Role of Vanadium Microalloying in Austenite Conditioning and Pearlite Microstructure in Thermomechanically Processed Eutectoid Steels

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Multipass torsion tests were carried out with two eutectoid steels, one microalloyed with vanadium, using different deformation sequences. The aim of the study was to investigate the potency of vanadium in the retardation of recrystallization for accumulating strain in the austenite. The study showed that at certain deformation conditions well defined non-recrystallization temperatures (Tnr) were observed in the vanadium microalloyed steel. As a consequence, an increase in the austenite grain boundary area per unit volume (Sv) was obtained which led to a refinement of the “ferrite unit” size in the pearlite.

KEY WORDS: eutectoid steel; vanadium microalloying; austenite conditioning.

1. Introduction

It is well known that with the application of thermomechanical processing to low carbon microalloyed steels a very fine grained ferrite microstructure develops. These treatments are based on the effect of Nb microalloying on the refinement of the austenite microstructure and the accumulation of strain below the non-recrystallization temperature.1 As a consequence, most studies have concentrated on the austenite evolution, analyzing both the influence of the microalloying elements and the rolling schedules.

With respect to pearlitic steels, the application of thermomechanical processing is less common. Most studies have concentrated on the influence of the cooling rate or undercooling in isothermal transformations on the interlamellar spacing, since this is the main microstructural parameter controlling the strength of the pearlite.2,3 In a similar way, precipitation hardening, mainly by the microaddition of vanadium, has been a complementary tool for improving the strength for a given interlamellar spacing.4 However, in industrial applications where toughness becomes a relevant property, other microstructural parameters also need to be considered. Among these, the most cited variables in the literature are the prior austenite grain size, the colony size and the microstructural unit with the same ferrite orientation, identified as the “ferrite unit”.2–7 This implies that the undercooling during the transformation is not the only parameter that requires control. Among other aspects (such as the cleanness of the steel), the toughness of pearlitic steels is mainly controlled by the size of the “ferrite unit” and the refinement of this microstructural feature is the only way to improve toughness.8–10 Taking into account how the austenite/pearlite transformation occurs, lowering of the “ferrite unit” size requires the refinement of the austenite microstructure.

Similarly to what occurs with low carbon steels, the application of thermomechanical processes leading to deformed austenite prior to transformation would allow “ferrite units” even finer to be obtained, that is to say lower than those achieved by the application of simple thermal treatments. In these conditions, in order to accumulate strain, a microalloying addition is usually required. In a recent work it has been reported that the application of thermomechanical treatments to a 0.03% Nb microalloyed eutectoid steel leads to strain accumulation in the austenite with a significant reduction in the “ferrite unit” for given cooling conditions.8 However for industrial applications one of the limitations in the use of Nb may be the low solubility of this element for the case of high C steels, which can reduce the efficiency of the microalloying addition and also impair hot ductility during continuous casting.

An alternative microalloying element is vanadium. This element can be completely dissolved before starting hot rolling and it has not reported as a factor that can significantly affect hot ductility during continuous casting. Nevertheless, VN or V(C, N) precipitation in austenite is a prerequisite to proceed with strain accumulation, as the vanadium solute drag effect in delaying static recrystallization is quite small compared to other microalloying elements (Nb and Mo).11

Previous studies carried out by Pickering and Garbuz with one and two step hot compression tests demonstrated that it was possible to accumulate strain in high carbon vanadium microalloyed steels.9 They found that the transformation from a fine unrecrystallized austenite led to a finer pearlitic microstructure which could result in an im-
proved toughness.

Nevertheless, there is a lack of knowledge concerning the potential use of vanadium for the accumulation of strain in eutectoid steels and the optimum deformation conditions to achieve this. This paper attempts to analyze the potency of vanadium to accumulate strain in the austenite through the application of thermomechanical treatments.

2. Experimental Procedure

Two eutectoid steels were considered for this study: a plain carbon one and another microalloyed with vanadium, both having the same base composition. The C–Mn steel was commercially produced in bar shape, while the C–Mn–V was cast in the laboratory by adding vanadium after remelting the above C–Mn steel. Afterwards the resulting slabs were hot worked to square ingots. The compositions of the materials are indicated in Table 1.

