PRODUCTION AND HEAT TREATMENT OF DUPLEX STAINLESS STEEL FORGINGS

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Abstract

Development of offshore oil and natural gas production requires seawater corrosion resisting materials. Duplex stainless steels represent a group of materials that show an interesting combination of strength properties and resistance to stress-corrosion cracking initiated by chlorides.

The production of duplex stainless steel in ZDAS Inc. includes primary and secondary metallurgic processes. For production of the basic liquid metal, the electric arc furnaces are used. The processing of liquid metal takes place in the secondary metallurgy equipment. For successful realization of heavy forgings made by open-die forging technology it is necessary to observe the specific conditions of forming and heat treatment.

The achieved microstructure of duplex stainless steel then shows a uniform proportion of ferritic and austenitic grains without undesirable inter-metallic phases.

Key words: Duplex Stainless Steel, Mechanical Properties, Forgings.

1. INTRODUCTION

Verification of possibilities of production of smith forged forgings from duplex steels in conditions of the company ŽĎAS, Inc. started on the basis of market requirements and in the effort for enlargement of production assortment in 2010. Progress of experimental works was realised in accordance with the plan of solution of the research project FR-T11/222 PROGRESTEEL.

2. MAKING OF STEEL AND CASTING OF INGOTS

Production of duplex steels in conditions of ŽĎAS, Inc. assumes use of primary and secondary metallurgy units. Electric arc furnaces with capacity of 14 and 20 tons are used for production of the basic melt. The subsequent processing on the secondary metallurgy unit requires at production of duplex steels processing in Ladle Furnace (LF) and in the units of Vacuum Oxygen Decarburing (VOD) and Vacuum Degassing (VD).

Within experimental works the possibilities of production of the duplex steel X2CrNiMoN2253 according to the EN10088-3 were verified [1]. Chemical composition of the steel X2CrNiMoN2253 according to the EN10088-3 is given in table 1.
Table 1: Basic chemical composition of the steel X2CrNiMoN2253 acc. EN10088-3 [1]

<table>
<thead>
<tr>
<th>(wt. %)</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>min.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>21.00</td>
<td>4.50</td>
<td>2.50</td>
<td>0.10</td>
</tr>
<tr>
<td>max.</td>
<td>0.03</td>
<td>2.00</td>
<td>1.00</td>
<td>0.035</td>
<td>0.15</td>
<td>23.00</td>
<td>6.50</td>
<td>3.50</td>
<td>0.22</td>
</tr>
</tbody>
</table>

From the perspective of the required chemical composition specified in table 1, the duplex steel X2CrNiMoN2253 belongs to the group of high alloyed Cr – Ni – Mo steels with very low carbon concentration and with increased nitrogen content. Requirements for chemical composition include also achievement of a *Pitting Resistance Equivalent* PRE = Cr + 3.3 . Mo + 16 . N > 35. Table 2 documents the achieved chemical composition of the experimental melt with the mass of 16.4 tons.

Table 2: Basic chemical composition of experimental heat - steel X2CrNiMoN2253

<table>
<thead>
<tr>
<th>(wt. %)</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>N</th>
<th>PRE</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.025</td>
<td>1.41</td>
<td>0.28</td>
<td>0.016</td>
<td>0.007</td>
<td>22.30</td>
<td>5.60</td>
<td>3.25</td>
<td>0.178</td>
<td>35.87</td>
</tr>
</tbody>
</table>

The resulting basic chemical composition of the experimental melt of duplex steel given in tab. 2 documents fulfillment of requirements for concentrations of alloying and tramp elements. At the same time the required value of the PRE was achieved as well.

Produced steel was poured into moulds for casting of forging ingots of the type 8K with mass from 1000 to 3100kg. Schaeffler’s diagram [2] was used for control of chemical composition in relation to the expected steel structure, which was performed on the basis of calculation of Cr and Ni equivalents according to the equations (1) and (2):

\[ Cr_{\text{ekvivalent}} = Cr + 2 \cdot Si + 1,5 \cdot Mo + 5 \cdot V + 5,5 \cdot Al + 1,75 \cdot Nb + 1,5 \cdot Ti + 0,75 \cdot W \]  \hspace{1cm} (1)

\[ Ni_{\text{ekvivalent}} = Ni + Co + 0,5 \cdot Mn + 0,3 \cdot Cu + 25 \cdot N + 30 \cdot C \]  \hspace{1cm} (2)

Schaeffler’s diagram in Figure 1 shows the expected share of ferritic and austenitic phases in the forging made of duplex steel with chemical composition given in table 2.

