LEAN ALLOY DESIGN FOR HIGH AND HIGHEST STRENGTH STEELS

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Abstract

The production of thermomechanically rolled plates is closely related to the microalloying element niobium (Nb). This element ensures both a strength increase and also the much desired combination with high toughness. This paper presents a few examples that highlight the effects of grain refinement and precipitation hardening for line pipe grades in the range of X60 and X70 and shows that the onset of accelerated cooling reduces the effect of Nb on precipitation hardening.

The application of a lower carbon, higher Nb-bearing steel influences grain refinement and toughness because rolling takes place at higher temperatures. The benefit of grain refinement for toughness is illustrated for several examples of steel grades in the range of X80 and X100. Furthermore, high strength structural steel grades with yield strengths up to 960MPa have been produced with different levels of Nb. The paper shows data from daily production as well as results from R&D work based on laboratory investigations.

Introduction

Historically the term high, higher, or even ultra-high-strength steels has changed considerably over the past 50 to 60 years. The same is true for the process route of such steels. In the past, the standard processing method for steel grades with yield strength levels of up to 500MPa used to be by quenching and tempering (QT) [1]. The usual historical chemical compositions are shown in Table 1.

<table>
<thead>
<tr>
<th>Grade</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>AL</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>V</th>
<th>Nb</th>
<th>Ti</th>
<th>Bor</th>
<th>N</th>
<th>CE_{in}</th>
<th>Pcm</th>
</tr>
</thead>
<tbody>
<tr>
<td>S 500 Q (1)</td>
<td>0.180</td>
<td>0.40</td>
<td>1.40</td>
<td>0.04</td>
<td>0.50</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.080</td>
<td>0.001</td>
<td>0.001</td>
<td>0.002</td>
<td>0.015</td>
<td>0.53</td>
<td>0.30</td>
</tr>
<tr>
<td>S 500 Q (2)</td>
<td>0.080</td>
<td>0.30</td>
<td>1.40</td>
<td>0.03</td>
<td>0.20</td>
<td>0.50</td>
<td>0.30</td>
<td>0.02</td>
<td>0.050</td>
<td>0.001</td>
<td>0.001</td>
<td>0.0002</td>
<td>0.0082</td>
<td>0.46</td>
<td>0.21</td>
</tr>
</tbody>
</table>

For higher strength levels in the range of Re 700 to 960MPa had to be achieved with high amounts of Mo, Ni, Cr. As one can see in Table 2, the disadvantages of such approaches are the high costs of using such alloys and the fact that weldability is impaired. Furthermore, the toughness was negatively influenced by inadequate cleanliness, which was quite different from the modern standard we have today.
These limitations and the increasing requirements of customers for high and ultra high-strength steel grades led to the development of new processing methods. After modernization of the plate mill and installation of a new cooling line with high performance as shown in Figure 1, voestalpine Grobblech GmbH (VAGB) expended considerable efforts into the research and development of optimized cooling methods for Nb-bearing line-pipe and high-strength steels.

Over the years the following steps have been taken for producing high-strength:

- Conventional QT treatment with “rich alloy” steels;
- TM rolling with subsequent quenching from rolling temperature and tempering;
- New considerations for leaner alloy design to counteract the steadily increasing raw material cost. This route also requires new processing methods such as TM rolling followed by online accelerated cooling with the ultimate goal to avoid additional heat treatment operations, and;
- Development of methods in order to produce modern materials in greater thicknesses.

TM steels without accelerated cooling could reach a maximum yield strength of 550MPa (X80) and Quenched and Tempered (QT) grades could achieve a yield strength of up to 900MPa with a chemical composition (Ni, Mo) that was fairly expensive at the time. Therefore, new process routes for using a leaner alloy design and the required changes in the production methods had to be developed. The new processes developed combined the advantages of TM rolling and saved time and costs for any subsequent heat treatment. Intensive research and development work provided the theoretical basis for the ideal combination of material quality, economy and shorter lead times. New production facilities enabled VAGB to apply and optimize the methods for accelerated cooling.
R&D Work at VAGB for Applying the Lean Alloy Design

The role of niobium in QT steels

As niobium has a lower solubility at the usual hardening temperatures of around 920°C, it has a very limited effect on increasing the strength for QT steels. The only exceptions are low and extra low carbon medium-strength (500MPa) steels, when higher hardening temperatures are applied. Table 3 shows as an example of the chemical composition for an extra low carbon grade with an unusually high Nb content.

