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The effect of microalloying elements on prior austenite grain growth of low-carbon slab material

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Abstract. The effect of microalloying elements on the prior austenite grain growth of slab materials during slab reheating has been investigated. The investigated materials were laboratory castings with two levels of carbon (0.05 and 0.09 wt.%) and different combinations of microalloying elements, such as V, Nb, Mo and Ti. Experimental results were compared to equilibrium Thermo-Calc® simulations predicting the solubility of different precipitates during slab reheating. Based on slab reheating experiments at temperatures between 1100 – 1250 °C, Ti is the only alloying element to hinder prior austenite grain growth effectively above 1200 °C. For the steel containing high carbon with Nb, V and Ti microalloying, the average prior austenite grain size was less than 50 µm after the slab reheating to 1250 °C, which can be considered very small. According to Thermo-Calc® calculations, stable Ti(CN) precipitates are formed from the liquid and are in solution until 1500 °C. Until 50-60 % of soluble NbC -precipitates, austenite grain size remained relatively small in Nb-alloyed compositions without Ti, but after that grain size increased drastically. For only V-alloyed steels, already at the soaking temperature of 1100 °C hardly any VC precipitates exists based on Thermo-Calc® simulations, and after that grain coarsening can be assumed to be controlled only by the coarsening rate of austenite grains.

1. Introduction
Controlling austenite grain growth during slab reheating can have a major effect on the final properties of thermomechanically rolled steels. It is well known that factors affecting the austenite grain growth are heating rate, reheating temperature and composition of the steels including different microalloying elements [1–6]. The use of microalloying, such as niobium, vanadium and titanium can effectively hinder the austenite grain growth at high temperatures [5,6]. The prior austenite grain size and structure can have a major effect on the formation and morphology of phases formed at lower temperatures, such as martensite, which can further be reflected in the mechanical properties of the steel [7,8]. By controlling the prior austenite grain size, mechanical properties, such as impact toughness of the steel can be significantly improved [7,9].

The solubility of different precipitates can be simulated using Thermo-Calc® software. By comparing the actual experimental data and simulated data from Thermo-Calc®, the accuracy of simulations can be evaluated and can be utilized in further studies.
2. Materials and experimental procedures

The materials used in this investigation are low-carbon steels containing nominally equal amounts of manganese (~1.3 wt.%), silicon (~0.17 wt.%) and chromium (~0.2 wt.%) but various combinations of the microalloying elements vanadium, niobium and titanium. Two different carbon levels are selected (0.05 and 0.09 wt.%) to evaluate the effect of carbon content on the hardness of the steels, see Table 1. All the steels contained V either 0.15 or 0.35 wt.%. Other variants had additions of Nb or Nb+Ti. The experimental steels were vacuum cast into approximately 70 kg ingots at the Tornio Research Centre of Outokumpu in Finland.

Table 1. Chemical compositions of investigated steels (in wt.%).

| Steel           | C  | Si  | Mn  | P   | S   | Cr | Mo | Ti | Nb | V  | N  |
|-----------------|----|-----|-----|-----|-----|----|----|----|----|----|----|
| loC-V           | 0.055 | 0.18 | 1.25 | 0.005 | 0.004 | 0.22 | -  | -  | -  | 0.19 | 0.0054 |
| loC-NbV         | 0.055 | 0.18 | 1.26 | 0.006 | 0.004 | 0.22 | -  | -  | 0.087 | 0.14 | 0.0059 |
| loC-Nb-V-Mo     | 0.054 | 0.18 | 1.26 | 0.006 | 0.004 | 0.22 | 0.09 | -  | 0.089 | 0.15 | 0.0054 |
| loC-NbVTi       | 0.050 | 0.17 | 1.29 | 0.006 | 0.004 | 0.22 | -  | 0.04 | 0.087 | 0.14 | 0.0053 |
| hiC-V           | 0.089 | 0.17 | 1.30 | 0.007 | 0.004 | 0.22 | -  | -  | -  | 0.38 | 0.0051 |
| hiC-NbV         | 0.093 | 0.16 | 1.26 | 0.007 | 0.003 | 0.22 | -  | -  | 0.083 | 0.34 | 0.0060 |
| hiC-Nb-V-Mo     | 0.087 | 0.16 | 1.25 | 0.006 | 0.003 | 0.22 | 0.09 | -  | 0.082 | 0.33 | 0.0063 |
| hiC-NbVTi       | 0.094 | 0.17 | 1.26 | 0.006 | 0.003 | 0.22 | -  | 0.04 | 0.083 | 0.33 | 0.0060 |
| hiC-NbV-LoTi    | 0.095 | 0.16 | 1.25 | 0.007 | 0.006 | 0.22 | -  | 0.01 | 0.081 | 0.33 | 0.0056 |