![Fig 1: Schaeffler’s diagram: expected structure of experimental material [2]](image-url)
As it is evident from Fig 1, the expected structure of duplex steel with chemical composition according to table 2 will be formed by the share of more than 40% of ferritic phase in prevailing austenitic structure.

Verification of properties of real products made of duplex steel was made by forming of cast ingots and by evaluation of the achieved mechanical properties and structure in pre-defined testing across the cross-section of the forging.

3. FORMING AND HEAT TREATMENT OF FORGINGS

The issues related to forming of duplex steels concern thermal mode of the forging and assuring of sufficient deformations without risk of formation of fissures and cracks. Thermal mode of forming of duplex steel must take into account a posy of occurrence of undesirable inter-metallic phase in temperature interval below 950°C. Duplex steel X2CrNiMoN2253 shows precipitation of undesirable phases in the temperature interval according to the precipitation diagram in Fig 2 [3].

As it is evident from Figure 2 it is necessary to maintain the temperature of the forging in the temperature interval above 950°C. When temperature of the forging drops below this value its structure is changed and inter-metallic phases are created. Presence of inter-metallic phases in structure of duplex steel then causes a distinct drop of the level of mechanical properties and corrosion resistance of the final product.

In spite of the fact that in the temperature interval above 950°C according to the diagram shown in Figure 2 it is impossible to assume a possibility of formation of inter-metallic phases, due to operational conditions of production of large forgings the issue of influence of long-term dwell of the forging in the interval of forging temperatures on the structure of the forging was investigated.

Experimental works focused on these issues were performed on the forgings made of steel from the experimental melt with chemical composition given in table 2. Preparation of ten samples for testing with dimensions of 100x100x150mm was made by forming of the ingot 8K1.1 with mass of 1000kg in the temperature interval from 1150 to 950°C with subsequent cooling on air. Verification of influence of long-term dwell at forging temperature consisted in the subsequent heating of testing samples to the temperature of 1180°C (furnace temperature) with dwells from 2 to 20 hours. The samples were during this dwell at this temperature taken from the furnace every 2 hours.
Fig 3 to 6 document the obtained resulting structures of the forgings made of the steel X2CrNiMoN2253 for selected times of dwell at the top forming temperature. Analysis of micro-structure was performed on the samples from axial part of the forgings and metallographic polished sections were made in longitudinal direction in respect to the axis of original ingot.

![Fig 3: Structure X2CrNiMoN2253 dwell at the top forming temperature 4 hours/air (500 x)](image1)

![Fig 4: Structure X2CrNiMoN2253 dwell at the top forming temperature 8 hours/air (500 x)](image2)

![Fig 5: Structure X2CrNiMoN2253 dwell at the top forming temperature 16 hours/air (500 x)](image3)

![Fig 6: Structure X2CrNiMoN2253 dwell at the top forming temperature 20 hours/air (500 x)](image4)

As it is evident from Figs. 3 to 6, micro-structure of all the analysed samples is practically identical. The structure is formed by ferritic and austenitic grains without obvious presence of inter-metallic phases.

With respect to operational conditions, where ingots of various mass and cross-section are processed, it is possible to state on the basis of results of this experiment that influence of duration of dwell of material at the top forming temperature on the risk of occurrence of undesirable inter-metallic phases is negligible.

4. RESULTS OF EVALUATION OF EXPERIMENTAL FORGING MADE OF DUPLEX STEEL

Experimental works aimed at verification of the obtained mechanical properties and structure of the forging made of the steel X2CrNiMoN2253 were performed on the forged bar with diameter of 345mm and length of 605mm. Table 3 summarises the basic requirements to the level of mechanical properties of the steel X2CrNiMoN2253 according to the EN 10088-3.
However, the above requirements to mechanical properties of the forgings made of duplex steel X2CrNiMoN2253 according to the EN 10088-3 are related to the maximum dimension of the forging of 160mm. The standard does not specify the required mechanical properties for the forgings of larger diameters.

At production of forgings for applications for offshore oil and gas extraction the forgings of larger diameters are required and mechanical properties are then specified by customers. Table 4 gives a concrete example of standard requirements to the forgings made of duplex steel X2CrNiMoN2253 determined for the segment of offshore oil and gas extraction.

