THE DEVELOPMENT OF NIOBIUM MICROALLOYED STEEL FOR THE PRODUCTION OF SEAMLESS TUBES

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1. Introduction

Seamless tubes are available with wall gages of up to 100 mm and outer diameters up to around 700 mm [1]. Such tubes are typically used for offshore pipelines and oil country tubular goods as well as for the automotive industry and construction applications. Due to weight and cost reduction efforts the demand for high strength grade seamless tubes is increasing. Many applications require high toughness in addition to high strength. The different rolling processes applied in production depend on wall gage and pipe diameter. The continuous mandrel mill is used to produce smaller gages and diameters; the plug mill covers medium gages and diameters; the pilger mill allows producing larger diameters and heavy wall gage.

The deformation and the temperature schedule during pipe rolling in seamless tube mills naturally depend on the process type. For all processes it is necessary however to apply a quench and temper treatment after rolling when a tube strength of X65 (min. yield strength of 448 MPa) or higher is to be produced.

The quench and temper treatment is usually an offline operation where the finished tube is heated above 920°C for 30 minutes to autenitize the steel. The hot tube is then quenched in water as shown in Figure 1 to obtain maximum hardness. In the tempering treatment the quenched tube is heated to above 640°C for one hour to reduce the as-quenched hardness and to increase toughness.

*Quench and temper treatment:*
- Austenitizing: $T > 920 \, ^\circ C$ for 30 minutes.
- Quenching: Rotary quenching unit.
- Tempering: $T > 640 \, ^\circ C$ for 60 minutes.

![Figure 1: Schematic representation of a typical quench and temper treatment for high strength seamless tube production.](image)

It is obvious that such a quench and temper treatment involves additional cost. Furthermore in a market situation with increasing demand for high strength tubes the quench and temper
treatment can become a bottleneck operation either reducing the output of a plant or requesting additional capital investment to increase the production capacity. It should thus be of high interest to find a metallurgical solution allowing to produce X65 pipe grade without having to apply a quench and temper treatment. The cost saving that could be achieved is estimated to around 60 $ per ton of tube produced. Besides the available quench and temper capacity could then be reserved for the production of premium grades such as X70 to X100.

In recent years the so-called HTP (high temperature processing) alloying concept, has been developed successfully by several steel producers [3, 4]. The HTP alloying concept is characterized by low carbon content (0.03-0.05%) and high niobium content (0.08-0.10%). The use of high Nb design meets the specifications and considerably reduces alloying costs when compared with other concepts. Meanwhile this alloying concept was successfully used in the production of pipe plate and strip. In several projects this steel was processed to longitudinal welded pipe as well as spiral pipe fulfilling API 5L specifications up to X80.

The current study investigated the possibility of applying the HTP alloying concept under seamless tube rolling conditions. Based on a typical rolling schedule of a continuous mandrel mill laboratory rolling simulations were carried out producing wall gages between 3 and 7 millimeters. The results shows that the strength level of X65 and under particular conditions even of X70 can be safely achieved without quench and temper treatment. The toughness of these steels was measured using Charpy V-notch testing at –20 C and revealed a very satisfactory level.

2. HTP alloy design

The HTP alloy design is based on a low carbon and a high niobium content as shown in Table 1. The manganese content can vary between 1.3 and 2.0% depending on the specific properties to be achieved. Besides manganese several other alloying elements such as chromium, copper, nickel, molybdenum and boron have been used in specific projects to adjust the material properties according to the processing conditions. Particular attention must be paid to the silicon content, which should be kept on a low level when the finished tube is to be galvanized.

