SOLIDIFICATION STRUCTURE AND PROPERTIES OF Nb-V MICROALLOYED STEELS

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ABSTRACT

A study is presented on the solidification structure of continuously cast billets of Nb-V microalloyed steels as affected by Nb content and by electromagnetic stirring. Two steel grades having a comparable composition and differing only in their Nb content were compared to a third non-microalloyed grade selected as a reference alloy. Investigations on solidification microstructure and on crystallographic texture showed that electromagnetic stirring was effective in reducing solidification defects in the as cast billets but it did not alter the structure and the corresponding properties of the steels. Hot ductility curves were determined by tensile tests at increasing temperature, showing a ductility trough in the temperature range 850-1050°C. The results were interpreted on the basis of chemical composition and of expected precipitates of the steels investigated.

KEYWORDS

HSLA steels, Nb-V steels, solidification, continuous casting, microstructure, hot ductility

INTRODUCTION

The properties of microalloyed steels are determined by a sequence of microstructural changes occurring on solidification, during thermomechanical processing and final cooling of steel products. The peculiar effects of the microalloying elements on the above changes are well documented [1]. Among the microalloying elements, niobium is frequently adopted due to its strong effects on structure and properties. Depending on thermal cycles during steel production, Nb can be in solution or in precipitates (typically of the carbonitride type) thus affecting recrystallization, grain growth, \( \gamma/\alpha \) equilibrium transformation and strain-induced ferrite transformation. It is well demonstrated that recrystallization and grain growth of austenite are significantly suppressed by the presence of NbC. These precipitates, can also act as preferred sites for ferrite nucleation, leading to enhanced transformation kinetics under suitable conditions. On the contrary, when in solid solution, Nb can retard the \( \gamma/\alpha \) transformation by segregating at the \( \gamma/\alpha \) interfaces and hindering the growth of ferrite by a solute drag effect [2-4].

Hot ductility is of great concern for specific wrought products and it is strictly related to precipitate condition. Hot ductility is generally evaluated by the reduction of area in tensile specimens tested at increasing temperatures. The poor ductility often encountered in the range 800-950°C can be accounted for to intergranular failure in the \( \gamma \) phase due to the formation of a soft ferrite film at grain boundaries or due to grain boundary sliding of the austenite. This latter phenomenon is greatly enhanced by particles located at grain boundaries and by the depletion in precipitates from the
matrix adjacent to boundaries, often occurring in Nb-V microalloyed steels. Based on these general statements, a large debate was raised in literature in recent years on possible modification of steel chemistry and their thermomechanical treatments to improve hot ductility while maintaining optimal end-product properties [5-7]. Especially the role of titanium was deeply investigated since, in addition to its effect as a grain refiner, it can either improve or deplete hot ductility, depending on the amount and size of nitrides formed and on their interaction with other precipitates [6,8-10].

In this paper, emphasis is put on the as cast structure and hot ductility of two Nb-V microalloyed steel grades featuring different contents of Nb and of a C-Mn grade taken as a reference alloy. Combined microstructural and mechanical analyses were presented to evaluate possible modification in steel properties as a function of steel composition and solidification condition.

MATERIALS AND EXPERIMENTAL PROCEDURES

Continuously cast billets of Nb-V microalloyed steels having the chemical composition given in Table I were investigated. Two of the steel grades (H-Nb and L-Nb) had comparable compositions and differed only in their Nb content, while a third non-microalloyed standard grade (STD) was selected from current production as a reference alloy.

Table I. Chemical compositions (mass %) of the steels investigated

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>N</th>
<th>Mn</th>
<th>Al</th>
<th>Si</th>
<th>Nb</th>
<th>V</th>
<th>Ti</th>
<th>S</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>H-Nb</td>
<td>0.14</td>
<td>0.0071</td>
<td>1.25</td>
<td>0.028</td>
<td>0.29</td>
<td>0.031</td>
<td>0.051</td>
<td>0.004</td>
<td>0.002</td>
<td>0.016</td>
</tr>
<tr>
<td>L-Nb</td>
<td>0.14</td>
<td>0.0061</td>
<td>1.27</td>
<td>0.027</td>
<td>0.23</td>
<td>0.022</td>
<td>0.052</td>
<td>0.002</td>
<td>0.003</td>
<td>0.014</td>
</tr>
<tr>
<td>STD</td>
<td>0.15</td>
<td>0.0080</td>
<td>0.86</td>
<td>0.019</td>
<td>0.24</td>
<td>0.002</td>
<td>0.003</td>
<td>0.003</td>
<td>0.007</td>
<td>0.014</td>
</tr>
</tbody>
</table>

A first theoretical analysis was carried out by calculating the equilibrium phases to be found in the investigated steels as a function of temperature. To this purpose, thermodynamic analyses were carried out using the Thermo-Calc software, selecting the TC-Fe steel database.

Experimental investigations were then carried out on the solidification structure developed under industrial continuous casting conditions, with and without the effects of electromagnetic stirring (EMS), on circular billets having a diameter of 360 mm.