Table 3: Specification of mechanical properties of duplex steel X2CrNiMoN2253 acc. EN 10088-3

<table>
<thead>
<tr>
<th>EN 10088-3</th>
<th>-20°C</th>
<th>+20°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Re</td>
<td>Rm</td>
</tr>
<tr>
<td></td>
<td>(MPa)</td>
<td>(MPa)</td>
</tr>
<tr>
<td>min.</td>
<td>450</td>
<td>650</td>
</tr>
<tr>
<td>max.</td>
<td>-</td>
<td>880</td>
</tr>
</tbody>
</table>

Contrary to the requirements to the level of mechanical properties of forgings made of steel X2CrNiMoN2253 according to the standard EN10088-3 the customers for offshore oil and gas applications require also evaluation of steel toughness at the temperature of −46°C. These conditions were therefore defined as the basic criterion for evaluation of successful production from the viewpoint of achievement of the required mechanical properties.

Forging ingot of the type 8K1.7 with the mass of 1390kg served as input blank for manufacture of the experimental forging. The ingot was processed by smith forging in the temperature interval from 1150 to 950°C. Technology comprised stamping and forging to the dimension with an allowance for machining. The achieved deformation ratio was 5.6. After completion of the forming process the forging was cooled on air. The processing followed by machining and heat treatment – solution annealing. The standard EN10088-3 specifies for solution annealing the temperature interval from 1020 to 1100°C. The experimental forging was heat treated at the bottom temperature of solution annealing equal to 1020°C with subsequent cooling in water.

Evaluation of mechanical properties and structure of the forging was made at the places distant from the frontal of the forging in the depth of ½ and ¼ of the forging diameter in longitudinal and transverse directions. Table 5 gives the obtained values of mechanical properties according to the place and direction of testing.

Table 4: Specification of mechanical properties of forgings made of duplex steel X2CrNiMoN2253 according customer specification

<table>
<thead>
<tr>
<th>-20°C</th>
<th>+20°C</th>
<th>-46°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Re</td>
<td>Rm</td>
<td>A</td>
</tr>
<tr>
<td>(MPa)</td>
<td>(MPa)</td>
<td>(%)</td>
</tr>
<tr>
<td>min.</td>
<td>450</td>
<td>620</td>
</tr>
</tbody>
</table>
Table 5: Mechanical properties - Forging made of steel X2CrNiMoN2253

<table>
<thead>
<tr>
<th>Direction</th>
<th>Tested position</th>
<th>20°C</th>
<th>-46°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Re (MPa)</td>
<td>Rm (MPa)</td>
<td>A (%)</td>
</tr>
<tr>
<td>Long</td>
<td>1/4T</td>
<td>554</td>
<td>702</td>
</tr>
<tr>
<td></td>
<td>1/2T</td>
<td>554</td>
<td>716</td>
</tr>
<tr>
<td>Trans.</td>
<td>1/4T</td>
<td>570</td>
<td>724</td>
</tr>
<tr>
<td></td>
<td>1/2T</td>
<td>554</td>
<td>727</td>
</tr>
</tbody>
</table>

It follows from table 5 that satisfactory strength properties of the experimental forging were achieved at both places and directions of testing according to the specification given in table 4. Yield point and ultimate strength surpasses substantially the required minimal values specified both on the standard EN100088-3 and in the customer material specification. The values of impact energy at the testing performed at the temperature of -46°C satisfy the customer requirements.

Other qualitative parameters of the forgings made of duplex steel comprise evaluation of structure and determination of the share of ferritic phase. The structure may not show presence of inter-metallic phases.

Analysis of structure of the forging and share of ferrite was performed at the place corresponding to a ½ diameter of the forging. Fig. 7 and fig. 8 documents the structure of the experimental forging.

![Fig 7: Structure of the forging made of duplex steel - 1/2 T – 100x](image1)
![Fig 8: Structure of the forging made of duplex steel - 1/2 T – 500x](image2)

Image analysis performed with use of LECO IA32 showed that the two-phase ferritic-austenitic structure contained 50.0 % share of ferritic phase.

Results of measurement thus confirmed the possibility of production and of achievement of the required mechanical and structural properties large forged products formed by smith forging from duplex steel X2CrNiMoN2253 according to the EN 10088-3 and satisfying the customers' specifications for applications in conditions of offshore oil and gas.

