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The effect of microalloying on the sheared edge ductility of ferritic steels

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Abstract. Sheared edge ductility plays a critical role in many automotive and structural applications. The effect of microalloying elements on the microstructure, mechanical properties, sheared edge quality and hole expansion properties of six thermomechanically laboratory hot rolled and coiled, approximately 4 mm thick, precipitation hardened ferritic steels have been investigated. The studied V-microalloyed steels had low carbon contents, 0.05 and 0.09 wt.\%, and, in some cases, microalloying additions of either Nb or Nb+Ti. Yield strengths were in the range 500 MPa to 750 MPa and hardness values between 176 and 260 HV10 depending on the carbon content and level of precipitation strengthening. Detailed microstructural analysis using laser scanning confocal microscopy (LCSM), FESEM and FESEM-EBSD revealed that all the steels consisted of mainly single-phase ferrite. FESEM analysis revealed the presence of relatively coarse precipitates at grain boundaries and inside some of the ferrite grains, but the number density of visible precipitates varied greatly between grains. The addition of Nb or Nb+Ti reduced the ferrite grain size, especially the size of the largest grains as defined by the 90th percentile in the cumulative grain area distribution (d\textsubscript{90}). Mean hole expansion (HE) ratios varied from 39 % to 76 % with the highest values associated with the lowest carbon contents. Despite raising the strength, microalloying with Nb or Nb+Ti had little if any detrimental effect on HE ratios compared to the V-microalloyed variants without Nb or Ti, presumably as a result of the grain refinement. Titanium nitrides acted as void nucleation sites close to the sheared edge. Microalloying with Nb and Nb+Ti led to a decrease in the depth of the shear affected zone (SAZ) and a smaller difference in hardness between the base material and the SAZ.

1. Introduction
Increasing demands for safety, reduced fuel consumption and emissions in the automotive and transportation industry have led to the increased use of high-performance materials in the last two decades. The most used solution has been high-strength steels (HSS) as they provide an excellent combination of weight saving potential and cost efficiency. Higher strength allows the use of thinner wall thicknesses and lighter designs. Part production is also more cost-efficient using high-strength steels compared to alternative material choices like aluminium or composites. However, increasing strength usually means lower ductility and huge efforts have been made to develop the formability of HSS.

In the automotive industry, hot-rolled steel with excellent combinations of strength and ductility as measured by elongation to fracture and hole expansion capacity can be used to increase the performance of suspension parts. It has been shown that heavily precipitation hardened fine-grained ferrite can produce better strength – elongation – hole expansion combinations than existing automotive advanced high-strength steels (AHSS) [1]. The improved hole expansion capacity of a precipitation strengthened single-phase ferritic microstructure is due to the absence of micron-sized hard microstructural components that tend to initiate cracking in the so-called multiphase advanced high-strength steels.
(AHSS). Generally, the edge crack sensitivity is affected by the microstructure of the steel [2,3] and the general quality of the sheared edge [4].

In order to achieve high strength ferritic steel, microalloying is needed for effective precipitation strengthening. The most commonly used microalloying elements for this purpose are vanadium, niobium, titanium and molybdenum, and many studies have shown the effectiveness of these microalloying elements [1,5–8]. This paper explores the effect of vanadium, niobium and titanium on the microstructure, mechanical properties, sheared edge quality and hole expansion properties of laboratory hot rolled, furnace heat treated approximately 4 mm thick steel plates with various carbon contents.

2. Materials and experimental procedures

The investigated materials used in this investigation were 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 were 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. 120 x 80 x 55 mm pieces of the castings were homogenized at 1250 °C for 2 hours and thermomechanically rolled to approximately 4 mm thick plates using 6 passes with approximately 20 % reduction in each pass. The temperature of the samples during rolling was monitored using thermocouples placed in holes drilled in the edges of the samples to the mid-width at mid-length. The finish rolling temperature was ~800 °C after which the rolled strips were air cooled to ~650 °C and transferred to a furnace at 630 °C. The furnace was the switched off to allow furnace cooling to room temperature thereby simulating the industrial cooling of coiled strip.

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

| Code   | C   | Si  | Mn  | P   | S   | Cr  | 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-NbVTi| 0.05 | 0.17 | 1.29 | 0.006 | 0.004 | 0.22 | 0.04 | 0.087 | 0.14 | 0.0053 |
| hiC-V  | 0.089 | 0.17 | 1.3  | 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 | 0.006 |
| hiC-NbVTi| 0.094 | 0.17 | 1.26 | 0.006 | 0.003 | 0.22 | 0.04 | 0.083 | 0.33 | 0.006 |

For microstructure analysis, samples were etched using 2 % Nital solution for approximately 15-30 seconds and examined using light optical microscopy (LOM), laser confocal scanning microscopy (LCSM) and field emission scanning electron microscopy combined with electron backscatter diffraction (FESEM-EBSD). EBSD analyses were carried out using an accelerating voltage of 15 kV, and a step size of 300 nm. The area scanned was ~150 x 150 microns.

