RECRYSTALLIZATION CONTROL TO OBTAIN UNIFORM FINE GRAIN SIZE IN HOT ROLLING OF HTP STEEL

S. V. Subramanian, H. S. Zurob and G. Zhu Department of Materials Science and Engineering, McMaster University, 1280 Main St. W., Hamilton, ON, L8S 4L7, CANADA

Keywords: Modeling, Microalloying, Niobium, Grain-size control, Recrystallization, Toughness, HTP

Abstract

In multi-pass rolling of microalloyed steel, the toughness properties are impaired by mixed grain size. In order to obtain a uniform fine grain size, it is essential to suppress partial recrystallization in the inter-pass times. In this regard, high niobium low interstitial high temperature processed (HTP) steel design offers distinct advantage in hot rolling. This is confirmed in the present work by physically-based modeling and experimental simulations. The application of the model for grain size control is demonstrated for two cases involving short (strip rolling), and long (Steckle mill rolling) interpass times respectively. The beneficial effect of high manganese design is also highlighted.

Introduction

In order to obtain good performance of line pipe steels at very low temperature approaching Arctic environment (around -60°C), high toughness is required at high strength levels. Grain refinement is regarded as the best option that brings about a simultaneous improvement in strength and toughness of steel for service at low temperatures. Ultra-fine ferrite grain size could be achieved by promoting high density of nucleation sites for ferrite grains. This can be achieved by increasing the austenite grain boundary area per unit volume (S_v) through austenite grain refinement and/or through the pancaking of austenite [1,2]. Deformation has the additional effect of enhancing the potency of the ferrite nucleation sites [3]. Thus the microstructural refinement can be obtained through adequate strain accumulation in austenite to bring about dynamic recrystallization prior to transformation [4] or strain induced phase transformation in austenite [5]. However, the toughness properties are impaired if mixed grain size is obtained. This could arise from non-uniform starting grain size at the commencement of finish rolling or partial recrystallization occurring in the interpass times during finish rolling or a combination of both.

In order to obtain uniform grain size during finish rolling, it is essential to suppress static recrystallization in the interpass times. Recrystallization control through microalloying is the subject of this paper. Analysis based on single pass deformation [6] shows that recrystallization nucleation will be significantly delayed due to the pinning of dislocations by strain induced precipitates, when the number density of precipitates approaches the dislocation node density.

This effect is distinct from the conventional Zener pinning argument which relies entirely on precipitation to pin the grain boundaries and thereby retard recrystallization [7, 8]. Our modeling has clarified the important role of both effects [6, 9]; it has been shown that the dislocation-pinning effect is most important at short times when the number density of precipitates is high and the volume fraction is low. The Zener drag condition on the other hand is important at relatively long holding times when the volume fraction of the strain-induced precipitates is adequately large to exert significant pinning pressure. The modeling also highlights the importance of solute niobium in the retardation of recrystallization through reduction of the boundary mobility, particularly, in low temperature window of rolling. The retention of a sufficient amount of niobium in solution in order to prevent the boundaries from moving at high mobility is one of the important results of the model [6, 9].

The extension of the single pass deformation model to multi-pass rolling is rendered difficult because of the limitation of site-saturation assumption. In multi-pass rolling, new dislocation nodes are generated with each pass and these interact with strain-induced precipitation, recovery and recrystallization. Zhu et al. [10] described a semi-empirical model to describe multi-pass rolling. The predictions of this model were validated against experimental trials on an advanced thermo-mechanical simulator (WUMSI) [11].

In this contribution we apply the physically-base single-pass model of Zurob et al [6, 9, 12] and the multi-pass analysis of Zhu and Subramanian to determine the optimum processing conditions for grain-refinement in the low temperature window of hot rolling. Special attention is given to the difference in rolling practice for short interpass times, typical of near-net shape processing, and long interpass times, typical of steckle mill finishing.

In what follows, the physically-based single-pass model is briefly reviewed (section 2). The model is then used to examine the effect of the steel chemistry, processing parameters and grain size on the recrystallization kinetics of HTP steel. Our analysis is extended to multi-pass deformation in section 3 and applications of our analysis to near-net-shape and steckle mill rolling are discussed in sections 4.1 and 4.2 respectively. General discussion and conclusions follow in section 5, while future work is summarized in section 6.

