DIROS 500 advanced quenched and tempered steel for pressure vessels.

R. Cawelius, M. Bockelmann, G. Luxenburger, P. Wolf, Dillinger Hütte, Germany present the properties of the steel grade and their advantage in the use for pressure vessels compared to conventional steel grades.

The national European codes for the construction of pressure vessels use few quenched and tempered steel grades with minimum specified yield strength over 355 MPa, except for the steel grades for higher temperature service (e.g. CrMo steels). In contrast to that the application of the ASME code makes the use of high strength steels for the construction of pressure vessels possible in a larger extend. E.g. the quenched and tempered grade SA 537 Cl 2 reduces the wall thickness remarkably compared to the normalised SA 537 Cl 1 or SA 516 Grade 70 grade. Also other grades like the quenched and tempered grades SA 517, 533, 543, can be applied. However since February 1997 the new European Standard EN 10028-6 defines a family of quenched and tempered fine grained steels for pressure vessel construction. Here the minimum specified yield strength is ranging between 355 MPa (P355Q) and 690 MPa (P690Q). The future European harmonised standard EN 13445 for construction of pressure vessels is still under construction but within the publication of the standard the use of these steels for pressure vessels will also come up in Europe.

On the other hand over the last decade the use of pressure vessels on offshore platforms - typical applications are separators and scrubbers - created the demand for lighter constructions. The most important requirements for the development of such high strength steel in this field of application was an optimised combination of tensile properties and the high toughness level at lower temperatures. That was the basis for Dillinger Hütte GTS to develop the DIROS 500 (DIROS = Dillinger Offshore Steel for Pressure Vessels) steel type. According to the special additional requirements to respect sour service conditions two sub-grades were defined: DIROS 500 HT (High Tensile) with a minimum specified yield strength of 500 MPa and DIROS 500 S (Sour gas) with a minimum specified yield strength of 460 MPa. The pressure vessels made of DIROS 500 are successfully in service on offshore platforms, mostly designed according British PD 5500 with supplementary requirements as per engineering specifications. Nowadays also the European Pressure Equipment Directive is used; in this case a particular material appraisal shall be applied.

Figure 1: Offshore separator made out of DIROS 500 S (with the kind consent of Hans Leffer GmbH): Gross weight 114400 kg, wall thickness 94 mm
Definition of the DIROS 500 steel series

DIROS 500 is a vacuum treated quenched and tempered fine grained steel. Due to its production route and its chemical composition DIROS 500 has excellent mechanical properties combined with good weldability. The DIROS 500 is an enhanced grade with certain similarities with P500QL2 of EN 10028-6 and ASTM/ASME A/SA 533 Type B or C. This steel is normally produced in a thickness range from 10 to 130 mm. In contrast to the European grades the specified minimum strength properties are constant for the defined thickness range.

DIROS 500 is used in two sub-grades:

- **High tensile strength grade** (HT) with minimum tensile strength of 600 MPa and a minimum yield strength of 500 MPa
- **Sour service grade** (S) with minimum tensile strength of 580 MPa and a minimum yield strength of 460 MPa fulfilling NACE MR 0175

<table>
<thead>
<tr>
<th>grade</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>N</th>
<th>Al</th>
<th>Cu</th>
<th>Mo</th>
<th>Ni</th>
<th>Cr</th>
<th>CEV</th>
<th>Pcm</th>
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<tbody>
<tr>
<td>HT</td>
<td>≤ 0.13</td>
<td>≤</td>
<td>1.00</td>
<td>≤</td>
<td>≤</td>
<td>≥</td>
<td>≤</td>
<td>≤</td>
<td>≤</td>
<td>≤</td>
<td>0.64</td>
<td>≤ 0.28</td>
<td></td>
</tr>
<tr>
<td>S</td>
<td>≤ 0.11</td>
<td>0.40</td>
<td>- 1.60</td>
<td>0.012</td>
<td>0.002</td>
<td>0.01</td>
<td>0.015</td>
<td>0.30</td>
<td>0.60</td>
<td>&lt; 1.00</td>
<td>0.60</td>
<td>≤ 0.60</td>
<td>≤ 0.26</td>
</tr>
</tbody>
</table>

