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Production of 20 mm Thick API PSL 2 X60 and X70 Grade Plates from a Nb-Ti Microalloyed Steel

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ABSTRACT

This study was undertaken to determine the controlled rolling and cooling conditions for the production of 20 mm thick American Petroleum Institute (API) X60 and X70 grade steel plates. Nb-Ti microalloyed steel slabs of 200 mm thickness were rolled at four different finish rolling temperatures (conventional, 950°C, 850°C and 800°C). In some trials, a water table was employed to provide accelerated cooling just after finish rolling. Mechanical tests (tensile, impact and drop weight tear-DWTT) and microstructural examinations were performed on the samples taken from the trial production plates. Fine grained and essentially ferritic microstructures with strength values satisfying the minimum yield strength requirement of 415 MPa for the API X60 grade were easily obtained in all rolling conditions. However, the minimum yield strength requirement of 485 MPa for the API X70 grade was reached only when accelerated cooling was applied after finish rolling. The minimum 85% shear fracture required by the DWTT of the API PSL 2 specification could be met when the finish rolling temperature was lowered or when accelerated cooling was applied regardless of the finish rolling temperature.

Keywords:
Controlled rolling, API X60, API X70, Accelerated cooling, Pipeline steel, DWTT

INTRODUCTION

There are currently hundreds of thousands of kilometers long petroleum and natural gas pipelines installed worldwide and new lines are being installed continuously [1, 2]. As the demand for petroleum and natural gas by the developed and developing countries has increased over time, the pipelines of larger diameter pipes that can work at higher operating pressures were required [2, 3]. This necessitated the development of steel plates with higher mechanical properties from which these pipes could be produced [4].

Pipeline steels should have high weldability since pipelines are installed by joining pipes to each other by welding. For this reason, pipeline steels must have low carbon content and low carbon equivalent values and this essentially implies ferritic microstructures [5, 6]. As a way of having high strength, high ductility and high weldability at the same time, grain size refinement mechanism is utilized in pipeline steels. By decreasing the ferrite grain size, both strength and toughness can be simultaneously increased without increasing the carbon content of the steel [5]. Pipeline steels also contain low amounts of alloying elements that are mainly added due to their roles in decreasing the ferrite grain size. Therefore, these steels are grouped under high strength low alloy (HSLA) steels [6, 7].

Throughout the world, the specifications established by American Petroleum Institute (API) are followed for the petroleum and natural gas pipelines. API Specification 5L classifies pipelines steels according to their required yield strength values [8]. For example, API X52 corresponds to a minimum yield strength requirement of 52200 psi. API grades X52 to X70 were developed during the 1950s to 1970s, while newer grades, X80 to X120, were adopted later [9, 10].

Based on the required properties, API 5L classifies pipeline steels in two main product specification levels as PSL 1 and PSL 2. The main difference between PSL 1 and PSL 2 specifications is towards the deformation behavior of the steel rather than its strength. As compared to the PSL 1, PSL 2 has additional impact and ductility requirements. Some of the mechanical property requirements for the API X60 and X70 grades that constitute the focus of this study are given in Table 1.

Table 1. Some mechanical property requirements for the API X60 and X70 grade steels based on the API 5L PSL 1/PSL 2 specification [8].

Grade	Yield Strength (MPa)	Tensile Strength (MPa)	YS/TS Ratio (max)	% Elongation (min)	Impact Energy* at 0°C (J, min)	DWTT**
PSL 1 X60	min. 415	min. 520	-	24	-	
PSL 1 X70	min. 485	min. 570	-	22	-	
PSL 2 X60	415-565	520-760	0.93	24	27-54	≥ 85 %
PSL 2 X70	485-635	570-760	0.93	22	40-68	≥ 85 %

*Required impact energy depends on the outer diameter of the pipe and the wall thickness.
**Drop Weight Tear Test (DWTT) is used to determine the degree of shear fracture.

