MATERIAL DESIGN OF HIGH STRENGTH/HEAVY GAUGE LINEPIPES FOR SOUR SERVICE

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Abstract

Linepipe material for sour gas service primarily needs to have crack resistant properties. However, applications of sour linepipes are expanding toward deep water or cold regions, which require higher toughness and/or heavier wall thickness as well as higher strength. Steel plates for high strength linepipes, Grade X60 and higher, are produced by controlled rolling subsequently followed by accelerated cooling. Mechanical properties of linepipe steels are strongly affected especially by the plate rolling parameters. Higher cooling rates in the accelerated cooling process after rolling give tremendous benefit for obtaining higher strength and superior toughness even for thicker plates. This paper firstly summarizes the design concepts for controlling crack resistance and mechanical properties of high strength linepipe steels for sour gas service. Optimum conditions of controlled rolling and accelerated cooling that balance crack resistant properties and toughness were investigated.

In order to fulfill the need for higher strength material, Grade X70 linepipe for NACE sour environments was developed by advanced plate manufacturing technology. An on-line heat-treatment process was applied after accelerated cooling, for precipitation hardening by fine carbides. Homogeneous microstructures without martensite-austenite constituents (MA) were also obtained through the thickness of the plate by this new process. It was proven that precipitation hardened ferrite-bainite steels have extremely high resistance against HIC for Grade X70. Mechanical properties and microstructural characteristics of this newly developed steel are introduced in this paper.

Introduction

Linepipe material for sour gas service primarily needs to have crack resistant properties. However, applications of sour service linepipes are expanding toward deep water or cold remote regions, which require higher toughness and/or heavier wall thickness as well as higher strength. In order to improve crack resistance properties, many kinds of treatment in the steelmaking and plate rolling processes are usually applied for sour linepipe steels. One of the basic treatments is to prevent initiation of HIC by controlling inclusions, such as MnS, in the steels. Therefore, lower levels of sulfur content and Ca treatment, which prevents the formation of elongated MnS by forming spherical CaS, need to be applied [1,2]. Another basic treatment is reducing centerline segregation, because this segregated area shows increased hardness and HIC mainly occurs in this area. For this reason, contents of C, P, Mn and other alloying elements that cause centerline segregation need to be limited. Conventional linepipe steels for sour service are produced by applying the above treatments. For the steels of Grade X60 and higher, the accelerated cooling process after controlled rolling is applied for obtaining high strength with lean steel compositions. Plate rolling conditions for sour linepipe steels also need to be controlled [3,4]. However, it becomes difficult to fulfill recent stringent requirements for sour linepipes, such as heavy wall thickness and higher toughness, even when applying the above measures.

Recent advances in plate rolling techniques enable one to improve the mechanical properties of steels. Higher cooling rates in the accelerated cooling process after rolling give tremendous benefit for obtaining higher strength and superior toughness even for thicker plates. This paper focuses on the effect of plate rolling conditions on sour resistant properties and mechanical properties of high strength sour linepipe steels. The effects of alloying elements and microstructure on the sour resistance of steels produced by recent plate manufacturing techniques are also summarized.

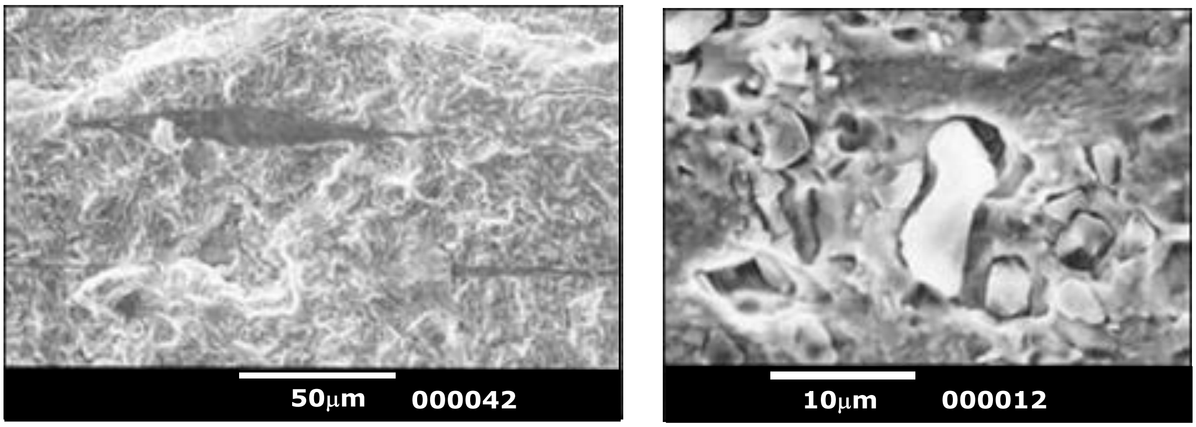
Although there is a strong need for total cost reduction in pipeline systems by using higher strength linepipes, application of sour resistant linepipe is mainly limited up to Grade X65, except for trial production of Grade X70 sour linepipes [5-7], at this time. Fine low carbon bainite is an excellent microstructure for balancing high strength and high toughness. However, there is a limitation for balancing higher strength and crack resistant properties, because susceptibility to cracking increases with increasing strength of the steels. Richer steel compositions for the higher grade steel may enhance the formation of harder second phases, such as martensite-austenite constituent (MA). HIC propagates easily along such second phases and susceptibility to HIC increases [3]. In order to develop sour resistant linepipe above Grade X65, homogeneous microstructural control and preventing the formation of MA by leaner chemistry are important.