Multipass torsion tests were carried out to determine the non-recrystallization temperature ($T_{nr}$). Specimens, having a gauge length of $l=12$ mm with a diameter of $2r=6$ mm, were reheated for 15 min at 1200°C. After reheating the samples were deformed applying 24 passes at decreasing temperature in the range 1150–800°C. The measured torque $\Gamma$ and the twist $\theta$ were converted to von Mises equivalent stress ($\sigma$) and strain ($\varepsilon$) with the help of these equations:

$$\sigma = \frac{3.3}{2\pi r^3} \Gamma, \quad \varepsilon = \frac{r \theta}{l^3}$$

The tests were performed using different pass-strains: $\varepsilon=0.2$–0.3–0.4. The strain-rate used in all the tests was $1.5$ s$^{-1}$, and the interpass time at each deformation stage was 10 s. During deformation a cooling rate of 1.5°C/s was applied. In a particular test, the strain per pass, strain-rate and interpass time were held constant. The values of the non-recrystallization temperature were between 85% and 94% of the whole scanned area.

The initial austenite grain size prior to deformation was determined in specimens directly quenched after reheating treatment. In most tests, the specimens were also directly quenched after deformation for the analysis of the austenite microstructure. Some additional tests were carried out applying natural cooling ($\sim 3$°C/s) after deformation for the study of the pearlite microstructure.

The metallographic observation of the samples was done on a surface parallel to the torsion axis at a distance close to the subsurface ($r^*=0.9r$; $r$ being the outer radius of the specimen). The austenite microstructure was characterized by the grain boundary area per unit volume ($S_g$) measured using the mean linear intercept method. This parameter is important in determining the kinetics of pearlite transformation since the grain boundaries provide preferential sites for nucleation. When the grains are equiaxed,$^{13)}$

$$S_g = 2N_L / 2L$$

where $N_L$ and $L$ represent the mean number of intercepts and the mean linear intercept value, respectively. For deformed microstructures, $S_g$ should be a weighted average of the values of $N_L$ in each of the three principal directions.$^{13)}$

Nevertheless, in the present work the number of intercepts was measured without any distinction between these directions. The number of intercepts with a circle was counted independently of the austenite microstructure whether or not it was a recrystallized or deformed one. This means that in the latter case the measurements cannot be taken as real values, although they are useful for comparison purposes.

The pearlite interlamellar ($\lambda$) spacing was measured using the linear intercept method proposed by Underwood.$^{14)}$

For EBSD (electron backscattered diffraction) observations of pearlitic structures, the specimens were polished down to 1 $\mu$m and the final polishing was with colloidal silica. Since the Kikuchi patterns of cementite in pearlite are not resolvable, only diffraction patterns arising from ferrite were considered. The scan step size was 1 $\mu$m long and the total scanned area was about $600 \times 600 \mu^2$.

The mean “orientation unit” size $D_p$ (as equivalent diameter) as well as the size distribution in area were quantified.

In order to index some badly or non indexed isolated points, the grain dilation clean-up method was performed on each scan. Specifically, the type of grain dilation cleanup was a single iteration so that the minimum grain size must have at least 3 points surrounded by 12° boundaries. In the statistics, “orientation units” smaller than 4.5 $\mu$m were rejected, these were “orientation units” with less than 18 points. The area fractions of the “orientation units” considered were between 85% and 94% of the whole scanned area in each case.

3. Results

3.1. Austenite Conditioning

The stress–strain curves obtained for both steels in the 24-pass torsion tests are shown in Fig. 1 for the case of a strain per pass of 0.3. The increase in the stress as the tem-

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Table 1. Chemical composition of the steels (mass %).