Tensile tests were performed using 4 mm thick longitudinal flat specimens according to the standard ISO 6892-1:2016 [9]. Three specimens were used for each steel.

Hole expansion (HE) tests were carried out using an Erichsen formability research testing machine according to ISO 16630:2017 [10]. The HE test consists of two steps. The 10mm punched holes have been shear cut with a 11.0 mm cutting die inside diameter with a resulting 12.5 % cutting clearance for 4 mm thick materials in conformity with ISO16630 requirements. The second step is the forcing of a conical expander tool into the pre-punched hole at 15 mm/min until a crack appears and extends through the test piece thickness. The limiting hole expansion ratio is obtained by measuring the expanded hole diameter in two perpendicular directions and expressing the increase in hole diameter as a percentage of the original hole diameter as described in the standard. For each material, three HE tests were carried out to characterize the scatter of the results.
3. Results and discussion

3.1. Mechanical properties and microstructures

Table 2 presents the average hardness values of hot-rolled and furnace cooled samples. For the low-C steels, hardness values were in the range ~175-220 HV10, and for the high-C steels ~200-255 HV10. The addition of niobium increased the hardness values clearly with both carbon levels, and the addition of Ti to Nb-V steels did not bring about any further increase in hardness. Based on these results, the level of V-alloying was insufficient to produce strong precipitation strengthening.

Regarding the tensile properties, Table 2 shows that the low-C steels achieved yield strength values of 428-577 MPa, while the high-C steels reached higher values in the range 537-644 MPa. Tensile strength values correlated well with the measured hardness values, and the ratio between hardness and tensile strength was 2.9-3.2. Total elongation values were in the range 13.6 – 19.9 % with the lower elongation values being associated with the higher tensile strengths. The achieved strength values were slightly lower than could be expected. Funakawi et al. [1] reported more than 800 MPa tensile strength with 0.04 wt.% carbon steels produced with a similar process route. The lower strengths in the present case are probably due to ineffective precipitation strengthening. Funakawi et al. [1] reported that precipitates were formed by interphase precipitation and were very fine with a diameter of 2-3 nm, which would result is considerable precipitation strengthening [11].

Figs. 1a and 1b present examples of the overall microstructures achieved after laboratory scale hot rolling and simulated coiling procedure. They were mainly ferritic with some pearlitic regions. Also random clusters of precipitates with a precipitation size of ~100-200 nm were apparent. Ferritic microstructures of investigated steels were locally slightly elongated due to the relatively low finish rolling temperature. Higher magnification, FESEM images of the steels loC-Nb-V and hiC-Nb-V-Ti steels are presented in Figs. 1c and 1d. Most of the ferritic grains contained a high density of precipitates, presumably M(CN) where M represents one or more of the microalloying elements. Precipitate diameters range from tens of nanometers to several hundred nanometers. Besides the intragranular precipitates, quite coarse precipitates can be seen at ferrite grain boundaries, which can be considered detrimental to ductility. During rolling, M(CN) precipitation can occur in the austenite or in any ferrite formed near the low finish rolling temperature, for example (see below). Also, precipitates can form at the ferrite - austenite interface or randomly in the ferrite. The large size of the precipitates seen in Fig. 1 implies precipitation at high temperatures and/or strong precipitate coarsening during the furnace cooling from 630 °C. A clear difference in the distribution of precipitates between ferrite grains can be seen in Figs. 1c and 1d: there are darker grains with a low density of visible precipitates and lighter grains with a higher density of visible precipitates. The reasons for these differences as well as the times and modes of M(CN) precipitation have yet to be established.