Optical and SEM microstructural observations were carried out to investigate the effects of Nb and of stirring on the resulting steel structure. For this purpose, the billets were cross sectioned, preliminarily analysed in their macrostructure by a Baumann etching procedure and further investigated from a microstructural viewpoint by cutting samples close to billet surface, billet axis and at half of billet radius. The crystallographic orientation of the as cast structure was also analysed by the Electron Back Scattered Diffraction (EBSD) technique to complete the frame of analyses on the steel structure.

The hot ductility of the steels was investigated by tensile tests carried out in the temperature range 750-1150°C. Cylindrical specimens having a gage length of 40 mm and a diameter of 10 mm were machined from longitudinal, radial and hoop directions with respect to original billet axis. The samples were then pulled to fracture and ductility as a function of temperature was evaluated through their reduction of area.

RESULTS AND DISCUSSION

Equilibrium phase constitution of the steels

The amount of equilibrium secondary phases in the austenite temperature range was evaluated by
using the Thermocalc code. Particular emphasis was given to the evolution of precipitates related to microalloying elements. Therefore, the temperature range corresponding to solid state transformation (700-1400°C) was specifically considered. AlN-type nitrides and complex carbonitrides having a general composition \((\text{Nb}_{x}\text{Ti}_{y}\text{V}_{(0.5-x-y)})\) \(\text{C}_{y}\text{N}_{(0.5-y)}\) were expected, according to well established experimental and theoretical works [1,11].

From figure 1 the molar fraction of carbonitrides (CN) and of the AlN phase to be expected as a function of temperature is depicted for the three steels studied. The formation of CN phase becomes significant under equilibrium conditions at temperatures lower than about 950 and 1100°C for the L-Nb and H-Nb steels, respectively, while the AlN phase appeared at a temperature of 1050°C, irrespective of steel composition.

When considering the carbonitrides, it is apparent from the data depicted in figure 2 that the stoichiometry of the complex \((\text{Nb}_{x}\text{Ti}_{y}\text{V}_{(0.5-x-y)})\) \(\text{C}_{y}\text{N}_{(0.5-y)}\) phase is strongly affected by temperature. In both the microalloyed steels, at the highest temperatures this phase actually represents a titanium nitride containing limited amounts of Nb, V and C. By lowering the temperature, depletion in Ti and N and enrichment in Nb, V and C continuously occurs. Eventually, at 700°C, this phase becomes a carbide particularly rich in Nb and V. The same trend also holds for the STD grade, although the variations with temperature are less marked due to much lower amount of carbide forming elements.

![Fig. 1. Predicted equilibrium molar fraction of CN (left) and AlN (right) phases as a function of temperature](image1)

![Fig. 2. Composition of the metallic elements (left) and interstitials (right) of the carbo-nitrides as a function of temperature for the H-Nb steel](image2)
The compositional variations predicted by the thermodynamic analysis are a consequence of the continuous mutual solubility of the carbides and nitrides formed by Ti, Nb and V and of the decreasing solubility of the different carbides and nitrides with temperature in the $\gamma$-iron [1,12-14].

Solidification structure of the billets

Representative macrographs of the solidification structure of the billets are given in figures 3-5. From careful inspection of solidification defects and extension of the different peculiar regions (chilled surface zone, columnar zone, equiaxed zone) of the as cast billets, it can be inferred that in the steels solidified with EMS, crack are significantly reduced or even removed. A comparison of the steels solidified without EMS also suggests that an increase of the Nb content corresponds to a greater number of radial cracks in the columnar zone and, irrespective of the effects of EMS, an increase in Nb content also results in a larger extension of the central equiaxed zone and a corresponding reduction of the columnar zone [15].

Fig. 3. Macrostructure of the H-Nb steel grade solidified with (left) and without EMS (right)

Fig. 4. Macrostructure of the L-Nb steel grade solidified with (left) and without EMS (right)
Microstructure and hardness

The microstructure of the as cast billets was mainly formed by a combination of ferrite and pearlite, as depicted in figures 6 and 7. In the Nb-containing steels (figure 6), allotriomorphic polygonal ferrite at prior austenite grain boundaries was often visible, together with a relatively large amount of polygonal ferrite grains at intragranular sites (idiomorphic ferrite) which are supposed to have nucleated from inclusions or precipitates inside the $\gamma$ grains [16]. The STD steel featured a slightly different structure consisting of occasional Widmanstätten ferrite aggregates growing from polygonal ferrite located at prior austenite grain boundaries and of relatively large plates of intragranular ferrite (often termed in literature acicular ferrite or intragranular Widmanstätten ferrite) growing from inclusions or from pre-existing polygonal ferrite, as shown in figure 7.
The pearlite nodules, often observed as irresolvable dark islands in the optical images, were better characterised by SEM observations, as reported in the representative micrographs given in figure 8. Irrespective of steel grade and of use of EMS, the measured interlamellar spacing was of the order of 0.2 micrometers. This morphological feature of pearlite colonies is governed by diffusion of carbon, which in turn depends on cooling rate experienced by the steel and on carbon concentration [16]. The constancy of interlamellar spacing is thus in full agreement with the comparable carbon content and solidification process experienced by the steels investigated. Finally, no definite trend of interlamellar spacing was noted as a function of sample location inside the billets.