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Al</th>
<th>N</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>C–Mn</td>
<td>0.79</td>
<td>0.79</td>
<td>0.21</td>
<td>0.006</td>
<td>0.025</td>
<td>0.28</td>
<td>0.13</td>
<td>0.004</td>
<td>0.011</td>
<td>0.002</td>
</tr>
<tr>
<td>C–Mn–V</td>
<td>0.79</td>
<td>0.83</td>
<td>0.19</td>
<td>0.011</td>
<td>0.019</td>
<td>0.28</td>
<td>0.13</td>
<td>0.007</td>
<td>0.011</td>
<td>0.110</td>
</tr>
</tbody>
</table>

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Fig. 1. Stress–strain curves obtained after multipass torsion for a strain per pass of $\varepsilon=0.3$: (a) steel C–Mn; (b) steel C–Mn–V.
perature drops is clearly evident, as well as a greater tendency towards hardening in the final passes, mainly in the vanadium microalloyed steel. From Fig. 1 the mean flow stress (MFS) corresponding to each pass has been evaluated by numerical integration. The MFS has been defined as the area under each stress–strain curve divided by the pass strain. The results obtained for both steels and different deformation sequences are shown plotted against the inverse absolute temperature in Fig. 2.

Following the procedure of Bai et al., the non-recrystallization temperature, \( T_{\text{nr}} \), can be determined by the change in slope in the MFS vs. \( 1/T \) representation. This change makes it possible to distinguish two different regions, as indicated in Fig. 2(b):

- Region I: where it is supposed that complete recrystallization between passes takes place and the stress increment from pass to pass is only due to temperature drop.
- Region II: where recrystallization between passes is inhibited. The stress increases more rapidly due to both, the temperature drop and the accumulation of strain.

From the graphs in Fig. 2 a different behavior between the C–Mn and the vanadium steel is clearly evident. The plain carbon steel does not show any slope change for the pass-strain of 0.4, denoting that in this case the material completely recrystallizes between passes in the overall temperature range, while for the strains of 0.2 and 0.3 a change in slope after the 16th and 20th pass, respectively, occurs. On the other hand, the vanadium microalloyed steel shows a well defined non-recrystallization temperature for all the deformation conditions. The \( T_{\text{nr}} \) values measured are drawn in Fig. 3 as a function of the strain per pass.

The austenite microstructure obtained for both steels after the application of the different deformation sequences is shown in Fig. 4. Significant differences are observed between both steels and also between the different sequences after the application of deformation. For the C–Mn steel and the lowest pass-strain (\( \varepsilon = 0.2 \)), the austenite microstructure appears slightly elongated, which indicates that some strain has been accumulated in the austenite according to the increase in the mean flow stress during the final passes (\( T_{\text{nr}} = 927^\circ \text{C} \)) observed in Fig. 2(a). For the other two strains (\( \varepsilon = 0.3–0.4 \)) the final austenite microstructure is more or less equiaxed, although for the case of \( \varepsilon = 0.3 \) some degree of accumulated strain is predicted in Fig. 2(a). For the C–Mn–V steel the refinement of the microstructure is clearly evident comparing to the plain carbon steel deformed under similar conditions. For the pass-strains of \( \varepsilon = 0.2 \) and 0.3 the final austenite appears very deformed (nearly unrecrystallized), although increasing the strain to
0.4, the austenite looks like more equiaxed (recrystallized).

The results obtained from the characterization of the austenite microstructures shown in Fig. 4 are indicated in Table 2. In the table the austenite grain size measured after reheating treatment (\(D_0\)) is also included. In general both steels show an increase of \(S_V\) as the applied strain increases, although in the vanadium microalloyed steel the opposite occurs between 0.3 and 0.4 pass-strains. On the other hand, it is observed that for similar deformation conditions the vanadium steel shows significantly larger \(S_V\) values than the plain carbon steel.

### 3.2. Pearlite Microstructure

Figure 5 shows several micrographs of the pearlite microstructure observed in both steels after deformation and natural cooling to room temperature (\(\approx 3^\circ C/s\) at 700°C), corresponding to pass-strains of \(\varepsilon=0.3\) and 0.4, Figs. 5(a, c) and 5(b, d), respectively. In these microstructures the interlamellar spacing and the hardness have been quantified. The results are shown in Table 3. In all cases some ferrite fraction decorating prior austenite grain boundaries is observed. For the C–Mn steel this fraction is somewhat larger for the pass-strain of \(\varepsilon=0.4\) compared to \(\varepsilon=0.3\), whereas the opposite occurs in the C–Mn–V steel, where more ferrite is observed at \(\varepsilon=0.3\). Moreover it is observed that the ferrite grains are significantly smaller in the C–Mn–V steel, compared to the plain carbon steel.