The effect of microalloying elements on austenite grain growth behaviour was studied. Four different target soaking temperatures were used: 1100, 1150 1200 and 1250 °C. Austenite grain growth behaviour was studied using relatively slow heating rates to simulate the actual slab soaking treatment before laboratory hot rolling trials used in our laboratory. Samples (~15x15x30 mm) were heated along with the furnace reaching the peak temperature after ~2 hours prior to water quenching to room temperature. After heat treatments, samples for light optical microscopy (LOM) and laser scanning confocal microscopy (LSCM) were prepared using picral etching and average mean linear intercept grain sizes were measured using a minimum of 200 grains from each sample. Thermodynamic and kinetic simulations including equilibrium phase diagrams, were executed using Thermo-Calc® (software version of TC2017a) with the TCFE9 thermodynamic database for Fe-based alloys.

3. Results and discussion

3.1 Prior austenite grain structures

Figure 1 presents the measured average austenite grain sizes after the ~2 hours slab soaking treatments in the range 1100-1250 °C. At the soaking temperature of 1150 °C, clear differences in grain sizes among the investigated steels started to occur, 1100 °C producing relatively small grain sizes in all cases, as seen in Figure 1. At the soaking temperature of 1150 °C, the addition of Nb to V-alloyed steel produced a clearly smaller grain size, as seen in Figure 2. At the soaking temperatures higher than 1150°C, only Ti-alloying hindered austenite grain growth effectively, producing significantly small grain sizes even at 1250 °C. In the case of the hiC steels, surprisingly both titanium contents produced small grain sizes, even though Ti/N ratios are different, hiC-Nb-V-Ti steel having ratio of ~6.7 and hiC-Nb-V-LoTi a ratio of ~1.8. It is known that generally the best effect of TiN particles is achieved with a Ti/N ratio close to the stoichiometric 3.42. Beyond the stoichiometric ratio the growth of TiN particles is greater leading to less effective austenite grain pinning [10,11]. Based on our results, both Ti/N ratios of ~1.8 and ~6.7 produced fine austenite grain structure even at the 1250 °C soaking temperature.
Figure 1. Mean austenite grain sizes based on more than 200 grains after heating at soaking temperatures 1100-1200 °C.

Figure 2 presents the prior austenite grain structures (LSCM) of the hiC steels with addition of vanadium, vanadium-niobium and vanadium-niobium-low titanium at soaking temperatures of 1150 °C and 1250 °C. The effect of niobium at 1150 °C can be clearly seen by comparing Figures 2a and 2b. However, at soaking temperature of 1250 °C, grains with the same size were discovered with average size of ~300 microns in case of hiC-V and hiC-Nb-V steels. Comparing Figures 2c and 2f to Figures 2a-2d, the hindering effect of titanium on grain growth is clearly evident even at the high temperature of 1250 °C.

Figure 2. Prior austenite grain structure of a) & d) HiC-V, b) & e) HiC-Nb-V, c) & f) HiC-Nb-V-loTi after heating to 1150 °C (upper row) and 1250 °C (lower row). Picral etching.

It is well known that in certain temperatures abnormal grain growth can occur due to the dissolution of precipitates leading to a non-uniform grain structure [12,13]. For the Nb-alloyed steels without Ti, the occurrence of abnormal grain growth started to be evident at 1150 °C, although the average grain
size was relatively small. Already at 1200 °C, the grain size of the Nb-alloyed steels without Ti was uniform and large indicating that the temperature range for strong abnormal grain growth was approximately 1150 °C ± 20 °C. Figure 3 presents the austenite grain structure of the hiC-Nb-V-Mo steel at the soaking temperatures of 1150 °C and 1200 °C. The non-uniform grain structure has started to form at 1150 °C, as seen from Figure 3a. Only for V-alloyed steels, the abnormal grain growth was not clearly evident in the temperature range of 1100 °C to 1250 °C implying that abnormal grain growth occurs at temperatures lower than 1100 °C.