Physically-based Model for Single-Pass Deformation

The time evolution of strain-induced precipitation and its interaction with recovery and recrystallization are the basic building blocks of any physically-based model for strainaccumulation in multi-pass rolling. The first of these building blocks was laid by Dutta et al. [13] who developed a physically-based model for the time evolution of strain-induced precipitation. This model captured the time evolution of the precipitate size and number density for a given set of processing parameters (temperature, deformation). Zurob et al. [12] used a similar precipitation model to examine the effect of precipitation on recovery. It was demonstrated that the precipitates pin the dislocation segments on which they form and this significantly retards the rate of recovery. Once the precipitates start to coarsen, some dislocations will be unpinned, and recovery resumes at a rate controlled by the coarsening kinetics of the precipitate. By coupling the above models of precipitation and recovery to a model of recrystallization it is possible to present a complete description of the microstructural evolution of microalloyed steels following single-pass deformation. Our interest in the present paper is the effect of solute and precipitate niobium on the recrystallization kinetics of HTP steels under strain accumulation conditions. The effects of solute and precipitate niobium on recrystallization are reviewed in section (2.1).

This is followed by a trend analysis of the effects of niobium content, manganese content, pass reduction, temperature and grain size on the kinetics of recrystallization (section 2.2).

2.1. Effect of Niobium on Recrystallization.

The effect of niobium on recrystallization can be broken up into three (interdependent) contributions; a) solute drag due to niobium in solution, b) retardation of recrystallization nucleation by a high number density of strain-induced precipitates and c) retardation of recrystallization growth due to boundary pinning (Zener drag). Each of these effects is examined next:

2.1.1. Solute Drag: Within the framework of the Johnson-Mehl-Avrami theory and in the limit of site-saturation, the kinetics of recrystallization is described by an equation of the form [12, 14]:

$$X = 1 - \exp\left(-N\left(\int M(t)F(t)dt\right)^3\right)$$
(1)

where X is the recrystallized fraction, N is the number of recrystallization nuclei, M and F are, respectively, the grain boundary mobility and the driving force for recrystallization. Solute additions hamper recrystallization by reducing the mobility of the recrystallizing grainboundaries. In the most general case, Cahn's theory of solute drag [15] leads to a non-linear relationship between the boundary velocity and the driving force for boundary migration. For high solute levels (or small driving forces) strong solute drag conditions exist and a large increase in the applied force results in only a small increase in velocity. At the other extreme, when the solute content is very low, or when the driving force is large, the alloy behavior approaches that of the pure material and a small increase in driving force results in a large increase in boundary velocity. A transition from strong to weak solute drag conditions can take place if the applied force is increased or if solute is depleted from solution. This possibility was emphasized in a recent contribution and is illustrated in Figure 1a; for a driving force of F₁, a jump from the slow to the fast velocity branch will take place if the of the concentration of niobium in solution drops from C₂ to C₃. The exact value of the critical niobium concentration



Figure 1: (a) The non-linear force-velocity relation predicted by solute drag theory. For a force of F_1 , breakaway takes place when the concentration drops below C_2 . (b) The temperature and concentration dependence of mobility as described by the slow branch of Cahn's [15] model.

needed to prevent boundary breakaway is difficult to estimate given the lack of experimental data on the breakaway phenomenon in steels. Nonetheless, the need to maintain sufficient

niobium in solution to prevent boundary breakaway is an important concept that should not be overlooked. In the present work, we'll assume that sufficient niobium is always present to prevent boundary breakaway. This is a very reasonable assumption in the case of HTP steels because these contain high niobium (0.08 wt%) low carbon (<0.04 wt%) and high manganese (~1.8 wt%). Assuming a linear force-velocity relationship, we can define the mobility as the inverse of the slope of the linear part of force-velocity curve shown in Figure 1a. The predicted effects of temperature and niobium content on the grain-boundary mobility can then be plotted as shown in Figure 1b. The advantage of the high niobium design of the HTP steel and the advantage of low temperature rolling for strain-accumulation are clearly evident from Figure 1. This point will be elaborated further in section 4 on the application of the model to strain-accumulation.

