Production of Large Diameter Pipes Grade X 70 with High Toughness using Acicular Ferrite Microstructures.

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Abstracts
It is well known that a low carbon bainitic microstructure (acicular ferrite) offers an excellent combination of strength and toughness, especially when formed from non-recrystallised austenite. This was demonstrated in a recent large diameter onshore pipeline project requiring over 100,000 tonnes of X70 pipe with wall thickness of 17.5 to 26.2mm. In addition, exceptionally high toughness requirements were also imposed for the base metal and the weld. The requirements were met by using a thermomechanically rolled plus accelerated cooled plate of the following composition: 0.05% C, 1.70% Mn, 0.075% Nb. The steel was Ti/N treated with no further additions of other alloying or microalloying elements. The metallurgical concept of this alloy design is reviewed and the production data of the final pipes with regards to the strength, toughness and weld properties are presented.

Introduction:

A recent onshore pipeline project asked for longitudinal double submerged arc welded pipes of 40” diameter and wall thickness of 17.5, 21.0 and 26.2mm respectively, being about 1% cold expanded. Besides requirements in the chemical composition and the cleanliness of the steel it was especially demanded that the steelmaking includes vacuum degassing, calcium and micro-titanium treatment and the processing of the plates follows the thermo-mechanical control process (TMCP).

The demands in mechanical properties of the pipes followed the API specification for grade X70 (YS > 485MPa, TS > 570MPa) and also the tensile strength of the weld had to match this minimum tensile strength of 570MPa. Relatively severe demands existed in the toughness of the pipes, which asked for > 85% shear area in the BDWT test at –5°C (each individual test > 70%) and absorbed energy in the Charpy-V-notch test at –20°C transverse pipe of > 190J (each individual test > 140J) and for the weld metal or heat affected zone of > 90J (each individual test > 60J).

The experience of the involved companies would have allowed to guarantee this property combination with thermo-mechanically (TM) rolled plates exhibiting a conventional microstructure of ferrite plus pearlite, but it was also demanded, that the pipes exhibit an ‘acicular ferrite’ microstructure.
Definition of Acicular Ferrite and Application in Line Pipes

In the literature the term ‘acicular ferrite’ is used for two different kinds of microstructures:

a) Generally the needle like pro-eutectoid ferrite is referred to as Widmanstätten ferrite. These ferrite side-plates are usually formed at grain boundaries. But the nucleation can be also intra-granular, e.g. at inclusions, and for this kind of Widmanstätten ferrite, a common mean to toughen the weld metal, the term acicular ferrite has been used (1).

b) Most of the publications referring to acicular ferrite consider a bainitic transformation (2) and the term bainitic ferrite is also used for this microstructure, which is carbide-free and thus different to upper or lower bainite.

Low carbon – manganese – molybdenum – niobium steels with a microstructure of bainitic ferrite have been produced with IPSCO already in 1971/72 and were successfully applied by the TransCanada PipeLine Company Ltd. for a major project (3). The addition of about 0.35% Mo to these steels of about 0.06% C, 1.75% Mn and 0.05 to 0.09% Nb was mandatory in order to restrict major formation of polygonal ferrite after air cooling of plates (4). Metallographic investigations confirmed, that the microstructure was not a classical bainite, which should consist of ferrite needles and cementite, but was a mixture of bainitic ferrite with its high dislocation density and carbon enriched martensite regions, mainly existing as interlath films and - to a minor extent - also as discrete islands. Sometimes the martensite phase could be detected already by optical microscopy but a clear picture needed transmission electron microscopy investigations (5). Figure 1 shows a typical CCT diagram of such steel. These carbon enriched martensite islands are formed at slower cooling rates. Such local brittle phases naturally limiting the toughness of the steel, thus a reduction of the carbon content itself is recommended for optimum property combinations (6).

![CCT Diagram of acicular ferrite pipe steel](image)

Figure 1: CCT Diagram of acicular ferrite pipe steel

A consequent lowering of carbon content has been demonstrated with the ULCB (Ultra Low Carbon Bainitic) steels and it was found, that even with levels <0.04% C further improvement of the impact energy can be achieved (7). In ULCB steel with max. 0.03%
carbon the molybdenum content was (partially) substituted by boron and/or nickel, dependent on the aimed for strength level of X65 to X80. Even though excellent property combinations have been reported for the base metal and the weldment (submerged arc weld and the girth weld as well), the application of ULCB steel was rather limited. Besides possible problems related to proper boron control during steelmaking, another inherent problem in very low carbon steel is a local interstitial-free status and thus the danger of intergranular fracture (8).