These microstructures were analyzed by EBSD to determine the size of the “ferrite unit”. Figure 6 shows examples of the grain maps obtained by OIM (Orientation Imaging Microscopy) analysis from the microstructures that came about after the application of both deformation schedules. An important point for these measurements is the misorientation criterion used to define the “orientation unit”. In Ref. 8) a 15° misorientation criterion was considered since it produced reasonable results when the OIM measurements were compared to the “facet size” measured from the fracture surfaces. However in Ref. 10), when several V microalloyed steels were studied, the application of the above criterion led to an “orientation unit” mean size larger than the “facet size” measured on fracture surfaces. In this case a 12° misorientation criterion brought better results. In the present study the fracture behavior was not looked into, but taking into account the similarity in composition of the ma-

**Table 2.** Microstructural parameters measured in the austenite after reheating treatment and after deformation sequence.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Test</th>
<th>Initial austenite grain size (D_0) ((\mu m))</th>
<th>(\varepsilon_{pas})</th>
<th>Austenite microstructure after deformation (D_f) ((\mu m))</th>
<th>(S_V = 2N_1) (mm(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>C–Mn</td>
<td>F1</td>
<td>135</td>
<td>0.2</td>
<td>unrecrystallized</td>
<td>132</td>
</tr>
<tr>
<td></td>
<td>F2</td>
<td></td>
<td>0.3</td>
<td>recrystallized (D_f=14 \mu m)</td>
<td>143</td>
</tr>
<tr>
<td></td>
<td>F3</td>
<td></td>
<td>0.4</td>
<td>recrystallized (D_f=13 \mu m)</td>
<td>154</td>
</tr>
<tr>
<td>C–Mn–V</td>
<td>F-V1</td>
<td>105</td>
<td>0.2</td>
<td>unrecrystallized</td>
<td>204</td>
</tr>
<tr>
<td></td>
<td>F-V2</td>
<td></td>
<td>0.3</td>
<td>unrecrystallized</td>
<td>292</td>
</tr>
<tr>
<td></td>
<td>F-V3</td>
<td></td>
<td>0.4</td>
<td>unrecrystallized</td>
<td>220</td>
</tr>
</tbody>
</table>

**Table 3.** Interlamellar spacing and hardness measurements in both steels after natural cooling (\(\approx 3^\circ C/s\)) following multipass deformation.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Pass-strain</th>
<th>Interlamellar spacing, (\lambda) ((\mu m))</th>
<th>Hardness (HV1)</th>
<th>(D_f) ((\mu m))</th>
</tr>
</thead>
<tbody>
<tr>
<td>C–Mn</td>
<td>(\varepsilon=0.3)</td>
<td>0.138 ± 0.025</td>
<td>278</td>
<td>10.8</td>
</tr>
<tr>
<td></td>
<td>(\varepsilon=0.4)</td>
<td>0.135 ± 0.024</td>
<td>279</td>
<td>11.0</td>
</tr>
<tr>
<td>C–Mn–V</td>
<td>(\varepsilon=0.3)</td>
<td>0.130 ± 0.016</td>
<td>348</td>
<td>7.6</td>
</tr>
<tr>
<td></td>
<td>(\varepsilon=0.4)</td>
<td>0.100 ± 0.012</td>
<td>377</td>
<td>9.7</td>
</tr>
</tbody>
</table>

**Fig. 5.** Pearlite microstructure observed in both steels after natural cooling (\(\approx 3^\circ C/s\) at 700°C): (a) and (b) C–Mn steel for pass-strains of \(\varepsilon=0.3\) and \(\varepsilon=0.4\), respectively; (c) and (d) idem for C–Mn–V steel.
terials used in this study to those studied in Ref. 10), the measurements were also carried out with a tolerance criterion based on 12° change in orientation between adjacent units. Therefore, the “ferrite units” were defined as regions completely surrounded by 12° boundaries. It is supposed that these regions may become single cleavage facets after fracture. Nevertheless, it should be pointed out that within these areas there can be significant orientation changes between points that are far away from each other. In fact the orientation units are made up of smaller areas that are slightly misoriented with respect to their neighboring areas, but the misorientation between non-neighboring points could be large (even more than 25°) within the same “orientation unit”.