2.1.2. Effect of Precipitation on Recrystallization Nucleation: A fine dispersion of precipitate particles may greatly restrict the rearrangement of dislocations. As a result, precipitation may delay the onset of recrystallization by limiting the recovery processes needed for the nucleation of recrystallization [6]. This phenomenon is taken into account by introducing the probability of nucleation function, Ψ , which is the ratio of the length scale at which the dislocations are able to move (thus the inter-particle spacing, l) and the length scale over which movement is needed to create a nucleus (diameter of a critical nucleus, D_c):

$$X = 1 - \exp\left(-N\left(\int \Psi(t)M(t)F(t)dt\right)^3\right), \quad \text{where}$$
(2a)

$$\Psi = l / D_c \tag{2b}$$

Since the number of particles determines the inter-particle spacing, the probability term is strongly dependent on the *precipitate number density* and not on the volume fraction. Given that precipitate nucleation usually takes place very quickly (< 1 s), this means that precipitate volume fraction can retard the nucleation of recrystallization even at very early times when the precipitate volume fraction is still very small. This point is demonstrated in Figs. 2a and 2b which show, respectively, the evolution of Ψ and the evolution of the Zener drag as a function of time.



Figure 2: Illustration of the evolution of the recrystallization nucleation probability term (a) and the Zener-drag term (b) as a function of time after deformation.

2.1.3. Effect of Precipitation on Recrystallization Growth: Precipitate particles also exert a pinning (Zener) force on moving grain-boundaries [7, 8, 14]. This force could slow down or even halt the growth stage of recrystallization [8, 14]. The important point to keep in mind is that the magnitude of the Zener force depends on the precipitate volume fraction and particle size. Consequently, it evolves relatively slowly and is not expected to have a significant effect on recrystallization for short interpass times. This is clearly shown in Fig. 2b. Zener drag force becomes significant only at a later stage when the precipitate volume fraction has increased adequately. Even though the process of recovery decreases the stored energy of dislocations, we note that a positive net driving force for recrystallization remains even after 100s. This driving force will result in the growth of the recrystallizing grains at a velocity which is determined by the grain boundary mobility, M, according to the equation:

$$v = M(G - Z) \tag{3}$$

It is important to note that the progress of precipitation will reduce the matrix solute content and in so doing it reduces the solute drag effect and precipitation is accompanied by an increase in the boundary mobility As a result, there is a delicate balance between the need for strong precipitation conditions in order to promote Zener pinning on the one hand and the need, on the other hand, to maintain sufficient niobium in solution in order to prevent the boundary from breaking away from its solute atmosphere. In an earlier contribution we showed that the optimum partitioning of niobium between solute and precipitates depends on the processing conditions [6]. This point will come up again in the discussions in section 2.2 and section 4.

2.2. Analysis of the Factors Affecting Recrystallization in HTP Steel.

Thermodynamic and kinetic factors govern the potential for large strain accumulation to the exclusion of static recrystallization. These are examined in the light of processing parameters for HTP steel. For example, the effect of lowering the temperature is to increase the chemical supersaturation for strain induced precipitation but decrease the kinetics of diffusion. The effect of lowering the temperature is to increase the time for recrystallization. The effect of strain is to increase the dislocation density, which increases the sites for strain induced precipitation. The stored energy of dislocations is the driving force for recovery and recrystallization. Since the critical strain for dynamic recrystallization is a function of temperature window. The effect of varying each of the processing parameters on the static recrystallization behavior of HTP steel is quantitatively analyzed using physically based model based on single pass deformation in the limit of site saturation assumption. The technological implications are annotated.

2.2.1. Effect of Temperature: To illustrate the effect of temperature on the kinetics of recrystallization we considered the evolution of the nucleation probability, the boundary mobility and Zener drag at 900, 840 and 760°C (Figure 3). These three temperatures were chosen to lie above, at and below the nose of the precipitation-temperature-time curve. The results shown in Figure 4 indicate that the rate of recrystallization decreases with temperature even when the temperature is below the nose of the PTT curve. The time for 50 percent recrystallization is delayed from about 3s at 900°C to ~100s at 840°C and more than 300s at 760°C. The high manganese content of HTP steel lowers the transformation temperature making it possible to carry out finish rolling at temperature as low as 760°C and to take advantage of the strong retardation of recrystallization under these conditions.