A more economic production of pipe steel with partial bainitic microstructure became possible with the introduction of accelerated cooling after thermomechanical rolling, even without any molybdenum alloying. Thanks to the finer grain size and the higher dislocation density of the bainite compared to polygonal ferrite, this microstructure lead to X 80 properties (9).

Figure 2 underlines the characteristics of bainitic ferrite compared to polygonal ferrite, offering besides higher strength also improved toughness owing to its finer grain size.

![Figure 2: Characteristics of bainitic ferrite and polygonal ferrite](image)

Accelerated cooling and producing a bainitic ferrite microstructure in low carbon steel became also mandatory for the production of pipe steel resistant against hydrogen induced cracking HIC (10). Besides the indispensable requirements in low sulphur contents plus sulfide shape control and low primary segregation in these steels, accelerated cooling helps to avoid secondary segregation during the ? to a transformation and thus a microstructure without any pearlite bands is obtained, which promote HIC propagation.

Like in steel relying on a ferrite plus pearlite microstructure also in all pipe steels with an acicular ferrite microstructure, more intensive thermomechanical rolling before the ? to a transformation leads to better toughness. It is correlated with a refinement of the bainitic ferrite lath as a result of dislocation cell structures within the deformed grains (11).
The Role of Niobium

When the amount of solute niobium is increased, retardation of austenite recrystallisation is observed at significantly higher temperatures, Figure 3 (12), thereby allowing the benefits of thermomechanical rolling to be achieved at higher temperatures. Low carbon contents and the fixing of nitrogen with titanium, an element with higher affinity for nitrogen than niobium, prevent niobium carbonitride formation and allow the higher niobium content to be easily dissolved during reheating of the slabs, Figure 4 (13,14). Such steels having less than 0.05 percent carbon, about 0.09 percent niobium and Ti/N treatment have specifically designed for high temperature processing (HTP) and are often referred to as HTP steel (15). Since the retardation of austenite recrystallization occurs already at relatively high temperatures, this concept allows a high deformation in the non recrystallization region of austenite, when relying on a usual thermomechanical rolling regime.

Figure 3: Retardation of recrystallisation by microalloying

Figure 4: Solubility of niobium carbide and carbonitride respectively
It can be taken for granted that in low carbon, titanium treated steel a typical reheating temperature of 1150° C or higher is sufficient to dissolve all NbC precipitates in the slab. Thus all the niobium will be in solid solution at the commencement of rolling. During austenite conditioning a portion of the niobium will be precipitated as strain induced carbide on dislocations. Using chemical extraction techniques described elsewhere (16), the undissolved and the strain induced austenite precipitates remain in the filter residue, while niobium in solid solution and the fine ferrite precipitates will be dissolved. This technique allows one to investigate the status of niobium at the finish rolling temperature.

Results of chemical extraction studies for a conventional pipe steel and a ‘HTP’ steel in relation to the equilibrium condition are shown in Figure 6. Conventional pipe steel, processed on a plate mill with a typical finish rolling temperature below 800° C, allows only a small portion of niobium to remain in solid solution and the amount of ‘soluble’ niobium is close to the equilibrium condition. A higher ‘soluble’ niobium content of almost 0.02 percent is observed in hot strip, where the final deformation steps are continuous and feature higher rolling speeds and shorter interpass times as well as a finish rolling temperature about 100 degrees Celsius higher than for plate rolling. In contrast the HTP steel shows a ‘soluble’ niobium content as high as 0.04 percent for typical finish rolling temperatures in a plate mill, with even higher values for hot strip processing.

Figure 6: Niobium in solid solution at different finish rolling temperatures for two pipe steels (determined by the chemical extraction method)

Niobium in solid solution at the finish rolling temperature is available for the formation of niobium carbide precipitates in ferrite, which have the appropriate size for additional strength increase via precipitation hardening. This strength increase amounts to about 100MPa for 0.03% niobium in solid solution at finish rolling temperature, when the particle size is 1.5 to 2nm (17). Additionally, niobium in solid solution has also an effect in reducing the α to a transformation.
The effect of microalloys on the transformation behaviour of HSLA steel at various cooling rates has been quantified elsewhere (18): Figure 6 confirms, that in comparison to the two other commonly used microalloying elements vanadium and titanium, solute niobium has the strongest effect in lowering the transformation temperature, and this effect gets even more pronounced with a higher cooling rate. This has been demonstrated already in an earlier literature review, evaluating the results of various investigations (19).

