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Effect of Reduction Ratio Below Austenite Recrystallization Stop Temperature on Mechanical Properties of an API X70M PSL2 Line Pipe Steel

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Abstract

Steel grades having high toughness and high strength are required for line pipes since gas and oil should be transported through them at high pressures. Thermo-mechanically controlled rolling processes are used for increasing both toughness and strength at low temperatures since the line pipes are exposed to harsh climatic conditions at full length and rather severe service conditions. Charpy, DWTT and tensile tests were conducted to determine of mechanical properties and measured toughness values were evaluated considering various criteria in this study. Fractured surfaces were examined by means of SEM to distinguish the ductile and brittle fracture areas. Thermo-mechanical rolling trials were performed at the temperature below the non-recrystallization temperature of austenite on an API X70M PSL2 grade steel, to increase the strength without sacrificing the toughness. Different reduction ratios between 60,7% and 72,8% were utilized and the effect of reduction ratios on mechanical properties and microstructures were investigated during the trials. It was observed that final grain size decreased and strength and toughness increased with increasing reduction ratios.

Keywords: X70, thermo-mechanical processing, Charpy impact test, drop weight tear test, toughness

1. INTRODUCTION

High-strength low-alloy (HSLA) steels which are commonly used for different structural applications have been developed by thermomechanically controlled hot rolling process to particularly combine high strength with good toughness [1,2]. However, due to recently increasing demand to use these steels in harsh service conditions, impact toughness at lowtemperatures has become a primary concern and key criteria for them.

The demand for natural gas and crude oil rapidly increases with industrial developments. Since many gas and oil fields are located in remote areas, it is necessary to transport the gas and oil

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from these areas to consumption areas economically. In this context, line pipes are considered as the most economical and safe transportation of the gas and oil across distant miles. In order to reduce the transportation costs by long-distance line pipes, higher operating pressures in line pipes are expected, which consequently require higher wall thickness or higher strength steel with excellent combination of high toughness and strength [3-9]. Therefore, the increasing demand for oil and gas transportation is a driving force for the development of specialized and cost-effective line pipe steels with improved toughness, strength, weldability and corrosion resistance [10-13].

Increasing the strength of line pipe material can reduce significantly the wall thickness and consequently weight of the material. Such savings are important especially for the installation of line pipes in distant areas, where any weight reduction can be crucial in reducing the basic costs such as the amount of welding consumables, transportation and manipulation of the pipes during construction [14,15].

In addition to high toughness, resistance to crackpropagation is also extremely important for the development of high strength line pipe steels. Therefore, in practice, appropriate combinations of toughness and strength ensuring resistance to rapid crack propagation are desired [16].

In the line pipe steels, there is no strictly specified elemental composition and microstructure [17,18]. The thermo-mechanically controlled process (TMCP) in hot rolling stage introduces ideal microstructure resulting in desired mechanical properties. In this respect, alloying composition is essentially designed to assist appropriate TMCP.

In TMCP, reheating, rolling and coiling temperatures, distribution of rolling reductions among the rolling passes and cooling rate can be adjusted to get the desired properties [10]. For instance, low finishing temperature and fast cooling rate significantly produces higher strength than high finish rolling temperature and slow cooling rate. Controlled rolling of heavy gauge material at high temperatures is an exclusive phase of TMCP temperatures, which finally produces various combinations of ferrite-pearlite-bainite in final microstructure.

Generally, controlled rolling of high strength line pipe steels such as API (The American Petroleum Institute) X70 steel consists of two phases [19]:

- Rough rolling, which is carried out at temperatures above non-recrystallization temperature (T_{NR}) of austenite and aims to achieve a recrystallized polygonal austenite microstructure across the thickness.

- Finish rolling, which is carried out below T_{NR} , but above A_{r3} (beginning temperature of transformation from austenite to ferrite during cooling) in order to obtain deformed nonrecrystallized in austenitic microstructure.

The T_{NR} can be determined or estimated through many methods. From the literature, T_{NR} can be estimated through empirical formulas and laboratory methods [20-23]. The Boratto's equation [20] given in Equation 1 is the most known empirical formula to estimate T_{NR} (°C).

