

## Effect of Nb Addition on the Hot Deformation Behavior and Microstructure of Low Carbon Steel

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### Abstract

Industrial trials for hot deformation production of low carbon steel with fine ferrite grains were carried out. The different hot rolling parameters including the amount of strain and strain rate at each of six rolling stands as well as the final deformation temperature were designed to produce the required microstructure. Two types of low carbon steels, with/without Nb-addition, were used in this study to evaluate the effect of Nb addition. Stress induced grain refinement has been successfully achieved, aided by Nb, resulting in an average grain size of 4 $\mu$ m following the coiling process. The flow stress calculated at each of the six stands was used to follow the structure evaluation during processing.

### Keywords

Interpass time, Rolling parameters; Hot deformation; Flow stress; Compact strip production; Grain refinement; Niobium alloyed steel

### Introduction

Developing steels with adequate combination of high strength and toughness has been the aim of numerous researches. High strength low alloy steels (HSLA), or micro-alloyed steels are mainly designed for better mechanical properties and/or greater resistance to corrosion than the conventional grades of carbon steels. Minor additions of elements such as niobium, vanadium, titanium, molybdenum, zirconium, boron, and even rare-earth metals have been investigated in literature. Krauss while explaining austenite grain size control, indicated that micro-alloyed steels containing small additions of alloying elements such as Nb, showed improved mechanical properties through grain size control and precipitation strengthening [1].

Wang et al studied the effect of Nb precipitates on austenite to ferrite transformation and considered Nb one of the most important additions for grain refinement in low carbon steels [2]. The solubility of Nb in austenite is limited, which is associated with the relatively large difference in their atomic sizes. A small Nb content in steel can lead to a strong tendency for forming carbo-nitride second phase precipitates, Nb (C, N), which does not suppress a possible segregation of some Nb solute atoms to the grain boundaries. The main contribution of Nb micro-alloy addition is the strengthening of steel as a result of grain refinement and both solid solution and precipitation strengthening mechanisms. Barbaro et al and Ricks et al found that Nb (C, N) particles act as nucleation sites

for acicular ferrite, whose interlocking morphology and its ability to pin dislocations are known to increase the steel strength [3,4].

Bakkaloğlu and Pereloma et al indicated the strong effect of Nb addition as a retarding factor for recrystallization kinetics which has been attributed to solute drag effect and/or strain induced precipitation [5,6]. However, the solute drag effect is not fully understood and the low content of Nb (C, N) particles in small sizes makes it difficult to be observed. Hence, their contribution to recrystallization mechanism is relatively difficult to be followed experimentally.

Thermo-mechanically controlled rolling with optimized parameters, such as the rolling temperature, rolling schedule and cooling conditions, was used in industrial scale to produce fine ferrite steels. A further development in this processing technique, mainly on laboratory scale, has managed to produce a finer grain size by employing higher reductions at relatively low rolling temperatures. The effect of rolling schedule on the final microstructure of hot rolled steel strips has to be verified for the on line production to reduce the gap between theoretical simulation prediction and the industrial scale.

There are various mechanisms of grain refinement techniques ranging from employing complex thermal deformation cycles to typical thermo-mechanical mechanisms. Dynamic strain induced ferrite transformation (DIFT) includes austenite to ferrite transformation during or rather after deformation at large strains. The deformation temperature is mainly above Ar<sub>3</sub> (the empirical austenite to ferrite

transformation temperature) and in general, the transformation occurs when the deformation is applied in the zone between Ar<sub>3</sub> and Ae<sub>3</sub> (the equilibrium austenite to ferrite transformation temperature).