The development of steel plates with superior mechanical properties to be used in pipelines became possible by utilizing the rolling operation not only for reducing slabs to the specified thickness but also for creating the microstructures that would produce the targeted mechanical properties. Controlled rolling or Thermomechanical Controlled Rolling (TCR) covers the selection of rolling temperatures, reduction ratios and rolling speeds considering the microstructural evolution behavior (kinetics of phase transformations, recrystallization, grain growth, and precipitation reactions, etc.) of the steel to obtain the desired microstructures [4, 6].

In typical rolling operations, the overall reduction in thickness is accomplished in two main stages, namely rough rolling and finish rolling. Conventionally, shaping is performed in successive passes where the rolling load per pass is kept at low values and rough rolling is followed by the finish rolling without any intentional waiting time in between. Hence, the whole shaping process is completed at high temperatures.

In controlled rolling, however, finish rolling is done at lower temperatures in order to prevent or minimize the recrystallization and grain growth of the deformed austenite so that the number of sites for ferrite nucleation is maximized. Therefore, there is an intentionally given waiting time between the rough and finish rolling stages, which is needed for the cooling of the steel being rolled. Inevitably, rolling at lower temperatures brings higher loads to rolls as compared to the conventional rolling since the flow strength of the steel is higher at lower temperatures [11]. The other concern caused by the waiting time between the rough and finish rolling stages is that the throughput of the rolling mill decreases with increasing waiting time and this means higher cost per produced plate.

The temperature below which austenite does not recrystallize is called non-recrystallization temperature (T_{nr}). When the finish rolling is performed below the T_{nr}, the nucleation of ferrite occurs while the deformed austenite grains are in the pancake form and thus, there are a lot of nucleation sites available (austenite grain boundaries and deformation bands) for a given volume [12]. Hence, microstructures consisting of finer ferrite grains can be obtained (Fig. 1).

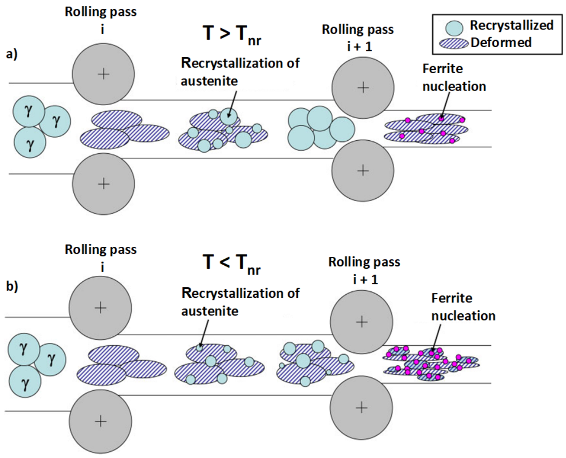

Figure 1. The effect of rolling a) above and b) below the Tnr temperature. The austenite grains deformed below the T_{nr} take pancake form and they provide a higher number of nucleation sites for austenite to ferrite transformation as recrystallization is limited. Adapted from [12].

The alloying elements such as Nb, Ti and V raise the T_{nr} so that the processing window for controlled rolling can be opened towards higher temperatures and thus, less waiting time is required. This also eases the rolling loads. The most effective alloying element to raise the T_{nr} is Nb [13]. It is possible to calculate the T_{nr} for a given steel composition through empirical equations of Boratto and Bai [13]:

Boratto equation:

$$T_{nr}(°C) = 887 + 464*\mathbf{C} + \left(6445*\mathbf{Nb} - 644*\sqrt{\mathbf{Nb}}\right) + \left(732*\mathbf{V} - 230*\sqrt{\mathbf{V}}\right) + 890*\mathbf{Ti} + 363*\mathbf{Al} - 357*\mathbf{Si} \quad (1)$$

Bai equation:

$$T_{nr}(°C) = 174\ log\left[\mathbf{Nb}*\left(\mathbf{C}+(12/14)*\mathbf{N}\right)\right] + 1444 \quad (2)$$

In addition to the T_{nr}, another temperature to pay attention to in controlled rolling is the austenite to ferrite transformation start temperature on cooling (A_{r3}) of the steel being rolled. If the rolling is done below the A_{r3} then ferrite grains are also plastically deformed during rolling since the steel is in two phase ($\alpha + \gamma$) region.