2.2.2. *Effect of Niobium Content:* Figure 4 shows the effect of niobium content on the recrystallization behavior of HTP steel. By lowering the niobium from 0.08 to 0.04wt percent, the time for 50 percent recrystallization is decreased from 200s to 10s, thus underscoring the importance of the high niobium design of HTP steels.



Figure 3: Effect of the effect of temperature on the recrystallization of HTP steel of grain size 25μ m and for pass reduction of 30%. (a) nucleation probability, (b) grain boundary mobility, (c) Stored energy & Zener force, (d) recrystallized fraction.



Figure 4: Effect of the bulk niobium content on recrystallization of HTP at 840°C and for a pass reduction of 20 percent. (a) nucleation probability, (b) grain boundary mobility, (c) stored energy & Zener force, (d) recrystallized fraction.

2.2.3. *Effect of Deformation:* Figure 5 shows the effect of deformation at 840°C on the recrystallization behavior of HTP steel. An increase in pass reduction of 20 to 30 percent decreases the time for 50 percent recrystallization from 300 to 200 s.

2.2.4. *Effect of Grain Size:* Figure 6 shows that the effect of decreasing the starting grain size is to accelerate the kinetics of recrystallization. An increase in grain size from 25 to 100 μ m is to increase the time for 50% recrystallization from 300 to 600s.



Figure 5: Analysis of the effect of pass reduction on the recrystallization of HTP steel at 840° C (grain size 25µm). (a) nucleation probability, (b) grain boundary mobility, (c) stored energy & Zener force, (d) recrystallized fraction.



Figure 6: Effect of grain size on recrystallization kinetics of HTP steel at 840°C and for pass reduction of 20 percent.

2.3. Summary of conclusions based on single pass deformation analysis of HTP steel:

Our discussion of the physically-based model of single pass deformation of HTP steel leads to five important conclusions:

(a) Strain-induced precipitates retard the nucleation of recrystallization even at the very early stages of precipitation by pinning the dislocations, which inhibits recovery. The probability of recrystallization nucleation is decreased when *the number density of the precipitates* is high. At this stage, the volume fraction of precipitates is too small to contribute to any significant Zener pinning.

(b) Precipitate particles exert Zener pinning force on moving grain boundaries. This depends upon *the volume fraction* of the precipitates, which evolves relatively slowly.

(c) The progress of precipitation depletes the solute from the matrix, which decreases the solute drag effect on the boundary and consequently increases boundary mobility. When the solute content is severely depleted, the boundary mobility will increase significantly offsetting the beneficial effect of precipitation in retarding recrystallization.

(d) The optimum design of base chemistry and rolling schedule is to control the strain induced precipitation kinetics to maximize the pinning force, while ensuring adequate solute niobium to prevent the boundary from breaking away from its solute atmosphere.

(e) The effect of manganese addition is to retard the kinetics of strain-induced precipitation, which leaves adequate niobium in solution to provide strong solute-drag in the low temperature window. In addition manganese has the effect of reducing the transformation temperature and therefore making it possible to take advantage of the retardation of recrystallization at low temperature. Thus the high niobium, high manganese design is beneficial for large strain accumulation in the low temperature window.

Multi-pass Rolling

The extension of the single pass deformation model to multi-pass rolling is rendered difficult because of the limitation of site-saturation assumption. In multi-pass rolling, new dislocation nodes are generated with each pass and these interact with strain-induced precipitation, recovery and recrystallization. Thus the approach taken in the present work is to capture the existing database on critical strain for dynamic recrystallization and the static softening kinetics of niobium microalloyed steel generated by Lutz-Meyer and coworkers [16] on a semi-empirical model. The database on static softening kinetics was analyzed using the Avrami equation to describe the recrystallization kinetics. The critical strain for static recrystallization is determined experimentally to complement the database. The basic concept is to avoid strain accumulation in the regime where static recrystallization would occur. Thus pass design for strip rolling should avoid strain accumulation in the partial recrystallization regime demarcated by critical strain for static recrystallization and the critical strain for dynamic recrystallization. Thus strain accumulation is either below the threshold for static recrystallization or above the critical strain for dynamic recrystallization. The pass design for steckle mill rolling aims at large strain accumulation using low temperature window by taking advantage of retardation of recrystallization by strain induced precipitation and adequate solute to exert solute drag and prevent boundary in breaking away from solute atmosphere.