Table 3, Chemical composition [mass%] of ELC high Nb grade.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>AL</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>V</th>
<th>Nb</th>
<th>Ti</th>
<th>Bor</th>
<th>N</th>
<th>CE_{IIW}</th>
<th>Pcm</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.025</td>
<td>0.16</td>
<td>1.51</td>
<td>0.011</td>
<td>0.0014</td>
<td>0.02</td>
<td>0.26</td>
<td>0.16</td>
<td>0.00</td>
<td>0.25</td>
<td>0.002</td>
<td>0.094</td>
<td>0.009</td>
<td>0.0002</td>
<td>0.0046</td>
<td>0.36</td>
<td>0.14</td>
</tr>
</tbody>
</table>

Thick plates at 40mm of this material were hot rolled for establishing the influence of different hardening temperatures on strength and toughness. The results are seen in Figure 2. Both the strength and, unfortunately, also the Charpy V transition temperature (50% FATT) increase with higher hardening temperatures. The strength increase is partially related to the improved hardenability caused by niobium in solid solution, but mainly by precipitation hardening effects during tempering at 600°C. Despite precipitation hardening, niobium generally has a positive effect on toughness due to the well-known effect of grain refinement developed in the as hot rolled microstructure.

Figure 2. Influence of hardening temperature on strength and toughness of QT plates, 40mm thickness (also tempered at 600°C).

The Role of Niobium in Medium-Strength TM Steels (X65 and X70)

A very important factor for the effect of Nb on strength is the reheating temperature, as this determines the amount of niobium that enters solution. In order to get the maximum effect of Nb an extra low carbon grade was selected for the test series. The chemical composition is given in Table 4.

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Steel plates of steel grade X70 at 20mm were rolled from a production heat in the laboratory and the final rolling temperature was kept at 850°C for all tests made. Cooling conditions were varied from air cooling (AC) to interrupted accelerated cooling (ACC) and direct intensive cooling (DIC). The cooling stop temperature was about 500°C for ACC and below 400°C for DIC, with a mean cooling rate of 40K/s. The results are documented in Figure 3.

The shaded column in Figure 3 characterizes the temperature range of the complete solution of Nb calculated by the formulas of Norberg and Aronsson (NbC) as well as Irvine (Nb(CN)). An increase in strength in all three cooling conditions is found up to a reheating temperature of approximately 1100°C. At this point all the Nb is in solution and further temperature increases have no niobium-related positive effect on strength; the negative effect on toughness, however, is well known [6].

In a second test series, laboratory heats were used in which the Nb content was varied within the usual ranges (see Table 5). It should be noted that the high nitrogen and sulphur contents, which are considerably higher than in actual production of X65 line pipe grades, are typical for laboratory heats.

Table 5. Chemical composition [mass%] of lab heats, grade X60 to X70.

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>AL</th>
<th>V</th>
<th>Nb</th>
<th>Ti</th>
<th>Bor</th>
<th>N</th>
<th>others</th>
<th>CEw</th>
<th>Pcm</th>
</tr>
</thead>
<tbody>
<tr>
<td>517/1</td>
<td>0.052</td>
<td>0.29</td>
<td>1.46</td>
<td>0.010</td>
<td>0.0003</td>
<td>0.023</td>
<td>0.25</td>
<td>0.08</td>
<td>0.01</td>
<td>0.02</td>
<td>0.002</td>
<td>0.0001</td>
<td>0.0001</td>
<td>0.0002</td>
</tr>
<tr>
<td>517/2</td>
<td>0.055</td>
<td>0.30</td>
<td>1.49</td>
<td>0.010</td>
<td>0.0028</td>
<td>0.026</td>
<td>0.25</td>
<td>0.08</td>
<td>0.01</td>
<td>0.02</td>
<td>0.001</td>
<td>0.022</td>
<td>0.003</td>
<td>0.0014</td>
</tr>
<tr>
<td>517/3</td>
<td>0.055</td>
<td>0.29</td>
<td>1.46</td>
<td>0.010</td>
<td>0.0024</td>
<td>0.026</td>
<td>0.25</td>
<td>0.09</td>
<td>0.01</td>
<td>0.02</td>
<td>0.001</td>
<td>0.043</td>
<td>0.003</td>
<td>0.0004</td>
</tr>
</tbody>
</table>
The reheating temperature was kept at 1150°C in all cases and the Nb content ranged between 0 and 0.043%. The results for a variety of Nb contents and different cooling modes are shown in Figure 4.