The metallurgical explanation can be given by comparing the atom radii of alloying elements, since all diffusion controlled processes such as the $\gamma$ to $\alpha$ transformation are retarded more effectively, the bigger the difference in the atom size of the base element compared to any other element in solid solution is. The niobium atom is 15.6% bigger than iron, while the difference of the two other microallying elements titanium or vanadium is lower, +14.8% and +6.2%, respectively. Since the difference in the atom size of molybdenum to iron, +9.4 %, is also much lower than that of niobium, the much higher effectiveness of solute niobium compared to molybdenum to increase hardenability becomes obvious.

Lowering of the transformation temperature effects the microstructure of thermomechanically rolled plus accelerated cooled plates as shown in Figure 7: it refines the grain size of polygonal ferrite and increases the volume fraction of bainite (18).
Trial Production

Considering the specification and having the metallurgical fundamentals and the in mind, an acicular ferrite microstructure asks for low carbon content of max. 0.06%, in order to guarantee that no toughness deterioration by hard martensite phases occurs. An economic production of < 0.06% carbon HSLA steel limits the manganese content to about 1.70% and also the specification asked for this level (max. 1.75% Mn).

However, even when using accelerated cooling after thermomechanical rolling one obtains only a partial acicular ferrite microstructure. Furthermore, such partial bainitic microstructure of steel with max. 0.06% C and 1.70% Mn cannot guarantee the strength level of thick wall X70, either. Therefore other elements, assisting to increase hardenability and adding to further strength increase by solid solution or precipitation hardening have to be alloyed. Within trial production of pipe plate with the thickest wall specified, i.e. 26.2 mm, the most promising alloy addition has been evaluated. The three tested variants were:

1. addition of Cu+Ni
2. addition of Mo
3. a higher niobium content

All steels had < 0.003% S, < 0.020% P and calcium treatment. The exact chemical composition of the investigated steels is given in Table 1, indicating also that a proper Ti/N treatment has been made.
The rolling schedule for all three variants was identical, i.e.

- reheat temperature: 1180 °C
- total final deformation: > 75 %
- finish rolling temperature: in the metastable austenite region
- accelerated cooling with about 12 °C/s to about 550 °C

All plates fulfilled the specified strength and toughness requirements, even when considering a certain reduction in yield strength (Bauschinger effect) as well as in toughness (the BDWTT was carried out at –15 °C, i.e. 10 degrees lower than specified for the pipe) as result of pipe forming process. Even though the carbon content is relatively low, the bainitic microstructure results in tensile strength values surpassing the specified minimum value of > 570MPa.

<table>
<thead>
<tr>
<th>Type</th>
<th>%C</th>
<th>%Si</th>
<th>%Mn</th>
<th>%Cu</th>
<th>%Ni</th>
<th>%Mo</th>
<th>%Nb</th>
<th>%Ti</th>
<th>%N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-Ni</td>
<td>0.06</td>
<td>0.25</td>
<td>1.68</td>
<td>0.17</td>
<td>0.19</td>
<td>0.045</td>
<td>0.018</td>
<td>0.005</td>
<td></td>
</tr>
<tr>
<td>Mo</td>
<td>0.06</td>
<td>0.25</td>
<td>1.68</td>
<td></td>
<td>0.16</td>
<td>0.045</td>
<td>0.018</td>
<td>0.005</td>
<td></td>
</tr>
<tr>
<td>Nb+</td>
<td>0.05</td>
<td>0.25</td>
<td>1.68</td>
<td></td>
<td>0.16</td>
<td>0.045</td>
<td>0.018</td>
<td>0.005</td>
<td></td>
</tr>
</tbody>
</table>

Table 1: Chemical composition and mechanical properties of preliminary trials for process optimization.