Table 1: Chemical composition of the HTP steel for seamless pipe of API 5L X65 grade.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>N</th>
<th>Nb</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.04</td>
<td>0.25</td>
<td>1.70</td>
<td>0.015</td>
<td>0.002</td>
<td>0.025</td>
<td>0.005</td>
<td>0.100</td>
<td>0.015</td>
</tr>
</tbody>
</table>

Investigations have confirmed that the recrystallization stop temperature ($T_{NR}$) of the HTP alloying concept being approximately 1060°C is significantly higher than that of conventional microalloyed low carbon steel being around 950°C. This can be due to the precipitation of NbC pinning the austenite grain boundaries but also to the solute drag effect of Nb in solid solution.

If a substantial amount of solute Nb is still present after finish rolling, the transformation behavior will be influenced as well [2]. A microstructure consisting of polygonal ferrite and low carbon bainite will be formed even under the condition of slow cooling in air. According to the CCT diagram shown in Figure 2 the amount of bainite formed depends on the cooling
speed and also on the deformation below $T_{NR}$. Low carbon bainite often referred to as acicular ferrite is characterized by a high dislocation density and a small effective grain size. The presence of this phase in a fine-grained matrix of polygonal ferrite is responsible for the favorable combination of high strength and good toughness.

Figure 2: CCT diagram of HTP steel (soaking at 1250°C) [5].

3. Continuous mandrel mill process and laboratory simulation schedule

The continuous mandrel mill process that was taken as a basis for the present investigation consists of different rolling steps as schematically shown in Figure 3. The reheating temperature of 1260°C assures complete dissolution of all NbC precipitated in the round billet. The reheated billet is then hollowed in the piercing mill, elongated and further reduced in the continuous mandrel mill. The corresponding rolling temperatures are typically above $T_{NR}$ providing a fully recrystallized and homogeneous austenitic microstructure. After retraction of the mandrel the hollow is transferred to a reheating furnace. During that transfer the temperature drops to a level between 800 and 700°C. After reheating final rolling is done in the stretch reduction mill at a temperature below $T_{NR}$. This thermomechanical rolling pancakes the austenite grain providing a fine ferrite grain size after the $\gamma$ to $\alpha$ phase transformation.

Figure 3: Schematic process flow of a continuous mandrel mill process with typical temperatures and rolling deformations.

The process described above was translated into a laboratory-scale rolling schedule to analyse the HTP alloy concept under different temperature – deformation conditions. The simulation schedule shown in Figure 4 summarizes the different conditions. The entry temperature into the reheating furnace varied from 670°C being just above the ferrite start temperature (Figure 2) up to 890°C. Finish rolling was performed with deformations ranging from 1.5 to 4 times
below \( T_{\text{NR}} \) with finish rolling temperatures (FRT) between 800 and 890°C. Finish rolling was followed by air cooling.

![Simulation schedule of laboratory rolling conditions.](image)

**Figure 4:** Simulation schedule of laboratory rolling conditions.

Two samples were quenched in a water tank directly after roughing in order to evaluate the state of Nb precipitation at this phase of the process.

### 4. Results of laboratory rolling simulation

**Figure 5** demonstrates that the total amount of 0.11% Nb is fully dissolved at reheating temperature (RHT). Finish roughing (FR) is carried out at temperatures slightly below \( T_{\text{NR}} \). The two samples quenched after roughing were analyzed by chemical extraction to determine the amount of Nb precipitated. This analysis revealed that 0.08% Nb is still in solid solution at the quenching temperature of about 920°C. This quantity is considerably higher than the maximum solubility under equilibrium conditions. The precipitation kinetics, which depends on time and temperature may not be fast enough to form precipitates during processing. The precipitation kinetics attains its maximum rate at around 900°C. An increasing amount of manganese retards the Nb precipitation speed at all temperatures [5].

![Niobium in solid solution before and after roughing.](image)

**Figure 5:** Niobium in solid solution before and after roughing.
The material rolled to final gage after reheating was machined into tensile test specimens and Charpy V-notch samples. From the tensile curve the yield strength ($R_{t0.5}$) and the tensile strength ($R_m$) were determined. Both quantities are shown as a function of the total reduction during finish rolling and the finish rolling temperature in Figure 6. The data indicate that the original aim to reach API 5L X65 grade is clearly achieved. Under most of the simulated conditions however, even the production of API 5L X70 grade appears to be feasible. The correlations are as expected. With increasing final deformation the yield strength increases whereas it decreases with increasing finish rolling temperature.