The strain accumulation model for grain size control is based on the assumption that the starting grain size is uniform. This is normally achieved in plate rolling by roughing where static recrystallization is allowed to progress between successive passes in roughing in order to refine the austenite grain size at the end of roughing to a size range of 25-40 microns. The strategy

adopted in strip rolling is to promote dynamic recrystallization at multiple stages to promote a uniform grain size.

Model Applications

In this section we examine the control of static recrystallization in HTP steels under two industrially important conditions. In section (4.1) we examine the case of long interpass times similar to those in steckle mill rolling. This is followed by a discussion, in section (4.2) of recrystallization control of HTP steels under short-interpass times that are typical of near net-shape processing.

4.1. Control of static recrystallization during Steckel mill rolling of HTP steel with long interpass time:

Steckel rolling mills are gaining popularity mainly due to lower capital costs and the flexibility in production quantities. There is also an attraction that very large slabs (40-50MT) cen be processed. The inter-pass time in Steckel mill rolling can be as long as 100 seconds in comparison with 2-3 seconds in a semi-continuous multi-stand rolling mill. There is little difference in roughing between the conventional and Steckel mill rolling. However, there is substantial difference in the finish rolling caused by static softening that occurs in the long interpass time in Steckel rolling. Modeling results, laboratory studies and mill trials have confirmed the distinct advantage of the high niobium (>0.08wt%), low interstitial (<0.03wt%C and <0.003wt%N) design in the development of high-strength, high-toughness plates for line pipe application using Steckel mill rolling.

The strategy for Steckel mill rolling of high niobium low interstitial steel consists of lowering the temperature of finish rolling to well below the nose of the precipitation-time-temperature (PTT) curve for strain induced precipitation of NbC [17]. A low nitrogen design is used with titanium added to a level some what below the stoichiometric requirement to tie up most of the nitrogen as high temperature precipitates of TiN. This allows the bulk of the niobium to be in solution for precipitation as NbC in the low temperature window during finish rolling. Niobium in solution is effective in retarding the static recrystallization in the low temperature window before the onset of strain induced precipitation of NbC. The pinning pressure due to strain induced precipitation of NbC is more effective than solute niobium in retarding the static recrystallization in the interpass time. Nucleation of the precipitates occurs at dislocations and growth of the precipitates is aided by the high diffusivity paths associated with pipe diffusion along the dislocations. The volume fraction of the precipitates from the first pass is only a fraction of the equilibrium volume fraction and the resulting pinning pressure is accordingly small. In subsequent passes dislocation movement as well as the creation of new dislocations take place and a fresh round of precipitate nucleation occurs. The depletion of niobium by growth of precipitates has to be controlled so that there is adequate solute niobium to maintain low boundary mobility through the solute drag effect. Adequate residual niobium, typically between 0.03 - 0.04 wt percent in the matrix is required for effective retardation of recrystallization through solute drag. The residual solute niobium is subsequently used to advantage to obtain additional strengthening (more than 100 MPa) due to transformation hardening and precipitation strengthening in the ferrite [18].

The strategy is to restrict the number of finishing passes so that precipitation depletes only a fraction (about 50 %) of the total niobium in the matrix. Rolling in the low temperature window is advantageous because the diffusion kinetics is slower making it possible to maintain a larger amount of niobium in solution. Typically the temperature window for finish rolling for a high niobium (>0.08wt%) steel is in the range of 850-730°C. Five passes or less are usually used for

the finishing of grade 80 line pipe steel with the pass reduction being in the range of 30-15 percent.