<table>
<thead>
<tr>
<th>Type</th>
<th>Re in MPa</th>
<th>Rm in MPa</th>
<th>Impact Energy at -40°C in J</th>
<th>CVNBDWTT at -15°C in % shear area</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-Ni</td>
<td>545</td>
<td>625</td>
<td>460</td>
<td>85</td>
</tr>
<tr>
<td>Mo</td>
<td>550</td>
<td>640</td>
<td>405</td>
<td>90</td>
</tr>
<tr>
<td>Nb+</td>
<td>535</td>
<td>610</td>
<td>465</td>
<td>95</td>
</tr>
</tbody>
</table>

Figure 8 confirms that all plates exhibit an acicular ferrite microstructure, as aimed for. By comparing the three alternatives, the variant with the higher niobium content shows the finer grain size and is more homogeneous. This observation corresponds with the fact, that the toughness is also slightly better, which can be traced back to niobium’s effect in retarding austenite recrystallization. As already mentioned, the retardation of austenite recrystallization occurs already at a higher temperature with higher niobium content and the more intensive austenite processing is especially helpful with thicker plates. Furthermore, the 0.03% higher niobium content guarantees also, that the microstructure becomes almost totally bainitic, thus the addition of higher amounts of alternative and commonly used alloying elements such as molybdenum can be avoided.
Figure 8: Microstructure of 26.2 mm TMCP plates of different alloy concept
Production Data

Following the described Nb+ alloy concept, with no additions of Cu, Ni, Mo, V or others and using the same processing parameters as described above, plates in 17.5 and 26.2 mm have been produced in two different steel companies, each one comprising its own oxygen steel making and plate rolling mill. These plates were transformed into longitudinal welded pipes of 1016.0 mm outer diameter by the UOE process.

The frequency curves of the production data obtained from the thicker and more demanding dimension 26.2mm are given in Figure 9 (yield and tensile strength, testing direction: transverse pipe) and Figure 10 (Charpy impact energy and BDWTT shear area, Testing direction: transverse pipe, testing temperature: –20° C and –5° C, respectively).

![Figure 9: Tensile Properties of 1016,0mm O.D. x 26.20mm W.T. X70 pipes (72 test results), base material, transverse pipe](image)

![Figure 10: Charpy impact energy (222 test results) and BDWTT shear area (126 test results) of 1016,0mm O.D. x 26.20mm W.T. X70 pipes, base material, transverse pipe](image)
Especially impressive is the high impact toughness, which surpasses the specified value of > 140J by a factor of three, but also the yield to tensile strength ratio, which was typically below 0.85, is remarkable low for such low carbon steel.

The microstructure in the heat affected zone (HAZ) of such low carbon steel - relatively high manganese steel is also bainitic. Thus also at this position excellent toughness can be expected as result of the fine effective grain size and the absence of local brittle phases. Figure 11 shows that no deterioration of excellent data from the base material occurred, confirming earlier results (16), except for a few data, where the HAZ had been slightly missed. In order to obtain good toughness in the weld metal, one has to take care, that also in this dendritic structure any hard constituents are avoided and the dissolution of about 50% of the base metal can be tolerated. Thus also low carbon consumables are needed, using alloying elements such as boron to promote transformation into bainite. Within this order the chemical composition of the weld metal was about:

0.06 %C, 1.75 %Mn, 0.04 %Nb, 0.02 %Ti and 20 ppm B

and Figure 11 shows that the specified minimum impact energy value of 60 J was also obtained at this position.

![Figure 11: Charpy-V-notch impact energy in the heat affected zone (225 test results) and the weld metal (279 test results) of 1016.0 mm O.D. x 26.20 mm W.T. X 70 pipes](image)

**Summary and Conclusion**

A thick wall onshore pipeline project X70 asked for high toughness and a microstructure of acicular ferrite. Bainitic microstructures offer excellent property combinations, when the carbon content is below 0.06%, thus avoiding the formation of hard and brittle constituents.
Even when applying accelerated cooling after thermomechanical rolling, a steel with 0.06% carbon and 1.7% manganese is not sufficiently alloyed to guarantee an almost fully bainitic microstructure and the specified strength. Therefore additions of elements adding to hardenability have to be made and it turned out, that a small increase in niobium from 0.045 to 0.075% results in the most homogeneous microstructure and relatively best toughness. This is also an economic solution since no further additions of elements like molybdenum or nickel are necessary.

All mechanical properties of the base material fulfilled the specification, especially the impact energy is outstanding. Also the toughness in the HAZ was excellent and also good weld metal toughness is achieved by using suitable consumables.

References

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