The A_{r3} for given steel can be calculated using the Ouchi equation [14]:

$$A_{r3}(°C) = 910 - 310*\mathbf{C} - 80*\mathbf{Mn} - 20*\mathbf{Cu} - 15*\mathbf{Cr} - 55*\mathbf{Ni} - 80*\mathbf{Mo} + 0.35*(t-8), \quad (3)$$
$$t: \textit{plate thickness } (mm)$$

Fine ferritic microstructures obtained by sole controlled rolling can satisfy the mechanical properties required for some API grades. However, in order to reach higher strength levels without increasing the carbon content, which deteriorates some properties such as weldability, accelerated cooling right after finish rolling was devised so that even finer and different ferritic microstructures (e.g. acicular, bainitic) can be produced [4, 9]. The practice which utilizes both controlled rolling and accelerated cooling together is called Thermo-Mechanical Controlled Processing (TMCP) [15]. A schematic showing the conventional rolling, thermomechanical controlled rolling and thermomechanical controlled processing is given in Fig. 2.

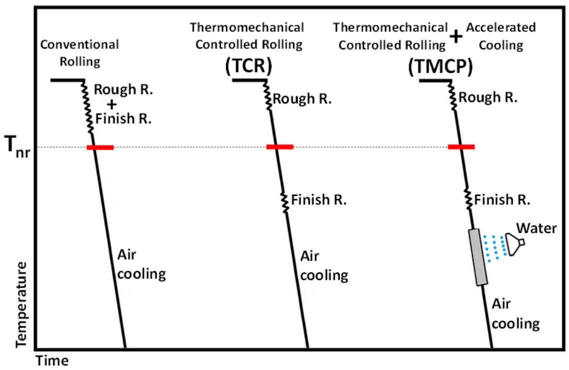

Figure 2. A schematic representation of conventional rolling, thermomechanical controlled rolling (TCR) and thermomechanical controlled processing (TMCP). T_{nr} = Non-recrystallization temperature.

Being an energy corridor in its region, domestic production of coiled API PSL 1 X52 and X60 grade steels with less than 20 mm thickness were previously studied in Turkey [16, 17] and they are commercially available. On the other hand, the domestic production of 20 mm or thicker API PSL 2 X60 and X70 grade steel plates, which are not coiled, was not realized yet in spite of the growing demand.

Producing thicker steel plates of higher grades is not straightforward due to a few reasons. First, the total amount of deformation experienced by the slab decreases as the final plate thickness increases. This, inevitably, makes it more difficult to obtain fine grained microstructures that would give the required strength values. Secondly, satisfying the ductility and toughness requirements of PSL 2 specification brings an additional challenge. Therefore, starting with the appropriate alloying, the controlled rolling and cooling conditions should be combined in such a way that the rolling mill would be able to produce these higher grade steel plates while minimizing the loss in throughput as much as possible.

As a contribution to the pre-commercialization studies [18], the effects of finish rolling temperature and cooling conditions on the production of 20 mm thick API PSL 2 X60 and X70 steel plates from 200 mm thick Nb-Ti microalloyed slabs were investigated in this exploratory work.

MATERIAL AND METHODS

Material

Two full size (12000x2000x200mm) slabs with chemical compositions (Table 2) compliant to the API X60 and X70 grades were sliced into small slabs (2000x2000x200mm) for the plate rolling trials. Trials were done in the hot rolling mill of an integrated iron and steel plant.

Rolling Trials

As mentioned in the Introduction section, the non-recrystallization temperature (T_{nr}) and the austenite to ferrite transformation start temperature (A_{r3}) should be considered when deciding for the controlled rolling conditions. Using the Boratto Eq.(1) and Bai Eq.(2) equations given before the T_{nr} of the slabs used were calculated as 1062°C and 1040°C. The A_{r3} of the slabs were found as 751°C based on the Ouchi formula Eq.(3) .