| Steel       | $R_{el}$ [MPa] | $R_m$ [MPa] | $A_e$ [%] | $A_{70}$ [%] | $R_{p0.2}/R_m$ [%] | HV10   | Rm/HV10 |
|-------------|----------------|-------------|-----------|--------------|---------------------|--------|---------|
| loC-V       | 428            | 515         | 12.4      | 19.9         | 85.9                | 176    | 2.9     |
| loC-Nb-V    | 555            | 639         | 11.5      | 18.2         | 88.0                | 221    | 2.9     |
| loC-Nb-V-Ti | 577            | 657         | 10.9      | 17.2         | 88.8                | 220    | 3.0     |
| hiC-V       | 537            | 640         | 9.7       | 15.0         | 85.7                | 202    | 3.2     |
| hiC-Nb-V    | 628            | 729         | 9.2       | 13.6         | 87.3                | 256    | 2.9     |
| hiC-Nb-V-Ti | 644            | 728         | 10.6      | 14.7         | 89.6                | 250    | 2.9     |
Table 3 shows mean equivalent circle diameter (ECD) ferrite grain sizes based on EBSD analyses. The addition of Nb can be seen to reduce the average ferrite grain size, and the size of the largest grains, as defined by the 90th percentile in the cumulative grain area distribution, d90%. The size of the largest grains in the microstructure, i.e. d90%, is more important than the mean size as far as impact toughness is concerned [12]. The grain maps constructed from EBSD and given in Fig. 2 also reveal how Nb additions to the V-microalloyed composition refine the grain for both carbon contents.

**Table 3.** Mean ECD grain sizes, d90% values and percentage of low-angle (2.5-15°) and high-angle (>15°) boundaries determined from the EBSD acquisitions (>200 grains).

| Steel     | Mean grain size [µm] | d90% [µm] | 2.5-15° [%] | >15° [%] |
|-----------|----------------------|-----------|-------------|----------|
| loC-V     | 2.9                  | 15.8      | 41.4        | 58.6     |
| loC-Nb-V  | 2.1                  | 9.4       | 32.2        | 67.8     |
| loC-Nb-V-Ti | 2.2             | 9.5       | 27.0        | 73.0     |
| hiC-V     | 4.0                  | 12.0      | 7.3         | 92.7     |
| hiC-Nb-V  | 2.5                  | 9.1       | 14.9        | 85.1     |
| hiC-Nb-V-Ti | 2.3             | 8.5       | 14.3        | 85.7     |
Figure 2. EBSD grain maps of a), c) loC-V, b) loC-Nb-V, d), f) hiC-V, e) hiC-Nb-V steels. Grain boundaries (red: 2.5-15°, blue: >15°)

The percentages of low-angle (2.5-15°) and high-angle (>15°) boundaries were determined from the EBSD data are presented in Table 3. It can be noticed that the low-C steels generally have a higher percentage of low-angle boundaries compared to the high-C steels, see Figs. 2e and 2f. Presumably, the differences in the substructure of the ferrite grains are due to the formation, deformation and recovery of ferrite at the low finish rolling temperature. A_3 temperatures were calculated using equation presented by Andrews [13]. The difference in A_3 temperatures between the loC-V and hiC-V steels were 36 °C (798 °C for hiC-V- and 835 °C for loC-V). The finish rolling temperature of ~800 °C is therefore lower than A_3 for the low-carbon steels, which is, of course higher than the equilibrium A_3.

3.2. Hole expansion tests
The results from the hole expansion tests can be seen in Fig. 3. The low-C steels achieved average HE values of 69 -76 %, and the high-C steels 39-52 %. Despite their higher strength and hardness, the Nb-V alloyed steels produced almost equal HE values as the V-alloyed steels without Nb, which is presumably a result of the smaller and more uniform ferritic grain structure brought about by Nb. Adding Ti to the Nb-V steels had no significant effect on HE, as can be seen from Fig. 3. It is well known that the standard hole expansion test can produce large scatter in the test results [14]. However, excluding the loC-V steel, the scatter of the present HE results were relatively small. Additionally, the HE tests were carried out for laboratory hot-rolled materials with occasionally heavily scaled surfaces, which can affect the scatter and decrease HE values.
Microstructural investigations were carried out on the hole expanded samples in order to investigate the cracking mechanisms, including void formation and the critical microstructural factors. It is known that void formation during deformation is controlled by microstructural factors such as carbides, inclusions and phase boundaries [15–17]. Fig. 4 presents the microstructures of investigated steels in the highly deformed area close to the punched and expanded hole.

Figs. 4a and 4c show the microstructure of the steel LoC-Nb-V in the highly deformed area. Only small voids can be found (Fig. 4c), mainly around inclusions, that have not grown and linked into cracks. The relatively soft and ductile ferrite matrix hinders crack initiation, even though voids form at inclusions. In the deformed zone of the steel hiC-Nb-V, long cracks originating at inclusions can be found, as seen in Figs. 4b, 4d and 4f. Cracks may have propagated along the coarse grain boundary precipitates. As seen from Fig. 4e, besides inclusions, TiN particles also nucleated voids in the steel hiC-Nb-V-Ti. This is due to the large hardness difference between TiN and the surrounding matrix. However, comparing the measured HE values, it seemed that TiN did not decrease the HE value. It can be assumed that the effect of TiN on hole expansion and crack propagation is summary of the size and the amount of the TiN particles and also the ductility of the surrounding matrix. For ferritic steels, the ductile matrix decreased the effect of TiN, although better results could be still expected without TiN particles.