Up to this point, our discussion has not taken into account the effect of the cooling rate on the amount of residual niobium. Zou and Subramanian [19] analyzed this problem quantitatively and developed a model which could predict the size and mole fraction of precipitates as well as the residual niobium content of the matrix for different cooling rates, ranging from 0.5 to 10°C/s. The growth of the precipitates occurs predominantly in the higher temperature region (>850°C). The time for diffusional growth is determined by the cooling rate. In Figure 7, the residual niobium is plotted as a function of cooling rate. The model, which is based on volume diffusion, shows that the matrix will be depleted of niobium only at slow cooling rates but volume diffusion contribution to particle growth is dominant only at high temperatures (>850°C). Even though the volume diffusion contribution is not significant below 850°C, there could be significant diffusional contribution from high diffusivity paths involving dislocations and grain boundaries, which are not considered in the model. However, if high diffusivity paths due to dislocations or grain boundaries enhance the effective diffusion coefficient, significant depletion of solute niobium dissolved in the matrix will occur. Based on the modeling results, the recommended strategy is to minimize the total time of processing below T_{nr} so that adequate solute niobium is retained to retard recrystallization. The other important parameter is the temperature window of finish rolling. By lowering the temperature window, the diffusional kinetics is decreased, which in turn increases the residual solute niobium well above the threshold for boundary break away. Thus the decrease of total processing time and the lowering of temperature window of finish rolling are identified as two important process parameters to be controlled in order to obtain high residual niobium required in higher grade line pipe steels (Grade 80 plus) produced by Steckle mill rolling [20].



Figure 7: Residual (Solute) niobium plotted as a function of temperature for various cooling rates; Model assumes ideal size and dispersion of particles and growth by volume diffusion in the matrix [19].

4.2. Control of static recrystallisation in strip rolling of HTP steel with short interpass time:

In order to suppress static recrystallization, the accumulated strain at each pass should be less than the critical strain for static recrystallization during inter-pass time at all stages of rolling. The accumulated strain should be controlled to bring about dynamic recrystallization at the required stages, depending upon whether grain refinement is required at one or more stages in the rolling schedule. If dynamic recrystallization is promoted at the last pass without any static recrystallization during inter-pass time, the ultra-fine austenite grain size can be achieved homogeneously prior to phase transformation. Thus the critical strain for dynamic recrystallization and the critical strain for static recrystallization are required inputs for process modeling. Zhu, Gao and Subramanian [21] have analyzed the database on critical strain for dynamic recrystallization ε_c in microalloyed steel generated by Lutz-Meyer and coworkers [16] and correlated the critical strain for dynamic recrystallization ε_c with Z through the following expression:

$$\varepsilon_c Dynamic = 0.048 \ln Z - 0.96 \tag{4}$$

A database for the static recrystallization of HTP steel (0.03%C-0.004 N- 0.015 Ti- 1.5%Mn- 0.097%Nb) was generated using the thermo-mechanical simulator (WUMSI) to correspond to conditions applicable in industrial strip rolling. The dynamic and static softening databases were then used as inputs into a quantitative model of multi-pass rolling in order to develop a new strategy for achieving fine grain size under strip rolling conditions. The result is a rolling schedule in which dynamic recrystallization was promoted twice. The first dynamic recrystallization in Pass 2 is to exert control over the starting grain size and the second and final dynamic recrystallization is used to control the austenite grain size before phase transformation. In the proposed strategy low-temperature rolling is used to advantage for strain accumulation. Table 2 gives the optimized rolling schedule for experimental simulation in thermo-mechanical simulator (WUMSI). In this schedule, the dynamic recrystallization was promoted twice, first at the 2nd pass and a second time at the 7th pass [22].

Pass Number	Time (S)	Thickness (mm)	Reduction(%)	Temperature (°C)
1	0.00	23.53	36.61	828
2	6.13	16.00	32.00	812
3	10.53	12.07	24.54	796
4	13.85	9.26	23.28	781
5	16.40	7.78	16.00	767
6	18.44	7.08	9.00	752
7	20.17	5.95	16.00	735

Table 1: The optimized finishing schedule based on industry trial

Using optimized finishing schedule, an average final grain size of about 1.5 micrometer with minimal spread is obtained. About 95 percent of the total grains were found to have grain size distribution in the size range of 0.3 to 3 μ m as shown in Figure 8. Figure 8(d) compares the grain size distribution that resulted from the optimized rolling schedule with the size distribution that resulted from earlier industrial practice which did not achieve effective grain-size control. The improvement shown in this figure clearly demonstrates the power of the modeling approach which is being proposed here.