From the maps in Fig. 6 the “orientation unit” size was measured by quantitative metallography methods, taking the mean equivalent diameter as the parameter for measurement. In Fig. 7 the size distributions obtained in all cases are represented. The mean values are indicated in Table 3.

4. Discussion

4.1. Austenite Conditioning

From Table 2 it is observed that for similar reheating conditions the austenite grain size reached in the C–Mn steel is slightly larger than that observed in the vanadium steel. This means that vanadium may have an effect as inhibitor of the austenite grain growth during this treatment. This effect has been previously reported in eutectoid steels.15

The results of Fig. 2 and Fig. 4 confirm that during the multipass deformation schedule the behavior of the austenite microstructure significantly changes when vanadium is added. Similarly, the results of Fig. 3 show a significant increase in $T_{nr}$ values at the considered pass strain interval. In a multipass deformation schedule, as occurs in industrial rolling, the softening and hardening mechanisms, including solute drag and strain induced precipitation, and the interactions between them will determine the $T_{nr}$ temperature. These interactions can be better evaluated via the calculation of the fractional softening that occurs between consecutive passes. As this softening must be determined under anisothermal conditions, the following equation was used16):

$$\text{FS} (%) = \frac{\sigma_y^i - \sigma_y^{i+1}}{\sigma_y^o} \times 100 \quad \ldots(3)$$

where $\sigma_y^i$ and $\sigma_y^{i+1}$ are the maximum and the yield stresses for both, the $i$-th (at temperature $T$) and the $(i+1)$-st (at temperature $T_{i+1}$) passes respectively, while $\sigma_y^o$ and $\sigma_y^{1st}$ are the yield stresses of a fully recrystallized material for the $i$-th and $(i+1)$st passes. In the present case the yield stresses were determined by the 5% total strain method.17 The stresses $\sigma_y^o$ and $\sigma_y^{1st}$ are determined from the following relationship which is derived from the values of the yield stresses measured in the stress–strain curves corresponding to the range of complete recrystallization.

$$\sigma_y (\text{MPa}) = -252 + \frac{417\,000}{T(K)} \quad \ldots(4)$$

The values of fractional softening calculated using Eq. (3) are plotted as a function of temperature in Fig. 8. In the figures the $T_{nr}$ values determined from Fig. 2 are also indicated. In the three conditions the differences between both steels are evident with a well defined deviation from the 100% fractional softening at higher temperatures in the vanadium microalloyed steel.

The presence of a $T_{nr}$ may be the result of the unavailability for recrystallization to be completed between passes, due to short interpass times and/or low pass-strains or to strain induced precipitation of carbonitrides. For the C–Mn steel $T_{nr}$ is observed at $\varepsilon=0.2$ and 0.3 pass-strains. In this steel precipitation is not expected and therefore, any strain accumulation should be related to the unavailability for recrystallization to be completed between passes as the temp-

![Fig. 6. Brain maps corresponding to the microstructures obtained after deformation: (a) and (b) C–Mn steel for pass-strains of $\varepsilon=0.3$ and $\varepsilon=0.4$, respectively; (c) and (d) idem for C–Mn–V steel.](image)

![Fig. 7. “Orientation unit” size distributions.](image)
perature drops. In the case of $\varepsilon=0.4$, recrystallization time diminishes and the process may be completed in the time between passes, therefore $T_{nr}$ is not observed.

In order to confirm this, the evolution of the austenite microstructure after each deformation pass was predicted following the structure of the model described in Ref. 18). The equations determined by Kuziak et al.\textsuperscript{19) for eutectoid steels were selected to quantify the time for static recrystallization, the recrystallized grain size and the grain growth after complete recrystallization. The results are shown in Fig. 8.

As can be observed, the model reasonably predicts the evolution of experimental values of the fractional softening in the case of the C–Mn steel, confirming that the increase of accumulated strain as temperature decreases with a well defined $T_{nr}$ value is a consequence of the unavailability for recrystallization to be completed between passes for $\varepsilon=0.2$ and 0.3 testing conditions.