Figure 4. Microstructures of HE samples, a) & c) LoC-Nb-V, b), d) & f) hiC-Nb-V, e) hiC-Nb-V-Ti steels.
3.3. SAZ measurements
The punching process prior to hole expansion generates plastic deformation and work hardening in the steel beneath the hole edge face. This region constitutes the shear affected zone (SAZ), which is known to affect the stretching properties of the edge [18]. Therefore, the SAZs of the investigated steels were characterized. Table 4 shows various cut edge and SAZ parameters. Also, Fig. 5 shows the resulting material hardness as a function of distance below the cut edge faces. SAZ depths were defined as the width of the zone whose hardness was at least 10% higher than that of the base material. \( \Delta HV \) represents the hardness difference between the base material and that 0.1 mm from the sheared edge face. Table 4 shows that the percentages of fracture and burnish areas were similar for all the steels. Also the rollover percentages were between 4-5% for all steels, giving no clear differences between various compositions. However, the addition of Nb and Ti clearly decreased the SAZ depth and the hardness difference between base material and the SAZ, as seen from Table 4. Based on the results from many different studies, Levy et al. [19] reported that the SAZ depth varies between 25 to 67% of the plate thickness. Therefore, the 6 - 11% values achieved in this study can be considered to be unusually low. It can be assumed that a lower SAZ depth will leave more work hardening capacity for the actual hole expansion leading to an improved hole expansion ratio. Visual inspection of surface quality of the punched holes did not show any major differences between investigated steels, as seen from Fig. 5b.

Table 4. Parameters determined from shear affected zone (SAZ). Percentage values are relative to the plate thickness.

| Steel          | Fracture [%] | Burnish [%] | Rollover [%] | SAZ width [%] | \( \Delta HV \) (0.1-BM) |
|----------------|--------------|-------------|--------------|---------------|---------------------------|
| loC-V          | 53.9         | 40.4        | 4.8          | 32.7          | 150                       |
| loC-Nb-V       | 56.5         | 39.6        | 5.2          | 17.1          | 132                       |
| loC-Nb-V-Ti    | 60.5         | 34.4        | 4.2          | 6.5           | 64                        |
| hiC-V          | 60.6         | 35.2        | 5.4          | 24.1          | 127                       |
| hiC-Nb-V       | 62.8         | 31.1        | 5.3          | 7.1           | 77                        |
| hiC-Nb-V-Ti    | 64.8         | 32.4        | 4.1          | 11.0          | 88                        |

Figure 5. a) Micro-hardness profiles of shear affected zones (SAZ) and b) surface quality of punched holes.

4. Summary and conclusions
Six different V-microalloyed steel compositions with two different carbon contents and additions of Nb and Nb+Ti have been investigated. Laboratory cast material was hot rolled, air cooled to \( \sim 650 \) °C, transferred to a furnace at 630 °C and furnace cooled to simulate the industrial processing of coiled strip.
The target was to achieve a precipitation strengthened ferritic microstructure. The materials were characterized with respect to microstructure, tensile properties, hardness, hole expansion performance and SAZ properties. Based on the results, the following conclusions can be made:

- The investigated ferritic steels achieved tensile strength values of 515-657 MPa in the case of the low-carbon variants, and 640-729 MPa for the high-carbon variants. The relatively high strength values were probably attributable to precipitation hardening mainly during coiling simulation at 630 °C. Microstructural analysis revealed ferritic areas with relatively coarse precipitates combined with precipitate-poor areas. Also, coarse grain boundary precipitation occurred. These observations indicate that interphase precipitation was not the dominant precipitation formation mechanism.
- Additions of Nb and Nb+Ti led to a refinement of the ferritic microstructure. EBSD results revealed that the low-carbon steels had more low-angle boundaries in the ferrite implying that ferrite was partly formed and was deformed during the hot rolling stage.
- Hole expansion tests showed that the low-carbon steels produced higher HE values due to a generally lower hardness. The addition of Nb and Nb+Ti, i.e. the refinement of the ferrite, produced almost equal HE values despite raising the steel hardness. For the Nb+Ti compositions, TiN inclusions as well as other inclusions acted as void nucleation sites.
- Analysing the shear affected zone revealed that Nb and Nb+Ti alloying decreased the depth of the SAZ, which is known to be beneficial for hole expansion.

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