The effect of static and dynamic recrystallization on the rolling load has been investigated using a strip rolling simulation model which indicated the importance of controlling the interpass times during different hot rolling stages. Kliber et al and Aranas et al have found, using simulation and modeling of the hot deformation process, that the interpass times are related to the mean flow stresses (MFS) at each rolling stand and are demonstrated to lower the shorter interpass times [7,8]. The mean flow stress values can be directly related to certain metallurgical phenomena, such as work hardening, recrystallization, precipitation and phase transformation. For larger interpass times the static recrystallization will be much more effective in reducing the work hardening retained during hot rolling stages. Moreover, less attention has been given to the effect of post dynamic treatment, i.e., during hot deformation using several rolling stands. During austenite transformation to ferrite, the formed ferrite subjected to further hot deformation can undergo a dynamic recrystallization. Such transformation sequence can have a pronounced effect on both the microstructure and the mechanical behavior of steel.

The present study focuses on the influence of Nb on the non-recrystallization temperature (TNR) of free-Nb and Nb-bearing steels. If processed correctly, the steels should meet the requirements of alloyed stainless steel (AHSS) grades. This study also aims to characterize and develop an understanding of the high temperature properties during hot rolling.

An industrial hot strip rolling mill with six stands was used for investigating the effect of various rolling parameters on grain refinement for low carbon steel with different Nb content. The microstructure evolution with rolling conditions and mechanical properties changes were carefully examined. However, the maximum on line load at each rolling stand cannot permit applying a high strain per pass, which might impose a limitation for the attained grain refinement. Furthermore, the ferrite grain refinement is known to be strain dependent, where about 80% reduction in a single pass is needed to refine only 50% of the deformed microstructure. Therefore, it is important to determine the maximum strain and interpass time for each of the six rolling stands which can be applied during processing to attain a relatively high effective grain refinement for the structure.

## Experimental/On Line Procedures

Two types of low carbon steels were used in this work; one with Nb addition and the other was Nb free. All alloying additions (in weight %) are shown in table 1.

The on line processing was carried out to produce 3 mm thickness sheets using a compact strip

production plant (CSP) with six mill stands followed by laminar cooling. The hot deformation processing parameters for both types of steels were similar and shown in table (2). However, the soaking temperature used was relatively higher for the Nb-steel at 1160 °C as compared to 1020~°C for the Nb-free steel. The Nb-solution temperature for the present used steel composition was calculated by Groni [9], giving a temperature of 1150 °C. The total hot deformation reduction was kept constant for both types of steels investigated. However, the reduction per pass as well as the entry temperatures at each rolling stand were different, see table (2). The final deformation and coiling temperatures were chosen to be also similar for both types of steels.

The samples were prepared for microstructure examination. The samples were first examined using an optical microscope. High magnification images were used to determine the average grain size using an image analyser (at  $n > 10$ ). Further examination was performed using a scanning electron microscope (SEM) Philips XL 30, at 20 kV. Energy-dispersive X-ray spectroscopy (EDX) was used in conjunction with SEM to detect the elements present in a specific area and/or spot.

The rolling parameters such as strain, strain rate, roll friction, rolling force and temperature at each pass were used to estimate the flow stress value for each pass and interpass times.

## Results and discussion

### Strip rolling and flow stress

#### C-Mn low carbon steel

The measured temperatures at each rolling stand during the deformation of the two steel compositions are shown in figure (1) with both the actual interpass times and the calculated flow stress values. The deformation temperatures are all above the Ae<sub>3</sub>, estimated to be 837 °C, for each type of steel except in the last stand. The hot deformation in the absence of phase change lead to an increasing flow stress from pass to pass due to the decrease in the deformation temperature and the increase of the accumulated strain, as shown in figure (1).

A rolling schedule was proposed and tested on the hot-rolling mill for both steel types as shown in table (2). Due to heat losses between stands, the mean temperature of the steel strip continues to decrease along the deformation line. Therefore, the actual deformation temperature at each stand was measured and is given in figure (1) with the other rolling parameters.