 $T_{NR} (^{\circ}C) = 464C + 887 + (6445Nb - 644\sqrt{Nb}) + (732V - 230\sqrt{V}) + 363Al - 357Si + 890Ti$ (1)

where C, Nb, V, Al, Si and Ti are the elements in the steel in weight percent (wt.%).

The accelerated cooling following controlled rolling determines the final microstructure through phase transformation of hot rolled austenite. After controlled rolling, deformed austenite must have high density of dislocations and grain boundaries to permit the nucleation of new phases [24].

The introduction of accelerated cooling after controlled rolling led to the production of higher strength steels [5]. Higher strength line pipe steels have been very successfully exploited worldwide in line pipe construction. These have been produced by accelerated cooling at cooling rates of 10 to 20 °C/s finalized with a coiling

temperature of 500-600°C, after which air cooling is used [14].

Additional reduction in grain size of the final microstructure is achievable by increasing the degree of rolling reduction; arranging the final rolling stage and abbreviating the interval between the end of rolling and the onset of cooling [25].

The production of X70 grade for gas and oil line pipes today is based on the controlled rolling with accelerated cooling [26]. Because of the nature of cooling in coil form, non-uniform structures are formed in coil products. Hence, the mechanical properties are non-uniform over the strip length since the external and internal turns in a coil are cooled more rapidly than the middle turns. To avoid a cooling-rate gradient and ensure uniform mechanical properties over the length of the strip, the coils of line pipe steels are subjected to accelerated cooling [26].

Low carbon steels have very good weldability and strength decrease due to carbon content decrease is compensated by the addition of vanadium, niobium and titanium in the micro-scale [27]. Softening caused by the reduction of carbon content is compensated by grain refining and precipitation hardening. The influence of microalloying elements on strengthening depends on the controlled rolling and accelerated cooling conditions.

Simultaneous achievement of good weldability, high impact resistance and high strength is possible by keeping carbon, sulfur, phosphorus, nonmetallic inclusions, and gases at the lowest possible level and appropriate selection of microalloy additions (Nb, V, Mo, Al, Ti) and Mn assuring achievement of required yield strength after applying meticulously designed TMCP [27]. The most significant reason of keeping low amount of carbon is, to get one-phase microstructure having fine grains after TMCP [20-28].

Line pipes can be suddenly fractured when the cracks initiate and propagate rapidly by explosion, impact or earthquake as they have been used under high pressure and low temperature conditions [28]. Fracture properties especially at low temperatures of line pipe steels have been widely evaluated by carrying out drop-weight tear tests (DWTT) and Charpy impact tests [4, 5-8, 16, 29-36], and the test results may show variations as the notch shape, dimension and thickness of the test specimens changes [28]. The DWTT has been adopted as the official test condition by the API (American Petroleum Institute) for line pipe steels since DWTT is quite consistent with the fracture appearance transition occurring in actual fracture period of line pipe steels [6].

85 % SATT (shear appearance transition temperature) obtained from the drop weight tear tests is used as a standard to determine FPTT (fracture propagation transition temperature) [5,8]. Chevron Notch (CN) DWTT specimens or Press Notch (PN) DWTT specimens are used for FPTT of line pipe steels according to the API RP 5L3 specification [37]. The absorption energies from the press notch and chevron notch DWT tests and Charpy impacts tests all can be used as the criteria to measure the unstable ductile fracture resistance in line pipe steels [5].

The 85 percent, % SA (shear area) measured from the DWT test is generally known as the "Battelle 85 %. Shear Area criterion" [6] to prevent the propagation of brittle fracture: if the measured SA is higher than 85 %, then material is supposed to withstand against the brittle fracture [17].

Crack-tip opening angle (CTOA) method and new approaches to DWT test are also introduced for systematic and more precise analyses as the toughness is greatly improved by ongoing studies on line pipe steels. However, the Charpy impact test being a simple testing method is still most commonly used method to evaluate transition temperature and absorbed energy of line pipe steels [6,16, 17].

As the rolling below T_{NR} is of primary concern to meet high toughness values in TMCP, the effect of hot rolling reduction ratio below T_{NR} on ductile fracture behavior in API X70M PSL2 line pipe steel was investigated in this study. Other rolling parameters and cooling rates were chosen identical in all trials to analyze the effect of hot rolling reduction ratio below T_{NR} . [10-12,16].