In contrast, for the C–Mn–V steel the significant deviation in the fractional softening from the plain C–Mn steel can be only due to strain induced precipitation, as vanadium solute drag exerts a minor effect in delaying recrystallization kinetics.\textsuperscript{11,20)}

Figure 9 shows a micrograph of the vanadium precipitates found in a carbon replica extracted from a sample immediately quenched after deformation following the schedule of test F-V2 (see Table 2), that corresponds to a pass-strain of $\varepsilon=0.3$, analyzed by TEM. The corresponding particle size distribution is also indicated in the figure. A relatively high density of precipitates is observed with a mean diameter of 22 nm. These observations denote that vanadium has precipitated in austenite during deformation and this precipitation through the retardation of recrystallization it produces would be the responsible for the $T_{nr}$ observed under these conditions in the microalloyed steel.

The interaction between recrystallization and precipitation can be analyzed in terms of the driving forces related to both mechanisms, $F_{REX}$ and $F_{PIN}$, respectively.\textsuperscript{21,22)} The net driving force for recrystallization is given by the difference between $F_{REX}$ and $F_{PIN}$. When the pinning force exerted by the precipitates overcomes the stored energy of deformation, $F_{PIN}=F_{REX}$, recrystallization stops. The stored energy of deformation is often expressed as:

$$F_{REX} = \frac{1}{2} \rho \mu b^2 = \frac{1}{2} \left[ \frac{\sigma - \sigma_y}{M \mu b} \right]^2 \mu b^2 \quad \ldots \ldots \ldots \ldots (5)$$

where $\mu$ is the temperature-dependent shear modulus of austenite, $b$ is the Burgers vector and $\rho$ is the dislocation density associated with deformation, which can be estimated as described in the above equation,\textsuperscript{23} with $\sigma$ and $\sigma_y$ the flow stress and the yield stress at the deformation temperature, respectively, $M$ the Taylor factor (3.1 for FCC crystals) and $\alpha$ a constant taking a value of approximately 0.15.

Different models have been proposed for the calculation of the pinning force $F_{PIN}$, depending on the assumptions made about which particles interact with the motion of the
boundary. In the present case the subgrain boundary model has been taken into account:

\[
F_{\text{PIN}} = \frac{3\gamma f_v l}{2\pi r^2}
\]

where \(\gamma\) is the austenite grain boundary energy \((0.8 \text{ J/m}^2)\), \(f_v\) the precipitate volume fraction, \(r\) the precipitate radius and \(l\) the average subgrain boundary intercept distance (taken as 0.5 \(\mu\m\)).

In the determination of \(F_{\text{REX}}\), the stress–strain experimental curves of the multipass torsion tests were considered, while in the calculation of \(F_{\text{PIN}}\) the measured mean particle size of 22 nm \((r=11 \text{ nm})\) was taken. For the precipitate volume fraction, \(f_v\), the VN equilibrium volume fraction at the corresponding temperature was calculated using the solubility product equation of Irvine et al.\(^{25}\) If \(T_{\text{sw}}\) is associated with precipitation it means that close to this temperature \(F_{\text{PIN}}\) should become larger than \(F_{\text{REX}}\). In this study it was found that about 30% of the total possible equilibrium fraction was required to have an \(F_{\text{PIN}}\) larger than \(F_{\text{REX}}\) when the \(T_{\text{sw}}\) was reached. The evolution of \(F_{\text{REX}}\) and \(F_{\text{PIN}}\), the latter calculated considering that 30% of the equilibrium fraction is involved in pinning for a precipitate radius of \(r=11 \text{ nm}\), is shown as a function of temperature in Fig. 10 for \(\epsilon=0.2\) and 0.4. In both cases the conditions corresponding to interaction between \(F_{\text{REX}}\) and \(F_{\text{PIN}}\) occur in a temperature range close to the measured value of \(T_{\text{sw}}\).