The changes in the actual flow stress were calculated from the force measurements for each of the six rolling stands, with the corresponding strain. Those values are directly related to the imposed rolling conditions. The stress appears to gradually increase with the number of passes, however, the rate of its increase is quite high following the first pass then it decreases down to the last stand. For the Nb free steel, the flow stress rate increased by 17 % after the first pass then dropped to 11% after the second pass

to reach a net rate of increase of only 5% at the final stand. A similar trend was observed with the Nb micro alloyed steel. The total increase of the flow stress from the first to the last stand was ~ 96 MPa and 113 MPa for C-Mn steel and Nb steel respectively. **Table 1** Chemical analysis.

Elements wt%	C%	Si%	Mn%	P%	S%	Ti%	Nb%	Al %	Cr%	Ni%	N%	V %
<b>C-Mn</b>	0.05	0.06	0.9	0.01	0.003	0.002	0.002	0.038	0.015	0.02	0.005	0.003
<b>C-Mn-Nb</b>	0.05	0.04	0.9	0.01	0.003	0.001	0.032	0.044	0.019	0.02	0.006	0.002

**Table 2** Hot deformation parameters.

Deformation parameter	Rolling Stand	C-Mn steel	C-Mn-Nb steel
Temperature, °C	Before first stand	1020	1160
	First stand	988	1030
	second stand	967	962
	Third stand	946	935
	Fourth stand	910	905
	Fifth stand	866	856
	Last Stand	822	825
Strain	Coiling	510	505
	First stand	0.52	0.54
	Second stand	0.4	0.38
	Third stand	0.33	0.31
	Fourth stand	0.31	0.29
	Fifth stand	0.26	0.24
Strain rate, mm/s	Last Stand	0.19	0.21
	First stand	5.41	5.15
	Second stand	16.3	15.52
	Third stand	36.58	37.54
	Fourth stand	64.44	67.17
	Fifth stand	72.19	73.21
Last Stand	77.36	78.4	

The initial rise in the flow stress values following the first rolling stand can be associated with the strain accumulation in the austenite matrix. The imposed strain should give an important rise in the dislocation density so that a transformation is possible. Simultaneously, along with the work hardening generated during the first stand of hot deformation, two distinct softening mechanisms are suggested to take place. Ghosh et al pointed out these softening mechanisms, namely the austenite to ferrite dynamic transformation (DT) and dynamic recrystallization (DRX), while studying the dynamic transformation of austenite [10]. It has been indicated that a lower critical strain is needed to start DT compared with the higher strain required for DRX. On the other hand, the actual flow stress values at each rolling pass can provide evidence for the structural changes such as recrystallization, work hardening, precipitation as well as transformation. These structural changes depend on the strain, strain rate, deformation temperature, interpass time and composition.

As indicated before, all deformation steps, except for the last pass, were carried out in the austenite zone for both studied types of steel. It follows that the relatively high rise in the flow stress was observed

Therefore, the most critical interpass condition occurs between passes 1 and 2 where both the highest deformation temperature and the longest interpass times are observed.