While air-cooling causes a considerable yield strength increase with higher amounts of Nb, this effect is not as great for ACC and DIC conditions. However, ACC and DIC materials generally show higher strength levels [7]. This figure also shows that the strength increase is not proportional to the Nb content. The reason for these facts is:

- The precipitation hardening effect increases with Nb, but is most effective at lower contents, and;
- In ACC, and especially the DIC process, the precipitation hardening effect is not fully complete because an increasing part of Nb remains in solution.

The graphs in Figure 3 and 4 clearly indicate that medium-strength steels require ACC or DIC cooling modes and micro alloying with Nb.

Additionally, the combined effect of niobium and vanadium was examined in a series of plates from lab heats (see Table 6) produced with different production routes. In comparison to table 5 the steels in Table 6 have no chromium and therefore the hardenability is reduced.

Table 6. Chemical composition [mass%] of laboratory heats, grade X60-X70.

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>AL</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>V</th>
<th>Nb</th>
<th>Ti</th>
<th>B</th>
<th>N</th>
<th>CEi</th>
<th>Pcm</th>
</tr>
</thead>
<tbody>
<tr>
<td>557/1</td>
<td>0.059</td>
<td>0.28</td>
<td>1.15</td>
<td>0.008</td>
<td>0.0049</td>
<td>0.02</td>
<td>0.03</td>
<td>0.03</td>
<td>0.00</td>
<td>0.02</td>
<td>0.001</td>
<td>0.001</td>
<td>0.001</td>
<td>0.001</td>
<td>0.0084</td>
<td>0.26</td>
<td>0.13</td>
</tr>
<tr>
<td>557/2</td>
<td>0.060</td>
<td>0.28</td>
<td>1.14</td>
<td>0.008</td>
<td>0.0044</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
<td>0.00</td>
<td>0.02</td>
<td>0.001</td>
<td>0.001</td>
<td>0.001</td>
<td>0.002</td>
<td>0.0082</td>
<td>0.26</td>
<td>0.13</td>
</tr>
<tr>
<td>557/3</td>
<td>0.067</td>
<td>0.27</td>
<td>1.13</td>
<td>0.008</td>
<td>0.0031</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
<td>0.00</td>
<td>0.02</td>
<td>0.067</td>
<td>0.041</td>
<td>0.001</td>
<td>0.002</td>
<td>0.0099</td>
<td>0.28</td>
<td>0.14</td>
</tr>
</tbody>
</table>

The addition of 0.04% niobium results in a noticeable strength increase for all three cooling conditions. A further addition of 0.07% vanadium brings a small strength increase for AC and DIC plates, while this was not observed for the ACC plates. Figure 5 provides the data for the influence of niobium and vanadium on the mechanical properties of lab plates in thickness of 20mm.
With systematic R&D work at VAGB within the last six years the proper production routes have been established for applying a leaner alloy design with TM rolling and optimized parameters for accelerated cooling. The main goal of this work was to avoid a subsequent heat treatment. The effects of Nb on hardness and strength were investigated by multiple regression analysis of more than 200 laboratory heats and samples from different production heats. The thermal and mechanical treatment of the samples was carried out in a dilatometer. Figure 6 shows the partial influence of vanadium (picture on the left) and niobium (right hand side) on the hardness of dilatometer samples. The samples were heated to 1150°C, deformed with different degrees in the range of 800 to 880°C and subsequently exposed to different cooling rates. The data in Figure 6 are valid for a mean chemical composition as follows: 0.08%C, 1.6%Mn, 0.2%Cr, 0.3%Ni, 0.1%Mo, 0.02%V, 0.03%Nb, 0.01%Ti and small additions of Boron [3].