2. MATERIALS AND METHODS

An API X70M PSL2 grade steel was used in the industrial trials. Chemical composition of the steel is as follows (wt.%): C:0.052; Si:0.211; Mn:1.63; Cu:0.16; Ni:0.21; Cr:0.13; Mo:0.12; N:0.0063 and Nb+V+Ti:0.089.

Slabs in size of 220 mm (t) x 8500 mm (l) x 1500 mm (w) were hot rolled in one stand 4-high roughing mill with 5 passes and seven stand 4-high finishing mill with 7 passes under thermomechanical controlled rolling conditions. Before rolling, all slabs were heated to 1220 °C and soaked for a specific period in the soaking zone of a walking beam type reheating furnace in order to dissolve all the Nb in austenite.

Two different transfer bar thicknesses were used for identical roughing conditions, in which final roughing temperatures were above the calculated T_{NR}. Thicknesses of the slabs were reduced to 73.5 and 56 mm in 5 passes in the roughing mill. Before transferring to the finishing mill, special practice was applied to the transfer bars to obtain a desired homogenous finish rolling temperature below the T_{NR} of material. The T_{NR} for this steel was calculated as 1134 °C according to the Equation 1. Entry of the materials to the finishing mill was conducted at about 1000-980 °C. A primary grain refinement effect was expected by rolling in the non-recrystallized region of austenite with high rolling reduction ratios. Rolling was finished at 800-820 °C (FRT, the temperature at the exit of the last finishing rolling stand). Following finish rolling, the materials were accelerated cooled on run-out table with a cooling rate of ~12-15 °C/s and coiled at 550-600 °C. In the trials, total reduction ratio varied from 60.7 % to 72.8 %.

2.1. Microstructural analysis

The cross-sectional metallographic specimens taken from transversal to rolling direction were polished and etched by a 2 % Nital solution, and microstructures were observed by an optical microscope.

2.2. Tensile and Charpy impact tests

Tensile rectangular specimens with 38 mm gauge width and 50 mm gauge length were prepared in the transversal, diagonal (45° to the rolling direction) and longitudinal directions and were tested at a crosshead speed of 5 mm/min using a 600 kN Zwick tensile test machine at room temperature. Charpy impact tests were performed on Charpy V-notch (CVN) specimens [38] in size of 10 mm × 10mm × 55 mm in transversal orientation in a temperature range from -80 to 0 °C using a Zwick impact tester of 450 J capacity. Ductile and brittle zones on the fracture surfaces of Charpy samples were also investigated by scanning electron microscope (SEM).

2.3. Drop weight tear tests

DWTT specimens were prepared in size of 305 mm \times 76.2 mm \times 20 mm in transversal direction in accordance with the API 5L3 [37] and ASTM E436-91 [39] specifications, and then a pressed notch was applied on to them. These specimens were tested with a DWT testing machine having a maximum energy capacity of 50,000 J at 0 °C. The special refrigerant was used to cool the DWTT specimens down to 0 °C by putting ethanol in the specimen pool in where specimens were immersed for 20 min. and then immediately tested. The percent shear area (pct. SA) [37] was calculated according to the Equation 2.

pct. SA =
$$(((71-2t)t-(3/4)ab) / (71-2t)t) \times 100$$
 (2)

where a, b and t are the width of cleavage fracture, length of cleavage fracture and full plate thickness respectively [37, 39].

3. RESULTS AND DISCUSSION

Variation of yield strength depending on the reduction ratio is shown in Figure 1. It was observed that yield strength increased as the reduction ratio below T_{NR} increased. The increase is more obvious on the transversal specimens with higher yield strength values, whereas lower yield

strength values were obtained on the longitudinal specimens.



Figure 1. Variation of yield strength with the reduction ratio below T_{NR} .

Compared to yield strength, it was observed that tensile strength slightly increased as the reduction ratio increased (Figure 2). Higher tensile strength values were obtained on the transversal specimens; whereas lower tensile strength values were observed on the longitudinal specimens.



Figure 3. Variation of elongation with the reduction ratio below $T_{\text{NR}}.$

Pct. SA value in DWTT, which is the key property in many line pipe applications increases with the increasing reduction ratio: pct. SA is about 50 at the reduction ratio of 60.7 % and increases up to 90 as the reduction ratio has been increased to 72.8 % (Figures 4 and 5). Here, only the 72.8 % reduction ratio below T_{NR} meets the "Battelle's criterion" to prevent the brittle fracture propagation.