The finish rolling temperatures and cooling conditions chosen for the rolling trials are listed in Table 3 and graphically shown in Fig. 3. In all trials, the thickness to enter the finish rolling was kept fixed at 60 mm and this corresponds to 70.0 % reduction in rough rolling and 66.7 % reduction in finish rolling, respectively. The number of roll passes and the reduction ratios per pass for the rough rolling (200 mm -> 60 mm) and the finish rolling (60 mm -> 20 mm) stages were automatically determined by the rolling mill control software. A water table was used for the accelerated cooling of the rolled plates in some experiments.

Table 2. API X60/X70 specification and the chemical composition of the slabs used in this work (wt.%).

Element	C	Si	Mn	P	S	V	Nb	Ti	Cu	N.	Cr	Mo
API X60 and X70 (max)	0.12	0.45	1.60	0.025	0.015		Nb + V + Ti ≤ 0.15		0.50	0.50	0.50	0.50
Used Slabs (max)	0.09	0.25	1.60	0.020	0.010	0.01	0.06	0.03	0.10	0.10	0.20	0.15

Table 3. Rolling and cooling conditions used in the trial productions.

Trial Code	*Temperature to enter Finish Rolling (°C)*	*Accelerated Cooling after Finish Rolling*
Conventional	*1090*	-
950°C-TCR	*950*	-
950°C-TMCP	*950*	*Yes*
850°C-TCR	*850*	-
850°C-TMCP	*850*	*Yes*
800°C-TCR	*800*	-
800°C-TMCP	*800*	*Yes*

In all trials, the enter and exit temperatures for the rough rolling (200mm -> 60mm) were about 1140°C and 1090°C, respectively.

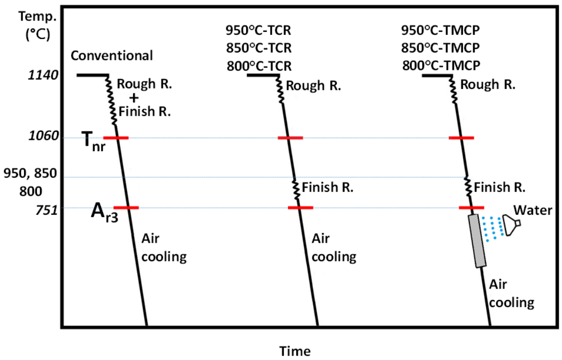

Figure 3. Graphical representation of the rolling and cooling conditions employed in the trials.

Characterization of the Trial Production Plates

Tensile tests, Charpy impact tests, drop weight tear tests (DWTT) and microstructural examinations were performed on the samples cut from the front, middle and end locations along the rolled plates to determine whether the related requirements of API 5L specification (Table 1) were satisfied. Tensile tests were done according to the ASTM A370 [19] standard using a Zwick/Roell Z1200 tensile test machine. The dimensions of the tensile test samples were 38 mm, 50 mm and 250 mm, respectively for the width, gauge and total length as shown in Fig. 4.a. The thickness of the tensile test samples was 20 mm which is the final thickness of the rolled plates. Impact tests were performed at 0°C and -20°C using standard Charpy notched samples of 10 mm*10 mm*55 mm. API RP 5L3 was followed for the DWTT sample dimensions (Fig. 4.b), implementation of the tests and interpretation of the results [20].

Microstructural examinations of 4% nital etched samples were performed using a Nikon Epiphot 200 inverted metallurgical microscope. Average ferrite grain size values of each sample based on the planimetric procedure of the ASTM 112 standard [21] were determined using the Clemex Vision (Clemex Technologies Inc., Longueuil, QC, Canada) and Image J [22] image processing softwares.

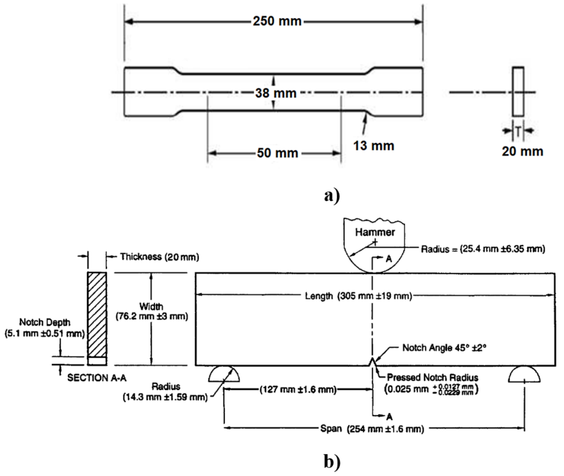

Figure 4. Sample dimensions for a) the tensile tests and b) the DWTT tests.