![Figure 5. Influence of niobium and vanadium on the mechanical properties of 20mm laboratory plates.](image)

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>Rp0.2</th>
<th>Rm</th>
</tr>
</thead>
<tbody>
<tr>
<td>557/1</td>
<td>150</td>
<td>500</td>
</tr>
<tr>
<td>557/2</td>
<td>180</td>
<td>550</td>
</tr>
<tr>
<td>557/3</td>
<td>200</td>
<td>600</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>Rp0.2</th>
<th>Rm</th>
</tr>
</thead>
<tbody>
<tr>
<td>557/1</td>
<td>150</td>
<td>500</td>
</tr>
<tr>
<td>557/2</td>
<td>180</td>
<td>550</td>
</tr>
<tr>
<td>557/3</td>
<td>200</td>
<td>600</td>
</tr>
</tbody>
</table>
These conclusive tests have revealed the following:

- Niobium shows twice the effect of vanadium, and;
- The strengthening effect of V and Nb is reduced when the cooling rate increases. The reason is that precipitation hardening is not fully effective because Nb or V remain in solution.

The Role of Niobium in High-strength Steels

High and even higher strength steels generally have higher alloy content and therefore in the past were mainly produced by Quenching and Tempering (QT). However, VAGB has developed new methods for the modern processing of such steels. With TM rolling the effect of precipitation hardening via Nb is utilized to a much higher degree as would be possible in the QT process. The new material designs are based on fairly low carbon contents and production routes that comprise thermomechanical rolling followed by accelerated cooling with or without tempering [3].

The basis for this development has been because austenite conditioning causes a high dislocation density that normally results in a decrease in hardenability. As an example, Figure 7 shows the results of dilatometer tests made with ELC X65 line-pipe material. The samples were heated to 1150°C and cooled to the deformation temperature in a range of 800 to 880°C with a cooling rate of 20°C/s. Various degrees of deformation were applied in a single step. After deformation the samples were cooled with 40°C/s to room temperature. It can be seen that an increase in the degree of deformation leads to reduced hardness and an increase in the $\gamma \rightarrow \alpha$ transformation temperature. The temperature range for austenite conditioning is most effectively opened up by additions of niobium. The austenite conditioning has a very strong positive effect on toughness, which is a prerequisite for meeting line pipe requirements.
The changes in the microstructure with different degrees of deformation are illustrated in Figure 8. Increasing deformation degrees cause a change from bainitic to a predominantly (acicular) ferritic microstructure.

Figure 7. Influence of austenite conditioning degree on hardenability and $\gamma \rightarrow \alpha$ transformation temperature on ELC X65 NbTi.

Figure 8. Microstructures of ELC-X65 NbTi at different deformation degrees, HNO$_3$-etchant.
In higher strength steels, e.g. in an X120, a contrary effect in the degree of deformation has been found as shown in Figure 9. Deformations up to a certain amount of Phi result in a hardness increase because of the bainitic/martensitic microstructure. The loss of hardness at extremely high deformation degrees in a single step is mainly caused by partial recrystallization of the austenite and the increased and $\gamma$ to $\alpha$-transformation temperature. It can be concluded that niobium has a positive effect both on the strength and toughness of higher and even higher strength steels.

Figure 10 provides the micrographs for different degrees of deformation. The pictures on the left and in the center reveal bainitic/martensitic microstructure whereas the high degree of deformation leads to the formation of noticeable portions of ferrite. Since the metallographic specimens were taken perpendicular to the direction of deformation in the dilatometer, the austenite pancaking cannot be observed.

Figure 9. Influence of austenite conditioning degree on hardenability and $\gamma$ to $\alpha$-transformation temperature on ELC X120.

Figure 10. Microstructures of ELC-X120 CrMoNbTiB at different deformation degrees, HNO$_3$-etchant.
Figure 11 reveals the influence of deformation on the hardness of two different steel grades (X65 and Alform 700) depending on cooling rate. The ferritic/bainitic X65 Nb suffers a substantial loss of hardness with a Phi value of 0.3, especially at high cooling rates. In the Alform 700 the deformation results in a very small increase in hardness for all cooling rates.

![Graph showing influence of deformation on hardness](image)

**Figure 11. Influence of deformation degree on hardness for X65 and Alform 700.**

**Influence of Cooling Conditions**

Figure 12 provides an example for the possibility of online production without tempering for steel grades with yield strengths of 700 and 900MPa. The chemical compositions of these low carbon grades are listed in Table 7.