Frequently the time to the “start” of precipitation is defined as the time to 5% of equilibrium precipitation,\(^{26}\) and this time is usually identified with the situation where \(F_{\text{PIN}}>F_{\text{REX}}\), bringing about a plateau in softening curves. Nevertheless, fractions between 6 and 13% were reported in Ref. 27) and between 14 and 27% in Ref. 28) for Nb(C, N) precipitation. It must be taken into account that in the present study precipitation was analyzed after the last pass, i.e. relatively far from the \(T_{\text{sw}}\) and some coarsening of the precipitates had possibly occurred. At initial stages of precipitation, close to the \(T_{\text{sw}}\) temperatures, a smaller precipitate size is expected, therefore lower volume fractions would be enough to stop recrystallization under these conditions. It has been estimated that a precipitate mean diameter of about 8 nm would be necessary with a volume fraction of 5% of the equilibrium precipitation. This is reasonable taking into account previous experiences relative to the size of precipitates found at initial stages of strain induced precipitation.\(^{27–29}\)

In the same figure the \(F_{\text{PIN}}\) corresponding to a nitrogen content of 40 ppm has been included for comparison, since this value is within the typical range of industrial eutectoid steel grades without specific nitrogen addition. The reduction in nitrogen from 110 to 40 ppm shifts the precipitation start temperature of VN in austenite to significantly lower temperatures, reducing almost completely the possibility of interaction between recrystallization and precipitation during practical rolling. This implies that a combination of V and nitrogen is required to enhance VN strain induced precipitation in austenite at temperatures sufficiently high to be able to accumulate strain.

This approach, comparing \(F_{\text{REX}}\) and \(F_{\text{PIN}}\) driving forces, provides a qualitative tool for understanding their interaction during a multipass deformation schedule, but does not consider the kinetics involved in both processes. Increasing the strain, both recrystallization and precipitation accelerate by a different amount.\(^{12,30\)}\(^{12,30}\) The recrystallization time has a greater dependence than precipitation time on strain (usually \(t_{0.5X} \sim e^{-n}\), with \(n\) taking values between 2 and 4, while \(t_{0.05} \sim e^{-0.5X}\), where \(t_{0.5X}\) and \(t_{0.05}\) are the time taken to reach a 50% recrystallization and the time for the start of strain induced precipitation, respectively). This implies that as the pass strain increases from \(\epsilon=0.2\) to 0.4 the higher degree of recrystallization brings about a decrease in the \(T_{\text{sw}}\) values, as described in Fig. 3. This decrease of \(T_{\text{sw}}\) with strain is typically observed in low carbon microalloyed steels as well.\(^{12,30,31}\)

The rise in \(S_v\) is related to the refinement due to successive recrystallizations in the temperature range where the material completely recrystallizes between passes \((T>T_{\text{sw}})\), refinement that is enhanced at higher strains. Additionally, the strain accumulation when the material does not recrystallize at all or only partially does \((T<T_{\text{sw}})\), contributes to an increase in \(S_v\), this effect being more important in the vanadium steel. Less retained strain for \(\epsilon=0.4\) (lower \(T_{\text{sw}}\)) and probably a larger availability for some degree of recrystallization to take place below this temperature explain why in the C–Mn–V steel \(S_v\) is lower at these conditions compared to \(\epsilon=0.3\). It must be taken into account that refinement due to successive recrystallizations is limited, being necessary to retain strain for additional refinement.

4.2. Pearlite Microstructure

In addition to the retardation of recrystallization, the vanadium precipitates in the austenite may also affect transformation behavior. In the case of low carbon steels these precipitates have been reported as very effective nucleation sites for ferrite grains, thus promoting additional refinement of the ferrite microstructure.\(^{32–36}\) In the case of eutectoid and hypereutectoid steels it has been reported that the addition of vanadium can change the nucleation manner of pearlite.\(^{37}\) The formation of VC precipitates might result in the depletion of carbon at austenite grain boundaries, inhibiting the formation of proeutectoid cementite and thus probably ferrite first nucleating at the VC/austenite interfaces. The formation of abnormal ferrite in hypereutectoid steels proposed by several authors is possibly related to this mechanism.\(^{38}\)
Therefore, the formation of ferrite in eutectoid and hypereutectoid vanadium microalloyed steels might be related to the precipitation of VC during deformation. A depletion of carbon around these precipitates promotes ferrite to first form in the VC/γ interface. Vanadium nitrides start precipitating at high temperatures (interaction with recrystallization), but as the temperature decreases vanadium carbides may also form in the austenite during deformation (below 890–900°C). The lower presence of ferrite for the largest pass-strain suggests that in this case it is probable that the precipitation of vanadium in the austenite is less favored. This may be due to the higher degree of recrystallization reached under these conditions, since in the absence of deformation a retardation in precipitation is expected, compared to the other case where the accumulation of strain favors precipitation to occur earlier. Linked to this accumulation of strain is a significant increase in the specific grain boundary area, $S_{γ}$, at these deformation conditions (Table 2). The increase in this parameter seems to promote the formation of ferrite in these eutectoid steels. The same was observed in the C–Mn steel, where the formation of ferrite also occurs, above all for the larger pass-strain of $ε=0.4$, where the highest $S_{γ}$ resulted.