RESULTS AND DISCUSSION

Tensile Test Results

The results of the tensile tests of the samples taken from the trial production plates are given in Table 4. The yield and tensile strength values of the conventionally rolled sample were determined as 427 MPa and 583 MPa, respectively. The corresponding values were 444 MPa and 585 MPa after finish rolling at 950°C (950°C-TCR), 425 MPa and 550 MPa after finish rolling at 850°C (850°C-TCR) and finally, they were 456 MPa and 562 MPa after finish rolling at 800°C (800°C-TCR). These results show that the yield and tensile strength values of the samples did not change significantly with decreasing finish rolling temperature when only controlled rolling was employed (i.e., no accelerated cooling). In a similar work by Korczak [23] to produce 20 mm thick plates, an average yield strength of 535 MPa and a tensile strength of 618 MPa were obtained after finish rolling of a 0.03% Nb - 0.07% V microalloyed steel at 800°C. However, it should be noted that, in addition to the differences in alloying, the starting slab was thicker (225 mm) and a different rolling schedule (77.8% reduction in rough rolling: 225 mm -> 50 mm, 60.0% reduction in finish rolling: 50 mm -> 20 mm) was used in that study.

The implementation of the accelerated cooling right after the finish rolling significantly increased both the yield and tensile strength values. The yield and tensile strength values of the sample finish rolled at 950°C and then subjected to accelerated cooling (950°C-TMCP) were 511 MPa and 638 MPa, respectively. The corresponding values were 502 MPa and 617 MPa after finish rolling at 850°C with accelerated cooling (850°C-TMCP) and finally, they were 528 MPa and 633 MPa when the finish rolling was done at 800°C followed by accelerated cooling (800°C-TMCP). Apparently, the application of accelerated cooling greatly improved both yield and tensile strength values while they were still rather independent of the finish rolling temperature.

It was observed that the TMCP samples had significantly higher deviations in their tensile test results as compared to the TCR samples (Table 4). The root cause for this higher deviation is thought to be due to the uneven accelerated cooling of the rolled plates by the water table.

Table 4. Tensile test results of the trial production plates.

Trial Code	Yield Strength (MPa)	Tensile Strength (MPa)	YS / TS Ratio	% Elongation
Conventional	427 ± 6.8	583 ± 5.1	0.73	34.5 ± 0.2
950°C-TCR	444 ± 6.7	585 ± 2.0	0.76	33.3 ± 0.5
850°C-TCR	425 ± 11.8	550 ± 1.5	0.77	34.2 ± 0.4
800°C-TCR	456 ± 6.2	562 ± 1.6	0.81	34.5 ± 0.3
950°C-TMCP	511 ± 28.0	638 ± 20.8	0.80	35.5 ± 1.4
850°C-TMCP	502 ± 38.7	617 ± 20.3	0.81	35.9 ± 2.8
800°C-TMCP	528 ± 34.2	633 ± 27.2	0.83	34.0 ± 1.1

A comparison of the yield and tensile strength values of the trial production plates with the API 5L specification shows that the minimum requirements for the API X60 grade were met by all samples (Fig. 5). On the other hand, the strength requirements of the API X70 grade were satisfied by only three of the seven trial productions, namely, 950°C-TMCP, 850°C-TMCP, and 800°C-TMCP. These results show that accelerated cooling is instrumental in obtaining API X70 grade plates from these slabs.