Table 7. Chemical composition [mass%] of steel grades with Y.S. 700 and 900MPa.

<table>
<thead>
<tr>
<th>Grade</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr+Mo+Ni+Cu</th>
<th>V</th>
<th>Nb</th>
<th>Ti</th>
<th>Bor</th>
<th>N</th>
<th>CEIW</th>
<th>Pcm</th>
</tr>
</thead>
<tbody>
<tr>
<td>alform 700</td>
<td>0.04</td>
<td>0.3</td>
<td>1.9</td>
<td>0.010</td>
<td>0.0003</td>
<td>&lt; 0.3</td>
<td>0.002</td>
<td>0.044</td>
<td>0.01</td>
<td>0.001</td>
<td>0.004</td>
<td>0.42</td>
<td>0.17</td>
</tr>
<tr>
<td>alform 900</td>
<td>0.07</td>
<td>0.3</td>
<td>1.7</td>
<td>0.007</td>
<td>0.0004</td>
<td>&lt; 0.9</td>
<td>0.003</td>
<td>0.040</td>
<td>0.01</td>
<td>0.001</td>
<td>0.004</td>
<td>0.53</td>
<td>0.23</td>
</tr>
</tbody>
</table>

Results of laboratory rolled plates at thickness of 20mm indicate the strong influence of the cooling stop temperature on the mechanical properties. Cooling stop temperatures well below 500°C must be selected in order to achieve high yield and tensile strength values. Whenever a high tensile strength is desired, or can be tolerated, the online process is a good production method. When a combination of high yield and low UTS is required, the processing window is within a range of +/- 25°C, which is extremely narrow. Figure 12 shows that lower cooling stop temperatures would lead to an increase in the tensile strength and a small decrease in the yield strength. Whilst slightly higher cooling stop temperatures would cause a dramatic decrease in the mechanical properties. To maintain such a small processing window in daily production over the entire plate length and thickness range requires extreme accuracy in the process control. Before TM rolling with accelerated cooling can be used in daily production, considerable work has to be put into improved plant technology and process models for the cooling line [3].

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The SEM micrographs in Figure 13 indicate that for cooling stop temperatures above 500°C the microstructure consists of upper bainite with remarkable amounts of coarse-grain carbon rich MA phases. Lower cooling stop temperatures below 400°C result in a bainitic-martensitic structure with a small concentration of finely dispersed retained austenite and carbides.

Figure 14 shows the influence of carbon content on mechanical properties of steels with otherwise similar chemical compositions. The laboratory plates were finished at about 850°C and exposed to accelerated cooling (DIC process). With higher carbon content the tensile strength has a steeper increase than that of the yield strength. In both cases the rate of increase declines and thus the yield-to-tensile ratio is lowered.
A low carbon content ensures high yield-to-tensile-strength ratios even at low cooling stop temperatures without tempering. Low carbon content, however, limits the yield strength. For achieving higher strength values beyond a yield level of 900 MPa it is necessary to increase the carbon content which results in a high tensile strength.

When customer specifications require high yield strengths combined with high yield-to-tensile ratios, higher carbon contents are necessary and in most cases a tempering treatment is also necessary.

The Influence of Tempering

In order to investigate the influence of niobium on the mechanical properties of high-strength TMCP steels the following laboratory heats were processed. The variations of Nb in the chemical composition can be seen in Table 8. All three grades have very good hardenability because of their high Cr, Ni and Mo content.