\[
V+Nb \leq 0.01 \%
\]

\[
CEV = C + \frac{Mn}{6} + \frac{(Cr + Mo + V)}{5} + \frac{(Cu + Ni)}{15}
\]

\[
Pcm = C + \frac{Si}{30} + \frac{(Mn + Cu + Cr)}{20} + \frac{Ni}{60} + \frac{Mo}{15} + \frac{V}{10} + 5B
\]

**Table 1:** Chemical composition according to heat analysis [%]

**Parent material properties**

The DIROS 500 mechanical properties defined in the material specification DH-E07-B of Dillinger Hütte are achieved, in respect to the requested plate thickness and post weld heat treatment (PWHT) condition, by an adapted chemical composition and optimised tempering conditions. The influence of heat treatment on mechanical properties can be expressed by the so called Hollomon-Parameter as a combination of tempering and PWHT /1/.

\[
HP = T_t \times \left( 20 + \log \left( t_t + 10 \left( \frac{T_{PWHT} \times (20 + \log t_{PWHT})}{T_t} \right)^{20} \right) \right) \times 10^{-3}
\]

with:  
\[
T_t = \text{tempering temperature [K]}
\]
\[
t_t = \text{tempering holding time [h]}
\]
\[
T_{PWHT} = \text{PWHT temperature [K]}
\]
\[
t_{PWHT} = \text{PWHT holding time [h]}
\]
The tensile test properties according to specification DH-E07-B are achieved as it is shown in figure 2 and 3. Due to the adapted design of tempering condition and chemical composition the expected decrease of yield- and tensile strength with increasing HP is minimised in the given thickness range. E.g. in case of
strong PWHT conditions the tempering temperature is reduced. This is the reason why Dillinger Hütte aims to agree the heat treatment of the plates individually with the client from case to case. Even the values in the vicinity of the minimum specified yield- and tensile strength are specifically the result of the individual design for the pressure vessel.

Hot tensile strength is often a design criterion for these pressure vessels. Yield strength values at elevated temperatures are often requested by the manufacturer e.g. if semi-cold forming (hot forming at temperatures lower than tempering temperature) is applied. Figure 4 indicates the hot tensile properties for DIROS 500 S.

Figure 4: Yield and tensile strength at elevated temperatures
DIROS 500 is used under many different service conditions which lead to considerable differences in requirements concerning impact toughness. Figure 5 shows the transition behaviour for DIROS 500 HT in a plate thickness of $t = 120$ mm with a HP of 18.3. The two curves (for quarter and mid thickness test location) show that up to -50 °C (normal test temperature of the data sheet) the values are still higher than the minimum specified single value of 35 J. Also it can be seen that the mid thickness toughness for temperatures especially lower -60 °C is lower than in quarter thickness due to typical cooling speed and segregation effects. In so far especially for very high thickness and increasing HP values the mid thickness properties can be one of the limiting factors in the requirement package.

**Forming**

**Hot forming**

Hot forming is applied for head manufacturing of DIROS 500 for plate thickness above 30 mm. A circular blank will be first heated to a temperature of about 1000 to 1050 °C in case of several forming steps or to about 950 °C in case of a single forming step. As the steel grade DIROS 500 is a quenched and tempered steel grade the heads and the segments respectively shall also be quenched and tempered after the final forming operation. The hot forming procedure and the subsequent quenching and tempering of DIROS 500 HT are not significantly different to DIROS 500 S.

The figures 6 to 9 show the consistency of the mechanical properties from initial plates and final products. The investigated specimens of the dished heads have been taken from the straight flange to meet the area with highest possible change in the material properties. The specimens of the multi segment heads have been taken from an excess length also after final forming and heat treatment. All forming steps have been performed in the heavy fabrication division of Dillinger Hütte GTS.
Figure 6: Comparison between tensile properties of initial plate and finished heads.