Discussion

5.1. Relative contributions of solute drag effect and Zener effect:

Quantitative analysis of the evolution of the stored energy of deformation and the Zener pinning force shows that a net-driving force for recrystallization is present under all industrial conditions. As a result, it is important to maintain low boundary mobility in order to minimize the growth of the recrystallizing grains. A high-niobium, low-interstitial design is essential to meeting this requirement. This design permits one to maintain adequate solute niobium leading to strong

solute drag conditions and low mobility. The high niobium design is also advantageous because the relative contribution of solute niobium in retarding recrystallization becomes significant particularly under conditions of large strain accumulation in low temperature window. This is clearly shown in Figure 3, where a significant contribution from solute niobium in reducing the boundary mobility is predicted at low temperatures in the range 760-840°C. At this point, it should be emphasized that the amount of niobium present in solution is not only a function of base chemistry but also of process variables. It is demonstrated that high solute niobium can be ensured by lowering the starting temperature of finish rolling as well as by decreasing the total time of finish rolling. The control of these process parameters are essential for maximizing the strength contribution from niobium through grain size control, transformation hardening and precipitation strengthening from NbC in ferrite. The latter two mechanisms can enhance the strength by well over 100 MPa.



(c)

(d)

Figure 8: Results of the WUMSI simulation with two sequences of dynamic recrystallization. (a) Rolling schedule which was designed to give dynamic recrystallization twice, (b) experimental load vs. displacement for the designed schedule, (c) the final grain size obtained and (d) the grain size distribution compared with an existing industrial schedule in which dynamic recrystallization occurred once only [22].

5.2. Prevention of partial recrystallization:

The window of partial recrystallization is a function of accumulated strain, temperature and time of holding. The modeling is used to identify the critical strain for static recrystallization as well as the critical strain for dynamic recrystallization for a given rolling schedule. If the threshold for

static recrystallization is exceeded, static recrystallization will commence, which should be avoided. On the other hand, if the strain exceeds the critical strain for dynamic recrystallization, softening by dynamic recrystallization would occur. Thus the model predicts a forbidden region in which partial recrystallization takes place. The lower bound of this region is given by the critical strain for static recrystallization while the upper bound is defined by the critical strain for dynamic recrystallization. The success of the model in avoiding partial recrystallization lies in capturing a comprehensive experimental database on the critical strains for static and dynamic recrystallization. The potential to obtain ultra-fine grain size by dynamic recrystallization to the exclusion of static recrystallization has been demonstrated in laboratory trials on WUMSI on HTP steel.

5.3. Control of starting grain size:

Roughing is used to control the starting grain size for finish rolling in conventional thermomechanical processing. The importance of controlling the starting grain size is demonstrated in strip rolling, where the grain size was refined at one stage in the fourth pass compared to refining the grain size twice by dynamic recrystallization (Figure 8d). The first grain refinement by dynamic recrystallization is intended to correct the variation in the starting grain size. The second and final grain refinement by dynamic recrystallization is intended to refine the grain size. The improvement in grain size by multiple grain refinement is demonstrated in the diagram given in Figure 8d.

5.4. Effect of retarding the kinetics of strain induced precipitation:

The effect of retarding the kinetics of strain induced precipitation through high manganese addition was shown to be beneficial for strain accumulation in high manganese steel particularly in strip rolling, where the interpass time is relatively short of the order of a few seconds. This demonstrates that at short times, the interaction of dislocation with strain induced precipitation plays an important role in altering the driving force for recrystallization. The delay in the onset of strain induced precipitation allows recovery to set in, which decreases the dislocation density, and hence the stored energy for driving the recrystallization.

Further Work

- (i) Basic science investigations on solute niobium are required to validate the model predictions on solute break away.
- (ii) A comprehensive database on the critical strain for static and dynamic recrystallization is required to improve the model prediction.
- (iii) Advanced thermo-chemical techniques are required to determine niobium solute activity in microalloyed steel.

Conclusions

- (1) A quantitative model for time evolution of strain induced precipitation and interaction of solute niobium and strain induced precipitation with recovery and recrystallization is used to analyze the recrystallization behavior of HTP steel and identify the key process parameters to suppress static recrystallization during interpass times.
- (2) The application of the modeling results for steckle mill rolling has identified the importance of adequate solute niobium to retard boundary mobility and hence static recrystallization

under conditions of large strain accumulation in the low temperature window of finish rolling HTP steel in the production of high strength, high toughness plates for oil and gas industry.