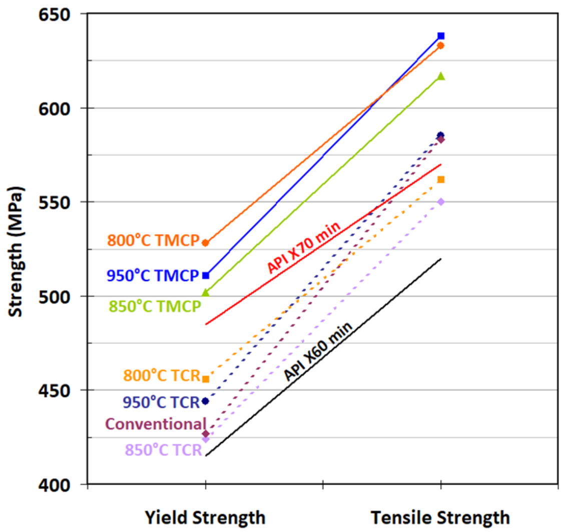

Figure 5. Comparison of the yield and tensile strength values of the trial production plates with the minimum requirements of the API X60 and X70 grades.

Impact Test and DWTT Results

As mentioned previously, the main difference between the PSL 1 and PSL 2 specifications is the requirements towards the deformation behavior of the pipeline steel rather than its strength (see Table 1). The impact test and DWTT results of the trial production plates are given in Table 5. It is found that except 950°C-TCR all samples satisfied the impact energy requirement of API PSL 2.

Table 5. Impact test and DWTT results of the trial production plates.

Trial Code	Impact Test Temperature (°C)	Impact Energy (J)	DWTT (%)
Conventional	-20°C	76 ± 17.2	0
950°C-TCR	-20°C	19 ± 4.8	0
850°C-TCR	-20°C	250 ± 3.4	90
800°C-TCR	-20°C	213 ± 5.9	90
950°C-TMCP	0°C	214 ± 6.7	90
850°C-TMCP	0°C	234 ± 13.8	95
800°C-TMCP	0°C	214 ± 6.9	95

For the DWTT results, the conventionally rolled plate resulted in 0% shear fracture indicating its brittle behavior (Fig. 6.a). Almost the same result was obtained after controlled rolling at 950°C, as seen in Fig. 6.b. Since the requirement of DWTT ≥ 85% is not satisfied, these two plates were appropriately labeled as API PSL 1 X60 based on their strength values. On the other hand, controlled rolling at lower temperatures (850°C-TCR and 800°C-TCR) or the application of accelerated cooling after a higher finish rolling temperature (950°C-TMCP) produced DWTT results with ≥ 85 % shear fracture (see Figure 6.c and Figure 6.d, respectively). Thus, these samples satisfied the ductility requirement of the API PSL 2 specification.

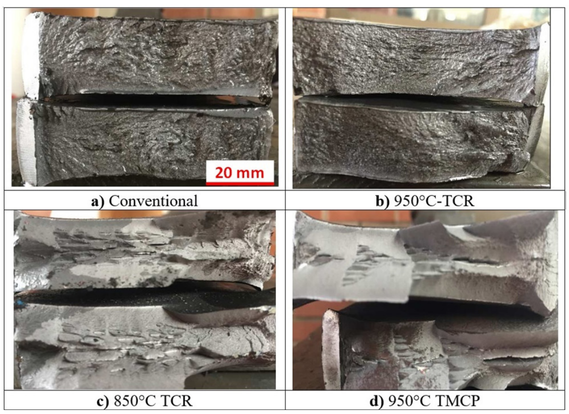

Figure 6. Fracture surfaces of the DWTT samples; a) conventionally rolled, b) finish rolled at 950°C, c) finish rolled at 850°C, and d) finish rolled at 950°C and accelerated cooling applied.

In order to determine the API grades satisfied by the samples, the mechanical properties given in Table 4 and Table 5 were compared with the API requirements listed in Table 1. The rolling conditions of the samples and the corresponding API grades satisfied are listed in Table 6.

Table 6. API grades satisfied by the samples based on their mechanical properties.