Figure 7: Comparison between impact toughness properties of initial plate and finished heads.
It is evident that an appropriate forming and heat treatment procedure does not impair the mechanical properties of the material.

Cold forming
On the other hand cold forming is the preferred forming process for the shell courses if bending facilities enable it. The following figures 10-12 show the influence of cold forming on the mechanical properties. As expected figure 10 indicates that the yield strength increases with an increased degree of cold deformation. The tensile strength increases also, but not as intensive as the yield strength. So the yield to tensile strength ratio increases and reaches about 1 after a cold-forming degree of 10%. A subsequent PWHT restores the yield and tensile strength approximately to the initial level.

Figure 10: Influence of cold deformation on yield and tensile strength
Figure 11: Influence of cold deformation on elongation without necking

Tensile tests after certain cold deformation rates show a similar behavior of the elongation values without necking in figure 11. After 10% of cold deformation the elongation in the tensile test is near to nil. That means after this deformation rate necking will appear more or less at once. But a subsequent PWHT after cold deformation restores the elongation behavior to the initial level.

The influence of a cold forming on the toughness is not so evident, particularly in case of quenched and tempered steel grades. On plate material of DIROS 500 S the influence of a cold deformation on the toughness behavior is shown in figure 12.
Figure 12: Transition curves of DIROS 500 S after different cold deformation with and without PWHT

The transition temperature is shifted to a higher temperature by the increasing cold deformation and a subsequent PWHT shifts the transition temperature furthermore. This tendency is observed as well in the location \( \frac{1}{4} \) thickness as in \( \frac{1}{2} \) thickness. One could expect that the shift of the transition curve after cold deformation will be partly restored after a subsequent PWHT but this depends on the initial tempering and the related toughness level. If the maximum toughness has been reached due to an optimized tempering, a further increase or a restore respectively of the impact toughness will not be possible. In contrast to the tensile properties (s. figure 11) the lost in impact toughness after cold deformation will not be restored by PWHT. The test results show in any case that the steel grade DIROS 500 has an excellent toughness even after cold forming, above the specified minimum value.

**Welding**

In general DIROS 500 has an excellent weldability due to an optimized chemical composition and the production route of this steel (blast furnace BOF converter route with subsequent vacuum degassing). To meet the required properties of the welded joint the German Stahl Eisen Werkstoffblatt SEW 088 and the publication No 2 of the European Community of Iron and Steel (ECIS) can be used as basic recommendation for the processing of these steel grades. But at present these papers are replaced by the European standard EN 1011 "Welding - Recommendations for metallic materials".

Some examples of welding consumables appropriate for the welding (ReH ≥ 500 MPa after PWHT) of the steel grade DIROS 500 are listed in this table 2.
Shielded metal arc welding using covered electrodes

<table>
<thead>
<tr>
<th>trade name</th>
<th>DIN 8529</th>
<th>AWS</th>
<th>manufacturer</th>
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<tr>
<td>Tenacito 65</td>
<td>ESY 5576Mn1NiMoBH5</td>
<td>E 9018-G</td>
<td>Oerlikon</td>
</tr>
<tr>
<td>Tenacito 70</td>
<td>EY 5075 Mn1NiB</td>
<td>E 8018-G</td>
<td>Oerlikon</td>
</tr>
<tr>
<td>SH Schwarz 3KnNi</td>
<td>EY 5065 NiMoBH5</td>
<td>E 8018-NM</td>
<td>Thyssen</td>
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<tr>
<td>OK 73.46</td>
<td>EY 5575 Mn1.5NiMoB</td>
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<td>Esab</td>
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</table>