(3) The model has clarified the distinct advantage of HTP steel in strip rolling to produce a uniform distribution of ultra-fine grains with high strength and high toughness through promoting dynamic recrystallization at multiple stages.

Acknowledgements

Experimental results on HTP were provided by Dr. Thomas Heller, Thyssen Krupp AG, Dr. R. Kaspar, Max Planck Institute, Dusseldorf, and Dr. C. Klinkenberg and Dipl. Ing. Klaus Hulka, Niobium Products Company, Germany. The contributions of G. R. Purdy, Y. Brechet and C. R. Hutchinson in the development of physically based model are gratefully acknowledged. The authors gratefully acknowledge the financial support of the NSERC of Canada and Niobium Products Company GmbH, Düsseldorf.

References:

[1] A. J. DeArdo, J.M. Gray and L. Meyer, Niobium 81, H. Stuart Ed., (TMS-AIME, 1984): 685.

[2] I. Kozasu et al., Microalloying 75, (Union Carbide Corp., New York, 1977): 120.

[3] S. Lacroix et al, <u>Austenite Formation and Decomposition</u>, <u>A Symposium on the</u> <u>Thermodynamics</u>, <u>Kinetics</u>, <u>Characterization and Modeling of</u>, E. B. Damm and M. J. Merwin, Eds. (TMS, 2003): 367.

- [4] P. D. Hodgson, <u>THERMEC'97</u>, T. Chandra et.al., Eds., (TMS, 1997): 121.
- [5] Z. Q. Sun, W. Y. Yang and J. J. Qi, Materials Science Forum, 475-479 (2005): 49.
- [6] H. S. Zurob et al, *ISIJ International*, 45 (2005): 713.

[7] C.S. Smith, Trans. Am. Inst. Miner. Eng., 175/15 (1948): 15.

[8] B. Dutta, C. M. Sellars, Mater. Sci. Technol., 3 (1987): 197.

[9] H. S. Zurob et al, Materials Science Forum, 500-501 (2005): 123.

[10] G. Zhu et al., <u>42nd Mechanical Working and Steel Processing Conference Proceedings</u> (ISS, 2000): 757.

[11] G. Zhu et al., <u>Proc.Int. Symp. on "Ultra – fine structured steels"</u>, E. Essadiqi and J.Thompson, Eds. (CIM, 2004): 71-85.

[12] H. S. Zurob et al., Acta Materialia, 50(2002): 3075.

[13] B. Dutta, E. J. Palmiere and C. M. Sellars, Acta Materialia, 49 (2001): 785.

[14] F.J. Humphreys and M. Hatherly, <u>Recrystallization and Related Annealing Phenomena</u> (Pergamon Press, 1996).

[15] J. W. Cahn, Acta Metallurgica, 10(1962): 789.

[16] G. Robiller and L. Meyer, <u>1st Riso International Symposium on Metallurgy and Materials</u> <u>Science</u>, N. Hansen, A. R. Jones and T.Leffers, Eds. (RISO National Laboratory, 1980): 311.

[17] E. J. Palmiere, C. M. Sellars and S. V. Subramanian, <u>Niobium Science and Technology-Proc. Int. Symp. Niobium 2001</u>, (Niobium 2001 Ltd., 2001): 501.

[18] L. E. Collins et al, <u>Microalloying 95 Conf. Proc.</u>, M. Korchinsky et al., Eds., (ISS_AIME, 1995): 141.

[19] S.V.Subramanian and H. Zou, Processing, <u>Microstructure and Properties of Microalloyed</u> and Other Modern High Strength Low Alloy Steels, (ISS-AIME, 1991): 23.

[20] S. V. Subramanian et al., <u>Proc. Int. Symp. on Low carbon steels for the 90's</u>, R. Asfahani and G. Tither, Eds., (TMS-AIME, 1993): 313.

[21] G.Zhu, D.Tan and M.Subramanian, <u>42nd Mechanical Working and Steel Processing</u> <u>Conference Proceedings</u> (ISS, 2000): 757.

[22] S.V. Subramanian et al., <u>Proceedings of the International Conference on Thermo-</u> <u>mechanical Processing: Mechanics, Microstructure and Control</u>, J.H. Beynon, Ed., (Institute of Metals, 2003): 148.