Trial Code	API Grade
Conventional	PSL1/X60
950°C-TCR	PSL1/X60
850°C-TCR	PSL2/X60M
800°C-TCR	PSL2/X60M
950°C-TMCP	PSL2/X70M
850°C-TMCP	PSL2/X70M
800°C-TMCP	PSL2/X70M

Microstructural Examinations

For microstructural examinations, samples were cut from the start, middle and end sections of each trial production plate. Optical microstructural images of the conventionally rolled, 850°C-TCR and 800°C-TMCP samples are given in Fig. 7. In accordance with the tensile test results presented in Table 4, it was observed that finish rolling at a temperature of 850°C (Fig. 7.c-d) and finish rolling at a temperature of 800°C followed by accelerated cooling (Fig. 7.e-f) produced much smaller ferrite grains as compared to the conventional rolling as shown in Fig. 7.a-b. Ferrite was mostly in polygonal form in all samples, which is a typical characteristics found in X60 and X70 grades [24].

Using the optical microstructural images, the average ferrite grain size values of the samples were determined and listed in Table 7. It is clearly observed that the application of accelerated cooling just after finish rolling is very effective in producing smaller grained (3.9 - 4.1 μm) microstructures as compared to the ambient cooling (5.2 - 6.4 μm). The average grain size values determined in this work were inline with the values reported in the literature [25, 26]. For example, in a similar study Masumi et. al found the average ferrite grain size to be around 5-6 μm without accelerated cooling [25]

CONCLUSION

As a part of a study to produce 20 mm thick API PSL 2 X60 and X70 grade steel plates, 200 mm thick Nb-Ti microalloyed slab pieces were rolled at different finish rolling temperatures (conventional, 950°C, 850°C and 800°C) and cooling conditions (ambient or accelerated cooling).

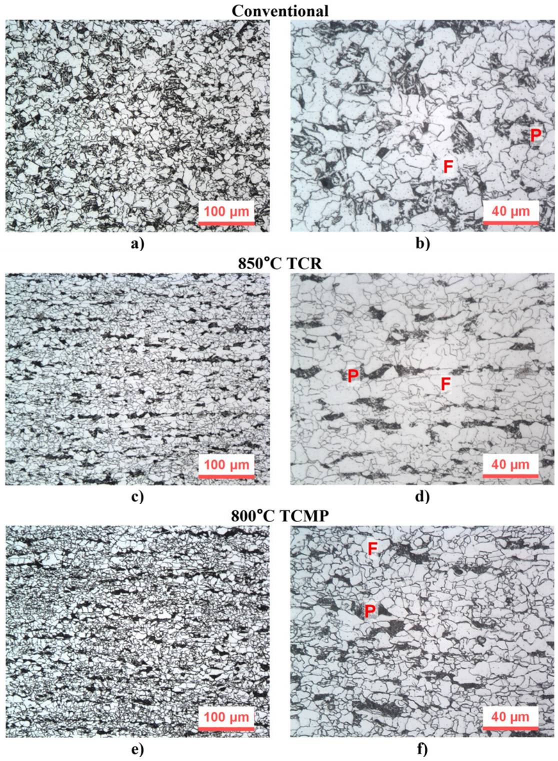

Figure 7. Optical microstructural images of the some of the samples taken from the trial production plates: a)-b) conventionally rolled, c)-d) finish rolled at 850°C, and e)-f) finish rolled at 800°C and accelerated cooled. Images were taken at 200x and 500x for the left and right columns, respectively. Etchant: 4 % nital.

Table 7. Average ferrite grain size values of the samples taken from the trial production plates.

Trial Code	Average Grain Size (μm)	ASTM Grain Size Number
Conventional	6.9	11.5
950°C-TCR	6.4	11.5
850°C-TCR	5.7	12.0
800°C-TCR	5.2	12.0
950°C-TMCP	3.9	13.0
850°C-TMCP	4.1	13.0
800°C-TMCP	3.9	13.0

- It is found that the strength requirements of the API X60 grade were easily satisfied in all rolling conditions.
- Very small grained and essentially ferritic microstructures producing the strength values required for the API X70 grade were obtained only when accelerated cooling was applied after the finish rolling.
- The drop weight tear test requirement of the PSL 2 specification was achieved when the finish rolling was started at lower temperatures of 850°C and 800°C regardless of the cooling type or when accelerated cooling was applied after a higher rolling temperature of 950°C.

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