redried acc. to manufacturer's indication

Submerged arc welding

<table>
<thead>
<tr>
<th>wire</th>
<th>flux</th>
<th>manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluxocord 41 also available with Ni ≤ 1.0% for sour gas application (41.1 mod)</td>
<td>OP 121TT</td>
<td>Oerlikon</td>
</tr>
<tr>
<td>OE-SD3NiMo1</td>
<td>OP 121TT</td>
<td>Oerlikon</td>
</tr>
<tr>
<td>OE-SD3NiMo1</td>
<td>OP 41TT</td>
<td>Oerlikon</td>
</tr>
<tr>
<td>Union S3NiMo</td>
<td>UV 121TT</td>
<td>Thyssen</td>
</tr>
<tr>
<td>OK Autrod S2NiMo1</td>
<td>OKFlux 10.61</td>
<td>Esab</td>
</tr>
<tr>
<td>OK Autrod 13.40 (S3NiMo1)</td>
<td>OKFlux 10.62</td>
<td>Esab</td>
</tr>
</tbody>
</table>

Gas shielded metal arc welding

<table>
<thead>
<tr>
<th>wire</th>
<th>shielding gas</th>
<th>manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluxofil 41</td>
<td>CO2/mixed gas M21 acc. DIN 32525</td>
<td>Oerlikon</td>
</tr>
</tbody>
</table>

Table 2: Examples of welding consumables

If DIROS 500 S shall be used under sour-gas service as per NACE MR 0175 the restricted chemical analysis by Ni ≤ 1.0% and hardness of max 22 HRC respectively in the base metal, weld metal and heat affected zone shall be regarded. Using conversion tables of hardness values evaluated by different test methods, e.g. ASTM E 140-95 the hardness value of 22 HRC is equivalent to 248 HV10. By converting these values the advise of the relevant standard shall be regarded. ASTM E 140 gives as a guidance that a conversion of hardness values is only applicable in case of homogeneous metallurgical structures. Figure 13 shows an example of an hardness profile over a submerge arc butt weld according W13 rev. o in which the fine and coarse grain heat affected zone are distinguished. It can be seen that the scattering in the fine grained and coarse grained heat affected zone is more than twice the scattering of the values in the base metal and in the weld metal respectively. This is the consequence of the fact that the heat affected zones do not have an homogeneous structure as per ASTM E 140 and that the hardness values were evaluated by the Vickers 10 method. The diameter of the indentation using HV 10 have been approximately 0.23 mm, different microstructures with different hardness have been met. In contrast using the Rockwell C method the diameter of the indentation would be about 0.75 mm. These values are more integrated values over the different microstructures in coarse and fine grained heat affected zone, schematically indicated by the dotted black line in figure 13. Nevertheless it is possible to meet 248 HV10 on DIROS 500 S but due to the larger scattering of HV10 method, single values could more easily exceed the maximum allowable value. In a worst case this could lead to an unnecessary delay of delivery resulting mostly in increased costs. Additional, scattering of the hardness values in the microstructures smaller than the diameter of the indentation using HRC will not affect the stress distribution or any crack propagation. For this reason we recommend our customers to inquire maximum hardness value only according NACE MR 0175 and 22 HRC respectively.
Of course not only the hardness restriction shall be met but also the tensile and toughness requirements. To assure that all of the specified requirements are met it is strongly recommended to discuss all the requirements and the fabrication and welding parameters with the material manufacturer. So he can check whether it is possible to meet all the parameters or to propose the best compromise. Further more fracture mechanics investigations on DIROS 500 at -20 °C have shown excellent results.

**Conclusion**

High strength quenched and tempered steel grades are more and more used in the fabrication of highly sophisticated pressure vessels. DIROS 500 is one of these modern steel grades. The report should inform you that is possible to meet the requirements specified by national rules and standards and specifications established by the end-users, e.g. Oil and Gas-Companies or engineering companies. Efforts in research are ongoing, e.g. in the research programs ECOPRESS, sponsored by the European Community, or a national research programme in the Netherlands to get further progress in the use of these modern steel grades and to assure their economical and safe use in pressure vessel technology.

**References**

/1/ I. Detemple, A. Demmerath; The Effects of Heat Treatment,