation temperature of 925°C, Fig. 4a), consists of martensite with less prominent martensite laths. Dilatometric curve, Fig. 2, shows that in addition to martensite, up to simulation temperature of 1150°C and with cooling time tₜ₈/₅ of 40 sec, certain quantity of bainite formed due to incomplete solubility of carbide phase and reduced content of alloying elements in solid solution of austenite, which caused the decrease of tempering-hardening capacity. Based on dilatometric curve, Fig. 2, percentage of transformation of ferrite into austenite was determined to be approx. 90%. At some prior austenitic grain boundaries, the presence of coarser carbides above 1 µm was observed. The presence of the carbides is most probably the result of short time spent on austenitisation temperature, considering that the carbides of M₂₃C₆ type completely dissolve at temperatures above 900°C [3].
The microstructure corresponding to austenitisation temperature of 1100°C, Fig. 4b), consists of tempered martensite with clearly visible martensite laths. Coarser carbide particles above 1µm are still present.

The microstructure corresponding to austenitisation temperature of 1300°C, Fig. 4c) and 1386°C, Fig. 4d, also represents tempered lath martensite. Carbide particles above 1µm at the prior austenite grain boundaries practically are not present.

The application of SEM enables more detailed analysis of distribution and size of the carbides of M23C6 type, most frequently separated at the boundaries of previously austenitic grains and at the boundaries of martensite laths as well. In Fig. 5a), one can see that M23C6-type carbides are coarser at the boundaries of previously austenitic grains than those separated at the boundary of ferritic subgrains. Distribution of carbide phase M23C6 after digital image post processing of Fig. 5a) is shown in Fig. 5b), and the results of measurement of carbide surface distribution in Fig. 5c).

Resulting photos and diagram for Tp= 1300°C are shown in Figs 5d), e) and f). By comparison of photos in Figs 5a) and b) with 5d) and e), one can see that quantity and size of the carbides present in the sample treated at 1300°C are significantly smaller, which is also confirmed by the results of statistic distribution of equivalent carbide diameter shown in Fig. 5c) and 5f) and given in Table 3.

Beside the carbides of M23C6-type, a significant role is that of carbonitride precipitates MX tested by transmission electron microscopy [5]. In present paper, this type of carbonitrides has not been tested.
Hardness. Hardness of parent metal of 230 HV1 was measured. Variation of hardness of simulated samples with temperature of simulation showed a drop at 925°C, Fig. 6, and that is why this temperature of 925°C was chosen as typical for examination. A significant drop of hardness of almost 40HV occurs at simulation temperature of 850°C that is somewhat above A_{C1} temperature. With an increase of simulation temperature, ferrite fraction transforming into austenite increases. At temperature of 925°C, this fraction was approx. 90%. At this temperature, there was no considerable dissolution of carbides and nitrides and transformation of C and N into solid austenite solution. Therefore, martensite formed at cooling had significantly lower content of C and N. Due to decreased content of C and N, repeated separation of M23C6 carbides and MX carbonitrides during subsequent heat treatment was limited resulting in hardness lower than that of base metal. Besides, carbonitride precipitates MX coarsed-became rough and coagulated, which reduced their hardening effect. Due to the phenomena specified, FGHAZ corresponding to simulation-
temperature interval from 900 to 950°C had the smallest hardness. With an increase of simulation temperature above 950°C, hardness increased due to higher solubility of carbides.

![Graph showing measured hardness of tested samples](image)

**Fig. 6. Measured hardness of tested samples**

**Conclusion**

It is necessary to investigate in more details the effect of the technology of manufacture of the components made of steel P91, including final heat treatment (normalisation + tempering), on the state of carbide phase in them, and through it on the structure in FGHAZ. Optical microscopy does not enable complete determination of the structure properties that are essential for fitness for purpose of the components and weld joints made of steel P91, about which care should be taken when defining the requirements according to item S6 of the ASTM 335-03 standard.

**Acknowledgement**

The authors express their appreciation to Ivo Blačić from the Belgrade Military Technical Institute, for invaluable help and suggestions in writing this paper.

**References**