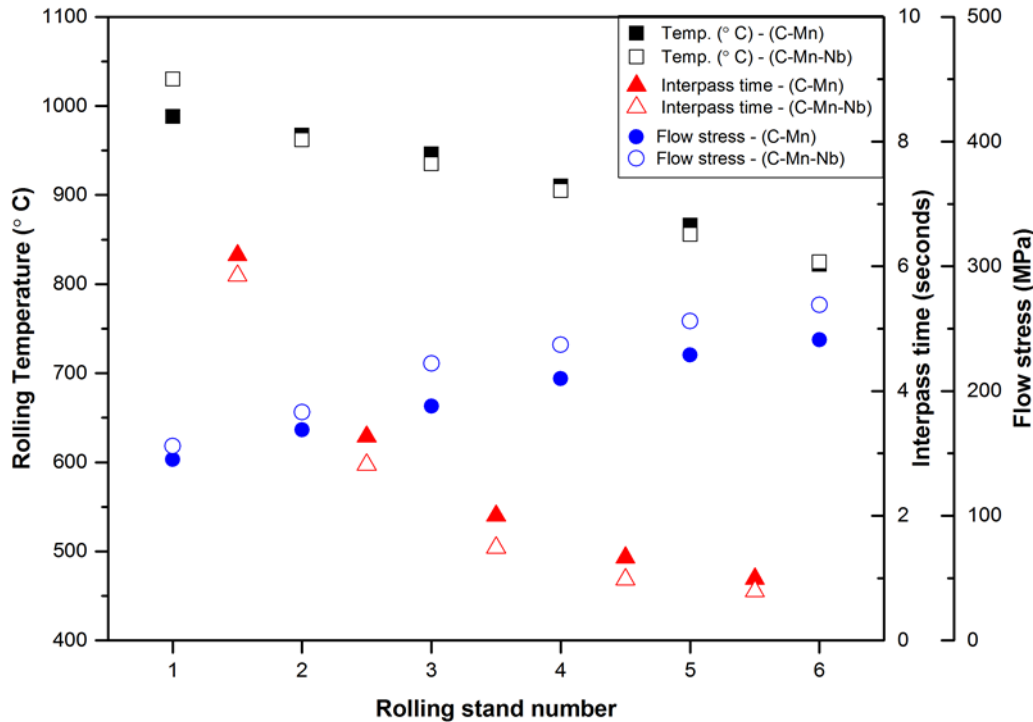
following the first pass which corresponds to the largest interpass time (6.2 s) for Nb free steel. This can be related to several processes acting individually or simultaneously to produce the structural changes. At the beginning, the deformation was carried at low strain rate and high amount of strain conditions under which a high strain accumulation was produced. Bhadeshia pointed that once a critical strain value is reached in certain structure location, dynamic austenite transformation (DT) of ferrite is the most likely to occur [11]. Similarly, but at higher relative critical strain, dynamic recrystallization of austenite would also take place during this stage of deformation. Nevertheless, no quantitative information can be provided in this work regarding the volume fraction of the suggested transformation. The occurrence of such transformation cannot be traced out because of the observed rise in the flow stress value in the following pass, despite the long interpass time between the first two stands where softening should be expected.

The retained hardening effect associated with a higher flow stress after the first interpass time can be attributed to the reverse transformation mechanism of DT ferrite phase back to the harder austenite phase. The reverse transformation has been confirmed to take place during interpass times and to be time dependent by a theoretical study of strip hot rolling process in a C-Mn steel [12]. Moreover, a static recrystallization of the dynamically deformed austenite is quite expected to occur and to be a function of the interpass time length leading to softening in the microstructure. The outcome of these two mechanisms on the microstructure is obviously in favor of increasing the retained hardening due to ferrite reverse transformation.

The hot deformation process at stands 3 to 6 is associated with a reduction in the interpass times, from 2 to 1second. A limiting softening by static recovery and /or recrystallization is likely to take place during those short interpass times. Meanwhile, the increases in flow stresses up to the final stand can be associated with the decreases of rolling temperature as well as the accumulated strain per pass leading to the enhancement of strain inducing ferrite transformation. It is therefore possible to conclude that reducing the interpass times during hot deformation acts as single pass of large strain deformation along with enhanced refinement process. Furthermore, in order to avoid static recrystallization during multi-pass processing, the final rolling temperature should be further reduced

below 825 °C, but this will be accompanied by an important rise of the rolling force. On the other hand, to increase the temperature at the entry of the

finishing stand, the kinetics of static recrystallization can be reduced by Nb-addition due to its expected retardation effect on grain boundaries movement.



**Figure 1** The change in the Flow stress and interpass times in both the C-Mn and C-Mn-Nb steels during industrial production.