The toughness was measured by Charpy V-notch testing at -20°C. The data points shown in Figure 7 are the average of three individual measurements for each rolling condition. The data population shows rather high scattering between 180 and 400 J. It is reasonable to assume that a minimum level of 200 J can be safely maintained under more reproducible rolling conditions.
Microstructural evaluation of the samples revealed a remarkable constancy of the ferrite, and bainite phase volume independent of the different rolling conditions (Figure 8). This suggests that the HTP alloy concept is very robust against process variations. The observed difference in strength is thus only caused by the ferrite grain size. The rolling conditions giving more severe austenite processing result in a finer grain size and thus better strength properties.

![Figure 8: LOM of for selected specimens after finish rolling and cooling.](image)

5. Industrial implementation in seamless pipe production

Based on the results obtained in the current laboratory rolling simulation it can be assumed that the HTP alloying concept allows producing at least API 5L X65 grade without quench and temper treatment. The conditions in the continuous mandrel rolling mill (see Figure 3) are best represented by the laboratory condition of high FRT and low final deformation. An important prerequisite for the success of the HTP alloy concept is that the material temperature at entry into the reheating furnace has not dropped below the ferrite start temperature. A partial phase transformation before final processing would force a substantial amount of the solute Nb to precipitate. Yet it is the solute Nb after finish rolling that brings about the transformation in the favorable ferritic-bainitic microstructure. Dedicated experiments have proven that when the temperature before reheating was below $A_{\text{R3}}$ the bainite volume in the final product becomes much reduced and the strength properties are fulfilling API 5L X52 grade only but still with excellent toughness. It could be however shown that such material can be repaired by a conventional quench and temper treatment to meet X65 properties.

Practically this means that the temperature of the rolling stock at entry into the reheating furnace should be on-line monitored. Material that has dropped below the ferrite start temperature must be rejected to either follow a repair treatment by quench and tempering or to be downgraded.

With particular process adaptations it should also be possible to produce API 5L X70 grade. This can be achieved by lowering the FRT and/or increasing final deformation (if the power
of the stretch reduction mill allows). In case that the stretch-reduction mill is not powerful enough the alloying concept can be extended to include solid solution hardening elements for strength increase. The extra additions, however, will result in alloying cost increase.

In other seamless pipe production processes, the HTP alloying concept is expected to work as well. For instance, in a Stiefel process piercing and elongation are performed at temperatures above 1170°C. The plug rolling temperature is typically ranging from 950 to 1100°C with 50% cross sectional reduction thus allowing for thermomechanical processing during these two steps. Lowering of the rotary furnace temperature by 50 to 70°C can shift plug rolling fully below the recrystallization stop temperature of the HTP alloy.

6. Conclusion

The HTP alloying concept based on low carbon and high niobium content was demonstrated to be capable of processing API 5L X65 and X70 high strength pipe grades by seamless tube rolling without using a quench and temper treatment.

Thus several cost advantages can be realized:
- Eliminating the quench and temper treatment saves approximately 50 $/t production cost.
- Liberating correspondingly production capacity on the quench and temper line helps to avoid capital investment in to additional facilities to satisfy the increasing market demand for high strength seamless pipe.
- The HTP alloying concept in the design presently tested works without expensive alloying elements such as molybdenum, vanadium and nickel.
- Eventually, reheating temperatures can be slightly lowered resulting in reduced energy cost.

Further work is in progress to elucidate the precipitation behavior of niobium and to explore opportunities for optimizing the alloy with respect to seamless pipe processing. Simultaneously industrial trials are being performed.

Acknowledgement

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References