![Figure 3. Prior austenite grain structure of HiC-Nb-V-Mo steel at soaking temperature of a) 1150 °C, b) 1200 °C. Picral etching.](image)

3.2 Thermo-Calc simulations

Thermo-Calc simulations were executed for all investigated compositions to simulate the dissolution of different precipitations under equilibrium conditions. The results of the simulations were compared with the results of actual soaking treatments, which can be assumed to be close to equilibrium conditions. Figure 4a and 4b presents the dissolution of precipitates for loC-Nb-V and loC-Nb-V-Ti steels. It can be seen that vanadium carbides dissolve already at ~800-900 °C (lines 5 & 4) depending on the Ti content, and niobium carbides at ~1220 °C (lines 4 & 3). Figure 4b shows that Ti(CN) precipitates only dissolve above 1200 °C (line 5). Figure 4c and 4d presents the dissolution of precipitates for the hiC-Nb-V and hiC-Nb-V-Ti steels. Higher carbon content increased the dissolution temperatures of all precipitates. Alloying with 0.04 wt.% Ti reduced the dissolution temperature of VC precipitations by more than 100 °C, as seen from Figures 4c and 4d (lines 4 & 3). Table 2 summaries the solubility temperatures of the different precipitates. Higher carbon content produces more stable carbides leading to higher solvus temperatures. The effect of VC carbides hindering the grain growth can be considered to be practically limited to temperatures less than 1100 °C, as seen from the dissolution temperatures (Table 2). Above 1100 °C, the austenite grain growth in the case of the V-alloyed steels, is controlled by the austenite grain growth rate without any pinning precipitates. Based on Thermo-Calc simulations, the low titanium content produced almost three times less Ti(CN) compared to the higher titanium content (0.015 vs. 0.04 wt.%), however it was enough to hinder the austenite grain growth effectively, as seen from Table 2.
Figure 4. Thermo-Calc simulations for a) loC-Nb-V, b) loC-Nb-V-Ti, c) hiC-Nb-V, d) hiC-Nb-V-Ti steels.

Table 2. Solvus temperatures of different precipitates based on Thermo-Calc.

| Steel          | Solvus temperature [°C] |
|----------------|-------------------------|
|                | VC | NbC | Ti(CN) |
| loC-V          | 1085 | -  | -      |
| loC-Nb-V       | 950  | 1210 | -     |
| loC-Nb-V-Mo    | 950  | 1220 | -     |
| loC-Nb-V-Ti    | 800  | 1200 | 1500 |
| hiC-V          | 1135 | -  | -      |
| hiC-Nb-V       | 1060 | 1235 | -     |
| hiC-Nb-V-Mo    | 1090 | 1235 | -     |
| hiC-Nb-V-Ti    | 895  | 1220 | 1500 |
| hiC-Nb-V-low Ti| 1010 | 1245 | 1500 |

Figure 5 shows the correlation between measured prior austenite grain sizes and calculated amount of NbC in solution. NbC precipitates hinder grain growth effectively until 50-60 % of NbC precipitates are in solution, as seen in Figure 5. The abnormal grain growth was clearly discovered at soaking temperature of 1150 °C in case of Nb-alloyed steels without titanium, and abnormal grain growth was
assumed to be strongest in the temperature range of 1130-1170 °C. Based on the Thermo-Calc simulations, in this temperature range, the amount of NbC in solution is approximately 40-70 %. When the amount of NbC in solution is less or higher than this range, the grain growth phenomenon changes from abnormal to normal grain growth.

![Figure 5. Measured grain sizes vs. calculated NbC in solution.](image)

4. Summary
The effect of microalloying elements on the prior austenite grain growth of slab materials during slab reheating have been investigated. The investigated materials were laboratory castings with two levels of carbon (0.05 and 0.09 wt.%) and different combinations of the microalloying elements V, Nb, Mo and Ti. Based on the results, the following conclusions can be made:
- With both carbon contents, niobium effectively hinders austenite grain growth up to the soaking temperature of 1150 °C. Above 1150 °C, more than 60 % of niobium is in solution based on Thermo-Calc simulations, and no longer able to prevent grain growth.
- Titanium inhibited the austenite grain growth even at the soaking temperature of 1250 °C. For hiC steel, both low (~1.8) and high (~6.7) Ti/N ratios produced small grain sizes.
- Although Thermo-Calc simulations showed that NbC solvus temperatures were higher than 1200 °C, their grain boundary pinning ability disappears at lower temperatures, where more than ~60 % of the NbC is in solution.
- Abnormal grain growth is associated with the dissolution of NbC precipitates. For Nb-alloyed steels without Ti, the temperature range for abnormal grain growth seemed to be at approximately 1130-1170 °C. Thermo-Calc showed that in this temperature region, 40-70 % of NbC precipitates are in solution.

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