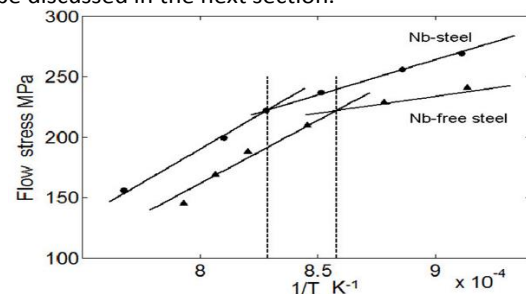
#### 0.032 Nb micro alloy steel

The presence of micro-alloy Nb addition in austenite during hot deformation is expected to have a retarding effect on both recovery and recrystallization leading to a higher accumulation of strain compared to C-Mn steels at similar rolling conditions. Bowden et al related this retarding effect to the segregation of Nb atoms on the interfacial area reducing its movement [13]. The actual flow stress patterns of these steels are shown in figure (1). The flow stress is typically increasing with lower deformation temperatures but in general, higher stress values were attained with Nb addition when compared to the Nb free steel. The total imposed strain and the final deformation temperature were the same, whereas, the total interpass time was less by about 2 seconds for the Nb steel.

The highest flow stress increase is observed for the first to the second passes with long interpass time of 5.85 seconds. Such increase in the flow stress cannot be attributed to similar metallurgical phenomena as for the above case of C-Mn steel. Therefore, the formed DT during hot deformation is not expected to undergo the same amount of reverse transformation during the interpass time. Ouchi et al and Chimani et al related this increase to the interface or boundary pinning by Nb solute atoms, i.e. drag effects [14,15]. Therefore, less retained work hardening would be possible for the second stand rolling. Moreover, dynamic recrystallization of austenite would also be delayed by the Nb drag effect leading to a higher accumulated strain in the deformed austenite. Such effect will enhance the nucleation rate for austenite transformation improving the grain size refining

mechanism. A similar reason could explain the less softening due to a reduced static recovery and recrystallization in the interpass time.

Therefore, the increase in the flow stress in the second pass can be associated with a retarding DRX during deformation as well as with limited static recovery and recrystallization in the interpass time. The amount of static softening during interpasses is time dependent and since time is reduced gradually between passes to reach 0.8 second in the last interpass, minimum softening can be expected for the last deformation stage. In addition, reducing the driving forces for static recrystallization, at this stage of the last two to three stands, would cause most of the dislocations generated by deformation to be consumed by dynamic recrystallization and ferrite nucleation during the last stage of hot strip rolling. A two stage effect can be observed for the flow stress changes during multi deformation process which will be discussed in the next section.



**Figure 2** Dependence of the mean flow stress on inverse absolute temperature for Nb and Nb-free steels. Results were obtained from compact strip production plant (CSP) with six rolling-mill stands.

## Two stage flow stress

The mean flow stress (MFS) is plotted against the inverse of absolute temperature at each rolling stand, as shown in Figure (2), indicating the MFS evolution during the multi-deformation process. For both steel compositions, an inflection point between two stages can be observed. This is likely corresponding to the  $T_{NR}$  value for both steel types. The steel with Nb-content shows a higher  $T_{NR}$  value compared to the free Nb-steel, 934 ° C and 893 ° C respectively. It is generally accepted to define  $T_{NR}$  as the non-recrystallization temperature above which the deformation allows for recrystallization to proceed. Deformation below such temperature results in a higher strain accumulation which enables nucleation for ferrite transformation. The non-recrystallization temperature can be useful mainly in designing the hot-rolling schedule. Various empirical formulas have been proposed to calculate  $T_{NR}$  among which the one suggested by Bai and mentioned in Groni [9] follows:

$$T_{NR} = 174 \log [Nb (C+0.857 N)] + 1444 \quad (1)$$

Another  $T_{NR}$  equation was developed by Fletcher [16]. However, the Fletcher equation ignoring pass strain is given by

$$T_{nr} = 849 - 349C + 676\sqrt{Nb} + 337V \quad (2)$$

$(R^2 = 0.72)$

where Nb, C, V and N are the steel alloying elements in weight percentage. The  $T_{NR}$  values calculated using equation 2 for both steel types agree well with the values obtained in the present hot-rolling condition, see table 3. However, the discrepancy found in these values can be related to the fact that all these equations do not account for the strain effect in their proposed formulas. Since the designed strain in each rolling pass is similar for both types of steels, the difference in their  $T_{NR}$  values could be mainly attributed to the Nb-addition which is found to increase it.