### Table 8. Chemical composition [mass%], laboratory heats of high-strength steels.

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>AL</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>V</th>
<th>Nb</th>
<th>Ti</th>
<th>B</th>
<th>N</th>
<th>CE_{exp}</th>
<th>Pcm</th>
</tr>
</thead>
<tbody>
<tr>
<td>602/1</td>
<td>0.079</td>
<td>0.30</td>
<td>0.82</td>
<td>0.008</td>
<td>0.0053</td>
<td>0.03</td>
<td>0.59</td>
<td>1.99</td>
<td>0.49</td>
<td>0.30</td>
<td>0.039</td>
<td>0.001</td>
<td>0.001</td>
<td>0.002</td>
<td>0.0062</td>
<td>0.59</td>
<td>0.25</td>
</tr>
<tr>
<td>602/2</td>
<td>0.081</td>
<td>0.30</td>
<td>0.81</td>
<td>0.009</td>
<td>0.0048</td>
<td>0.03</td>
<td>0.59</td>
<td>1.99</td>
<td>0.49</td>
<td>0.30</td>
<td>0.037</td>
<td>0.041</td>
<td>0.002</td>
<td>0.0003</td>
<td>0.0068</td>
<td>0.59</td>
<td>0.25</td>
</tr>
<tr>
<td>602/3</td>
<td>0.083</td>
<td>0.29</td>
<td>0.81</td>
<td>0.009</td>
<td>0.0035</td>
<td>0.04</td>
<td>0.58</td>
<td>1.97</td>
<td>0.49</td>
<td>0.30</td>
<td>0.036</td>
<td>0.083</td>
<td>0.002</td>
<td>0.0003</td>
<td>0.0071</td>
<td>0.59</td>
<td>0.25</td>
</tr>
</tbody>
</table>

The reheating temperature for rolling was 1,150°C and the final rolling temperature was again in the range of 850°C. After being rolled, the plates were cooled either in still air (AC) or treated by DIC (direct intensive cooling) to low temperatures.

It was surprising in both the AC and the DIC condition that no noticeable influence of niobium was observed in the mechanical properties of 15 mm laboratory plates. The reason for this phenomenon is as follows: In the AC (air cooled) condition the precipitation
hardening effect is suppressed because of the low $\gamma \alpha$ transformation temperature. In the case of DIC, precipitation hardening was inhibited because of the high cooling rates and the low cooling stop temperatures. The expected strength increase of the Nb-related TM effect did not take place. The high concentration of alloying elements had already caused the TM effect, which can also be seen in the Nb free alloy (see Figure 15).

![AC](image1)

![DIC](image2)

**Figure 15. Mechanical properties of TMCP steels with different Nb contents (plate thickness 15 mm).**

A strong positive influence of niobium is noticed up to approximately 0.04% after the TMCP processed 15mm laboratory plates in both AC (air cooled) and DIC (direct intensive cooling) condition are tempered, as shown in Figure 16 [5].

![AC+600°C/L](image3)

![DIC+600°C/L](image4)

**Figure 16. Influence of Nb content on mechanical properties of tempered laboratory plates, thickness 15mm.**
Figure 17 shows the influence of tempering temperatures on the mechanical properties of alform 960 in thickness of 15mm. Maximum UTS is reached in the as rolled condition and for annealing temperatures up to 300°C. The YS has its maximum around 300-400°C. The material exhibits high work hardening ability in the as-rolled or low-tempered condition. The best Charpy toughness properties are achieved in the as-rolled condition. The usual tempering temperatures in the range of 560-640°C result in very high yield-to-tensile ratios and are only advantageous for improved bending and forming properties as well as for reducing internal stresses. At 700°C a severe strength drop is observed due to partial austenitization.

Figure 17. Influence of tempering temperatures on mechanical properties of alform 960.

Development of High Toughness

General Influences on Toughness

The development of toughness is shown in Figure 18. Grade S355 produced in the old fashioned way was found to possess just adequate toughness levels. Therefore, by improving the steel cleanliness and microalloying with 0.020%Nb resulted in noticeable toughness increases along with a higher upper shelf energy and a lower transition temperature [4]. The proper application of TM rolling with accelerated cooling, coupled with a lean alloy design with a low carbon content and NbTi microalloying led to an X80 grade with excellent toughness properties [8]. The Charpy tests were made in accordance with DIN specifications and a comparison with ASTM for X80 only is also shown in the figure.
The reason for the difference in toughness is well understood especially when the microstructures of the two materials are compared as shown in Figure 19. The banded and rather coarse-grained ferritic-pearlitic microstructure is typical for the S355 grade and the reason for the toughness properties which meet the standards. In contrast, the alloy design for the X80 and the related fine tuning of the TM-rolling and cooling conditions result in high-strength and also in superior toughness values [3].