A refinement of the pearlite microstructure (in terms of “ferrite unit” size) in the C–Mn–V steel compared to the plain C–Mn steel, as well as after the application of a strain per pass of $ε=0.3$ in the vanadium steel compared to $ε=0.4$ is clearly evident. In both cases the refinement is related to the increase in the specific grain boundary area of austenite, $S_{γ}$ (Table 2). Since austenite grain boundaries are preferential nucleation sites for pearlite, increasing the value of $S_{γ}$ allows a larger number of pearlite nuclei to form, therefore the microstructure, in terms of “ferrite unit” size, is refined. This is expected to bring about an improvement in the toughness properties of the pearlite microstructure. As previously mentioned, microalloying with vanadium has shown to produce a refinement in the final ferrite microstructure in low carbon steels. This refinement is based on the enhancement of ferrite nucleation through particle-stimulated nucleation on V(C, N) particles present in the austenite before transformation. From the present results the possible refinement of pearlite due to additional nucleation on particles cannot be distinguished because of the large difference observed in the specific grain boundary area values between both steels. Larger values of this parameter arise in the vanadium microalloyed steel which makes difficult to separate the effect of the different refinement mechanisms. Further work is required to shed light on this subject.

Looking at the microstructure it can be seen that the interlamellar spacing values determined in the C–Mn steel for both deformation conditions and in the C–Mn–V steel for $ε=0.3$ are very similar, close to $λ=0.13$ μm. However, a significant increase in the hardness is observed in the vanadium microalloyed steel due to precipitation hardening contribution. In this steel there are also some differences in the interlamellar spacing resulting slightly larger for $ε=0.3$ compared to $ε=0.4$, with a lower hardness. Both, coarser pearlite structure and the presence of a higher fraction of ferrite may explain the lower hardness measured in this case. The interlamellar spacing is usually related to the transformation temperature, i.e. the cooling rate at continuous cooling conditions, although austenite microstructure may have presumably some influence. In low carbon steels the increase of $S_{γ}$ parameter related to the accumulation of strain leads to higher transformation start temperatures. Similarly, for a given cooling rate, higher transformation start temperature, thus coarser interlamellar spacing, would be expected for pearlite as $S_{γ}$ increases. This seems to occur with the vanadium steel, where the larger interlamellar spacing is observed for the highest $S_{γ}$ value corresponding to $ε=0.3$. Moreover, an increase in hardenability due to vanadium addition can be also argued, since similar values of $λ$ were obtained in both steels, although $S_{γ}$ was significantly larger in the vanadium steel.

5. Conclusions

(1) Well defined non-recrystallization temperatures ($T_{nr}$) are observed in the eutectoid vanadium microalloyed steel during multipass deformation, making possible to accumulate strain in the austenite when deformation is applied below this temperature.

(2) The non-recrystallization temperature has been related to the interaction between recrystallization and strain induced precipitation of vanadium nitrides in the austenite. This interaction was analyzed in terms of the driving forces corresponding to both mechanisms, $F_{REX}$ and $F_{PIN}$, respectively. It was observed that precipitate pinning force overcome the recrystallization driving force in a temperature range close to the measured $T_{nr}$ values, confirming that precipitation was the mechanism responsible of recrystallization retardation.

(3) The accumulation of strain produces a significant refinement of the austenite leading to high values of the specific grain boundary area ($S_{γ}$) which may produce, after transformation, a significant refinement in the “ferrite unit” in pearlite.

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