**Table 3** Values of TNR calculated and obtained from CSP results.

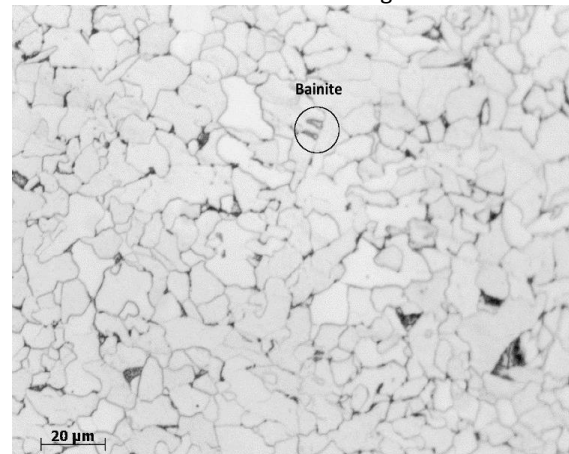
Steel	$T_{NR}$ ° C		
	Equation (1)	Equation (2)	Present work
Nb-free steel	754	872	893
Nb-steel	965	959	934

Coldren and Cryderman found that at high enough temperatures (i.e. above TNR), the observed increase in MFS could be related to the decrease in the deformation temperature along with the solute drag effect associated with Nb-addition to steel [17]. While below TNR, only partial or no recrystallization takes place. Moreover, the shorter interpass times at this stage can lead to strain accumulation which enhances the strain induced precipitation of NbC, which is expected to form at lower temperatures in contrast to NbN which is likely to form at higher temperatures. Furthermore, the high strain will also accelerate the austenite to ferrite transformation due to high potential nucleation sites. The changes in the

hardening effect for each of the two stages can be associated with the steep rise in the flow stress at the third deformation stand for Nb-steel, taking place at a deformation temperature of 935 ° C, as shown in figure 1. Such rise in the flow stress has been attributed to the precipitation effect following the transition temperature above 940 ° C as indicated previously in Figure 2.

## Microstructure analysis

The observed microstructure of C-Mn steel following the hot-deformation processing indicated the presence of a rather homogeneous equiaxed ferrite grain distribution with an average size of 6  $\mu$ m, as shown in Figure 3. Moreover, some of the grains appear elongated in shape which can be related to a subsequent deformation following the refinement transformation, i.e., they were deformed during the final stages of deformation. Relatively small quantities of pearlite phase are also observed mainly on the grain boundaries. Faint substructure boundaries appear inside some of the grains indicating the occurrence of either recovery or a further deformation for the refined ferrite grains.



**Figure 3** Microstructure of C-Mn steel.

It is well known that Mn is amongst other certain alloying elements which when added to steel promote the formation of bainite or acicular ferrite and limit the polygonal ferrite and pearlite formation. Sakuma et al showed that increasing the Mn content can contribute to prolonging the nucleation time of ferrite, thus, favors the formation of low temperature phases [18]. Mn-addition is well known to shift the CCT curve to the right allowing intermediate phases such as bainite to form at relatively lower cooling rates. In this study, the presence of ~ 0.9 %Mn only lead to the formation of a very small amount of bainite, which appeared inside the grains, see figure (3), in a dominant polygonal ferrite matrix. This microstructure suggests that the rate of cooling is the main controlling factor for the observed phases.