Analyses on crystallographic orientation of the $\alpha$-phase were carried out by EBSD in order to evaluate possible effects induced by EMS during solidification. From results on all of the sample conditions investigated, it was noticed that no preferred texture had developed on solidification and further solid-state transformation of the structure, as reported in the representative map shown in figure 9. This statement was confirmed for the steels solidified either with and without EMS.
Fig. 9. Orientation map of grains (left) and polar figure (right) of a selected area of the structure obtained by EBSD on the H-Nb steel sample solidified with EMS.

Finally, in table II a summary of the average hardness values measured in the samples investigated is reported. All the data are consistent in showing a slight increase of hardness at midthickness of the billets and, as expected, higher hardness values for the microalloyed Nb-V steel grades. Also for this case, EMS does not seem to have a significant effect.

Table II. Hardness values of the samples investigated

<table>
<thead>
<tr>
<th>steel grade</th>
<th>EMS</th>
<th>HRB</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>surface</td>
</tr>
<tr>
<td>H-Nb</td>
<td>Y</td>
<td>87,5 ± 2,4</td>
</tr>
<tr>
<td></td>
<td>N</td>
<td>-</td>
</tr>
<tr>
<td>L-Nb</td>
<td>Y</td>
<td>86,3 ± 0,6</td>
</tr>
<tr>
<td></td>
<td>N</td>
<td>88,0 ± 0,1</td>
</tr>
<tr>
<td>STD</td>
<td>Y</td>
<td>73,8 ± 1,9</td>
</tr>
<tr>
<td></td>
<td>N</td>
<td>73,7 ± 1,1</td>
</tr>
</tbody>
</table>

Hot ductility of the steels

The hot ductility curves reported in figure 10 for the steel investigated with and without EMS have common features that will be discussed together [15]. A well measurable trough in ductility is noted for both conditions in the temperature range 850-1050°C, which can be accounted for by the onset of intergranular failures at austenite grain boundaries. A recovery in ductility at lower temperatures depends on the formation of a large amount of softer ferrite redistributing the strain originally localised at γ-boundaries whereas, in the high temperature region, ductility increases as soon as the austenite starts to dynamically recrystallize while it is deformed [5]. On the basis of these statements well accepted in literature, the present experimental data are interpreted considering that an increasing content of Nb would promote a larger fraction of precipitates that can retard the onset of the dynamic recrystallization regime, thus increasing the width of the ductility trough toward higher temperatures. At the low-temperature end of the trough, the two Nb-containing steels exhibit a comparable behaviour, in accordance with the comparable composition and therefore with the expected amount of ferrite formed during cooling.
Finally, it is worth considering that the amount of ductility loss was generally comparable for the three steel grades, but slightly worse in the STD samples at temperatures lower than 900°C. Interpretation of such results would deserve accurate TEM analyses to comparatively evaluate the amount of grain boundary precipitates in the samples. However, it might be preliminarily suggested that for the steels investigated, γ-grain boundary embrittlement would be mainly promoted by the AlN phase and by sulphides located at grain boundaries, as reported in literature for similar steel grades [5,17]. The slightly large content of sulfur (see table I) and the higher amount of AlN phase expected for the STD steel (see figure 1) as compared to the Nb-containing grades support these hypotheses.

CONCLUSIONS

The theoretical and experimental analyses carried out in this study allowed to draw the following conclusions.

− Computational analyses on phase constitution of the steels showed that carbonitride precipitation becomes significant, under equilibrium conditions, from a temperature of about 950 and 1100°C for the L-Nb and H-Nb steel, respectively. The AlN phase forms at 1050°C irrespective of steel composition.

− Evolution of the composition of the carbonitride phase as a function of temperature allowed to appreciate that there is a rapid transition from almost-nitride to almost-carbide condition during cooling.

− The as cast structure of the billets demonstrated that EMS was effective in significantly reducing solidification defects and that, when EMS was not active, an increase in Nb content resulted in a greater number of radial cracks in the columnar zone and in a larger extension of the equiaxed central zone.

− The microstructure of the steels investigated was substantially comparable with only slight differences related to the shape of ferrite as a function of steel composition. No difference in preferred crystallographic orientation of the grains was measured in the steels solidified either with and without EMS.

− Hot ductility curves of the steels investigated showed the expected loss in ductility in the temperature range 850-1050°C. Interpretation of data on the basis of chemical composition and of expected precipitates of the steels suggested that Nb can retard the onset of the dynamic...
recrystallization regime by forming carbonitrides, thus increasing the width of the ductility trough toward higher temperatures.

REFERENCES