5. LABORATORY ANALYSIS OF INTER-METALLIC PHASES IN DUPLEX STEEL X2CRNIMON2253

Development of technology for production of duplex steel X2CrNiMoN2253 forgings concerned also the issues related to occurrence of inter-metallic phases (χ, σ phase). The principle objective of the
experiment consisted in artificial creation of inter-metallic phase in two-phase ferritic-austenitic structure in order to obtain thus knowledge about state of material in case of unsatisfactory structure of duplex steel.

Conditions for formation of inter-metallic phases in original satisfactory ferritic-austenitic structure of the forging made of experimental melt with the chemical composition given in table 2 consisted in heating of the sample of duplex steel with dimensions of 25x25x25mm to the temp. of 750°C with a dwell of 48 hours with subsequent cooling on air.

Afterwards metallographic analysis of structure was made with focus on quantitative and qualitative evaluation of inter-metallic phases. Micro-structure was developed with use of the agent Beraha II + K$_2$S$_2$O$_5$. Fig. 9 and fig. 10 shows the resulting structure.

As it is evident from Figures 9 and 10, individual phases are sufficiently contrast, inter-metallic phases (χ-phase, σ-phase) have white hue at use of the agent Beraha II + K$_2$S$_2$O$_5$, ferrite is blue of brown, and austenitic grains are grey.

Crystallographic data on investigated sample were obtained by technique of Electron Backscattered Diffraction (EBSD), which is based on analysis of the Kikuchi's lines rising from the surface of strongly inclined sample in the SEM chamber. The EBSD analysis was used for determination of the share of individual phases from the area with dimensions of 400x200μm.

Figure 11 presents an illustration of a map from the EBSD analysis, from which the share of inter-metallic phases was evaluated. Values of the share of phases as determined by the EBSD analysis were the following: 12.5% of ferrite, 72.8% of austenite, 14.7% of inter-metallic phases.
In the next step structure of steel was evaluated in the area of occurrence of inter-metallic phases with use of EDX analysis. Figure 12 documents appearance of structure and points of measurement used for determination of chemical composition.

Results of EDX analysis in table 6 document significant deviations of chemical composition at the points of measurement in comparison with the chemical composition of steel as specified in table 2.

**Table 6:** Chemical composition of inter-metallic phases in duplex steel – EDX

<table>
<thead>
<tr>
<th></th>
<th>Cr</th>
<th>Mn</th>
<th>Ni</th>
<th>Mo</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spectrum 1</td>
<td>17.2</td>
<td>1.2</td>
<td>6.6</td>
<td>0.9</td>
<td>Rest.</td>
</tr>
<tr>
<td>Spectrum 2</td>
<td>16.6</td>
<td>1.6</td>
<td>6.6</td>
<td>1.4</td>
<td>Rest.</td>
</tr>
<tr>
<td>Spectrum 3</td>
<td>17.5</td>
<td>1.7</td>
<td>7.3</td>
<td>2.0</td>
<td>Rest.</td>
</tr>
<tr>
<td>Spectrum 4</td>
<td>21.1</td>
<td>1.2</td>
<td>1.7</td>
<td>0.8</td>
<td>Rest.</td>
</tr>
</tbody>
</table>

Occurrence of inter-metallic phases distributed on the austenitic grain boundary and touching the ferritic grain changes principally character of duplex steel structure. In connection with the occurrence of inter-
metallic phases (χ-phase, α-phase), chemical composition of which is similar, based on presence of iron, chromium and molybdenum, it is possible to assume a substantial drop of service properties of the final product, particularly of mechanical properties and resistance to corrosion.

6. CONCLUSIONS

Experimental works focused on possibilities of production of smith forged forgings made of duplex steel in conditions of the company ŽĎAS, Inc. proved successful fulfilment of the requirements to mechanical properties of the final forging made of the duplex steel X2CrNiMoN2253 according to the EN 10088-3. Satisfactory two-phase structure of large smith forged product made of duplex steel was also achieved thanks to observation of specific conditions for forming and heat treatment. The works performed in the area of analysis of inter-metallic phases in duplex steel contributed to broadening of knowledge about appearance of structure of inappropriately processed blank. On the basis of documentation of results it is possible to make in industrial conditions metallographic evaluations of structure of forgings with stress on control of occurrence of inter-metallic phases. Qualification of ŽĎAS, Inc. for the market segment with products made of duplex steels requires respecting of conditions of existing customers and assumes continuation of collaboration with universities and research working sites.

ACKNOWLEDGMENTS

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LITERATURE

[1] EN10088-3 Stainless steels– Part 3: Technical delivery conditions for blanks, bars, wires, iron sections and polished products made of stainless steels for general use