Nb addition to steel is known to delay the austenite transformation by reducing the grain boundaries mobility. Akben et al found that if NbC precipitates form, this would decrease the free Nb atoms in austenite [19]. Delaying the austenite transformation during hot-deformation is likely to increase the dislocation density which increases the possible nucleation sites for low temperatures

phases. This nucleation process will impede the growth of  $\gamma/\alpha$  interface as well as the carbon diffusion to transformation interface, i.e. increasing austenite stability. Therefore, at high cooling rates, the martensite phase is expected while at low cooling rate pearlite phase will take place. In the present case with 0.032%Nb, the hot deformed microstructure shown in figure 4 indicates a more effective grain refinement with an average size  $\sim 4\mu\text{m}$  compared to the free Nb-steel. This in turn confirms the important role of Nb in grain refinement through enhancing nucleation and limiting coarsening processes.

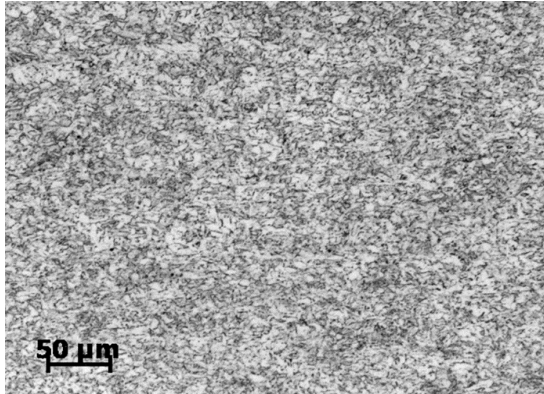


Figure 4 Microstructure of C-Mn-Nb steel.

Figure 5 shows a SEM for Nb-steel microstructure where the pearlite lamellar structure dominates at the grain boundary areas and fully bainite grains are randomly distributed in the ferrite matrix. The bainite volume fraction is relatively higher compared with Nb-free steel, confirming Nb-role in enhancing low temperature phase transformation as Nb moves CCT curve up.

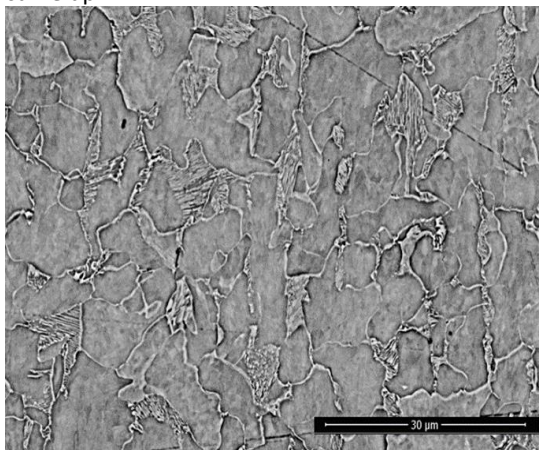


Figure 5 SEM of C-Mn-Nb steel.

A rather small drack feature appears only in some grains and can be associated with being NbC precipitates. This precipitate formation can be enhanced by the high temperature strain in the presence of both Nb and C with high diffusivity at these conditions of hot rolling deformation.

## Conclusions

- An industrial scale rolling scheme is designed to enhance stress induced grain refinement to take place during on line hot-deformation of low carbon steel with and without Nb-addition. An average grain size of  $6\mu\text{m}$  (for Nb-free steel) and

$4\mu\text{m}$  (for Nb alloyed steel) is obtained following the coiling process.

- The mean flow stress of each of the six rolling stands is not only function of temperatures but is also reverse transformation dependent.
- Reducing the interpass times between stands maintains static recrystallization at minimum during multi-passes deformation.
- The softening effect related to the retardation of reverse transformation attributed to Nb-addition is counter balanced by an increase in the passes flow stress due to a retardation of both dynamic recrystallization and austenite to ferrite transformation.

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