Influence of Accelerated Cooling on Toughness

It is a well known fact that in medium-strength line pipe grades (X65, X70) accelerated cooling has a very positive effect on toughness since no formation of splits occurs in the fracture area. This leads mainly to high upper shelf values in the range of 400 to 500J with the ASTM pendulum and to a reduction of transition temperatures.
In order to evaluate the influence of different kinds of cooling on toughness, 20mm thick plates of alform 700 were rolled in the laboratory. The reheating temperature for rolling was 1,150°C and the final rolling temperature was in the range of 800 to 850°C. Air cooling resulted in a high transition temperature combined with low strength ($R_{p0.2} \sim 500\text{MPa}$, $R_m \sim 700\text{MPa}$). With respect to toughness, the advantage of accelerated cooling with CST below 400°C is shown in Figure 21. Both a transition temperature of -100°C and a high strength level ($R_{p0.2} \sim 800\text{MPa}$, $R_m \sim 940\text{MPa}$) were achieved.

The reason for the poor properties of the air-cooled material is due to the microstructure, which is shown in Figure 22. The air-cooled sample (on the left) has upper bainite with a high portion of MA phases (carbon enriched martensite and retained austenite), whereas the DIC structure (on the right) is predominantly martensitic. A similar situation prevailed as shown earlier in Figure 13.
Influence of Austenite Conditioning on High-strength Steels

Laboratory plates of alform 960 with a chemical composition according to Table 8 were rolled to a thickness of 10mm at various final rolling temperatures. After the plates were rolled, accelerated cooling was applied with a cooling stop temperature below 450°C.

Table 8. Chemical composition [mass%] of alform 960.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>AL</th>
<th>V</th>
<th>Nb</th>
<th>Ti</th>
<th>Bor</th>
<th>N</th>
<th>others</th>
<th>CEw</th>
<th>Pcm</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.07</td>
<td>0.3</td>
<td>1.9</td>
<td>0.009</td>
<td>0.008</td>
<td>0.04</td>
<td>0.002</td>
<td>0.05</td>
<td>0.01</td>
<td>0.0014</td>
<td>0.0046</td>
<td>Cr, Ni, Mo, Cu</td>
<td>0.45</td>
<td>0.20</td>
</tr>
</tbody>
</table>

Figure 23 shows the influence of the final rolling temperatures on the mechanical properties and the toughness of the laboratory plates. The highest strength was achieved because of the optimum state of austenite conditioning in the FRT range of 800°C (also see Figure 9). The highest toughness is also achieved within a similar temperature range, a fact which is understood when the microstructures are compared in Figure 24 [2].

Both microstructures are predominantly martensitic, for which reason the grain boundaries of the former austenite are easily recognized. The former austenite at FRT 755°C shows intensive pancaking and a very fine secondary structure [9]. Pancaking is less pronounced at a FRT of 920°C, and the secondary structure is coarse, which goes to explain the drop in toughness and strength.
Economy and the need of good weldability require materials with a “lean alloy” composition and fairly low carbon contents. The additional benefit of combining high strength and excellent toughness can only be achieved with optimized processing parameters including online-accelerated cooling.

The lean alloy design with low carbon contents is a well-established processing method up to a yield strength of 960MPa. Excellent toughness (upper shelf energy 100J at -80°C for 10mm plates) can also be achieved in alform 960 when proper final rolling and cooling stop temperatures are selected. Niobium has become the most helpful addition for obtaining these properties. It is less effective in QT steels, because there is only a very small portion of Nb in solution at the standard quenching temperatures. The optimum useful amount and/or a combination with other microalloying elements have to be investigated for each individual case.
The modernization of the plate mill, including the installation of a high capacity cooling line, has enabled voestalpine Grobblech GmbH to apply these new and more efficient processing methods for line pipe grades and highest strength steels. The research and development work carried out for using a “lean alloy design” has been described in the paper. The effect of different Nb contents on strength and toughness has been investigated in TM steels for a variety of cooling methods. The goal of this work has been to avoid subsequent heat treatment. Since the lean alloy design requires fairly low carbon contents, such steels provide excellent weldability as an additional benefit.

R&D work in the future will concentrate on fine tuning the alloy design and the process parameters to extend the lean alloy design to high-strength steels with good toughness, including plate thicknesses up to 40 mm and beyond. Assistance in this work will be provided by modern investigation methods such as atom probe [9].

References


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