Figure 4. Variation of pct. SA in DWTT with the reduction ratio below T_{NR} .



Figure 2. Variation of tensile strength with the reduction ratio below T_{NR} .

It was observed that elongation slightly decreased with increasing reduction ratio (Figure 3).

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Reduction ratio below T _{NR} (%)	Sample 1	Sample 2	Sample 3
72.8			
70.3			
67.5			
64.2			
60.7			

Figure 5. Fracture surfaces of DWTT samples associated with reduction ratio below T_{NR} .

The effect of total % reduction ratio under T_{NR} on the Charpy V notch impact values on different test temperatures is shown in Figure 6. The improving effect of reduction ratio below T_{NR} on the V notched Charpy impact values can significantly be seen at -80 °C in Figures 6 and 7. Charpy V notch impact values increased and the type of the fracture turns into the ductile fracture from brittle fracture as the reduction ratio increased. The increase in the impact energy values is more obvious on the samples tested at -80 °C than the samples tested at higher temperatures. The increase of the reduction ratio ensures achieving the desired crack-propagation resistance.



Figure 6. Variation of Charpy V-notch impact values with the reduction ratio below T_{NR} .

Microstructural variations on the investigated steel are shown in Figure 8 depending on the reduction ratio. An obvious decrease in ferrite grain size (from 12 to 13,5 ASTM Grain Size Number according to linear intercept method) is observed in the microstructures with increasing reduction ratio below T_{NR} . Also, acicular ferrite (AF) formations replace pearlite in the microstructure with increasing reduction ratio below T_{NR} while volume fraction of pearlite in the microstructure decreases.

SEM investigations were carried out on Charpy specimens tested at -80 °C to confirm the nature of fracture depending on the reduction ratio. SEM images of Charpy test specimens tested at -80 °C are given in Figures 9 and 10 as examples. Totally dimple area which represents ductile fracture is observed at a reduction ratio of 72.8 % and this is clearly visible in Figure 9. Fracture mode is cleavage fracture which is transgranular brittle fracture at a reduction ratio of 60.7 % as can be seen in Figure 10.

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Reduction ratio	
below TNR (%)	
72.8	
70.3	
67.5	
64.2	
60.7	

Figure 7. Fracture surfaces of Charpy V-notch impact test samples associated with reduction ratio below T_{NR} (Test temperature -80 °C).

Reduction ratio below T _{NR} %	X500	X1000
72.8		
70.3		
67.5		
64.2		
60.7		

Figure 8. Microstructures associated with reduction ratio below T_{NR} .



Figure 9. SEM image of Charpy test specimen tested at -80 °C (reduction ratio: 72.8 %).



Figure 10. SEM image of Charpy test specimen tested at -80 °C (reduction ratio: 60.7 %).

High dislocation density is generated by the rolling reductions below T_{NR} and some strain energy retains in the austenite, this energy is the mechanical driving force for grain refinement and nucleation of acicular ferrite [24, 25, 26]. It was shown that the rolling deformation prior to the transformation into the acicular ferrite is more effective in increasing the amount of the acicular ferrite phase than chemical alloying and increasing the cooling rate [27]. The dislocations by the rolling reduction do not survive in every case for the transformation into acicular ferrite but the rolling operation induces a finer grained and dislocated "pancaked" austenite before transformation that will provide a high nucleation rate of acicular ferrite during the transformation [3, 27]. Ferrite grain size effects on the yield strength rather than the tensile strength through the Hall-Petch relationship [28, 29]. All of these can hinder the commencement of plastic deformation and cause an increase in the yield strength in the steel.

4. CONCLUSIONS

According to the results achieved in the trials below calculated T_{NR} temperature, it appears that yield strength significantly increases, tensile strength slightly changes, and elongation decreases in some degree. DWTT and impact toughness increases with increasing reduction ratio. Metallographic examinations show that final grain size decreases with increasing deformation ratio, and thereby confirm that fine grain sized microstructure increases toughness and strength together. Based on the trend of the experimental results of this study, it is expected further improvements on the mechanical properties when the reduction ratio increased beyond to max. reduction ratio (72.8 %) applied in this study.

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