An advanced TMCP (Thermomechanical Controlled Processing) or “Heat-treatment On-line Process” (HOP™) is applied after accelerated cooling. In this process, high strength can be achieved by precipitation hardening of fine carbide, which is mainly NbC, using leaner chemistry, while conventional TMCP mainly utilizes transformation strengthening. Homogeneous microstructures without MA can be obtained through the thickness of the plate by the new process. In this paper, metallurgical features and mechanical and microstructural properties of the steel manufactured by this process are discussed. Mill trial production results of Grade X70 sour resistant linepipe are also introduced.

Effect of Microstructure on Crack Resistant Property

<u>Effect of Non-Metallic Inclusions</u>

Prevention of crack initiation is a primary issue for improving crack resistance in sour environments. MnS inclusions are the most harmful initiator of HIC. Figure 1(a) shows a typical fracture surface of HIC for a steel which contains 0.0018% S and without Ca addition.



(a) 0.0018%S steel (b) 0.0003%S-0.0027%Ca steel

Figure 1. Fracture surface of typical HIC.

Elongated MnS inclusions act as an initiator of HIC. Therefore, as a basic measure, sulfur contents must be reduced for the steels used in sour environments. Because of progress in steelmaking processes, sulfur contents can be reduced down to a quite low level, under 0.0008 wt.% (8 ppm). Ca treatment usually is applied in order to prevent formation of elongated MnS and to control the sulfide inclusion into a spherical shape. However, there is an optimum Ca content for crack resistance [1,8]. An example of the fracture surface of the steel with low sulfur content and Ca treatment is shown in Figure 1(b). Ca oxide inclusions are seen on the fracture surface. If the Ca content is relatively high compared with the S content, excess Ca may form oxides that can act as an initiator of HIC. Several parameters that represent effective Ca content have been introduced [1,5,9], and Ca contents need to be carefully controlled in a narrow range, for example, ratio $3 < [Ca]/[S] < 8$, where [Ca] and [S] are contents of Ca and S in weight percent.

<u>Effect of Centerline Segregation</u>

The number of non-metallic inclusions that act as crack initiators can be reduced by controlling S and Ca contents, though it is impossible to eliminate them completely. Also, for the steel plate produced from continuously cast slab, it is difficult to completely eliminate the centerline segregation that can cause centerline cracking in the HIC test. Centerline cracking can be prevented to a large degree by applying accelerated cooling after controlled cooling which produces a bainitic microstructure instead of a ferrite-pearlite microstructure [3,10]. However, centerline segregation causes different microstructures with higher hardness for higher strength steels.

Figure 2 shows a SEM micrograph of centerline cracking for a 0.05%C-1.5%Mn-0.0005%S-Ca treated steel. The microstructure of this steel was basically upper bainitic, though lower bainite or MA constituents were seen in the center region. These hardened microstructures were obtained by segregation of alloying elements, such as Mn and C and cracks propagated along this hardened region. Mn and C are the principal elements in centerline segregation, causing increased hardness in the mid-thickness region [11] and resulting in centerline cracking in HIC test.

Figure 3 shows the maximum hardness of the middle thickness and quarter thickness regions for the low S-Ca treated steels with different Mn contents. Microscopic hardness was measured by Vickers hardness testing with a load of 0.098 N. Almost the same hardness was obtained in the middle and quarter thickness region for the 1.2% Mn steel. On the other hand, the hardness of the mid-thickness region was quite high, about 300 HV, for the 1.5% Mn steel, while the quarter thickness hardness was under 250 HV. The critical maximum hardness for cracking in a 100% H_2S environment, HV_{crit}, is also indicated in Figure 3 [12], which suggests that the mid-thickness region of the 1.5%Mn steel is highly susceptible to HIC. On the other hand, centerline cracking can be prevented by reducing Mn content for the low C-low S-Ca treated steel.

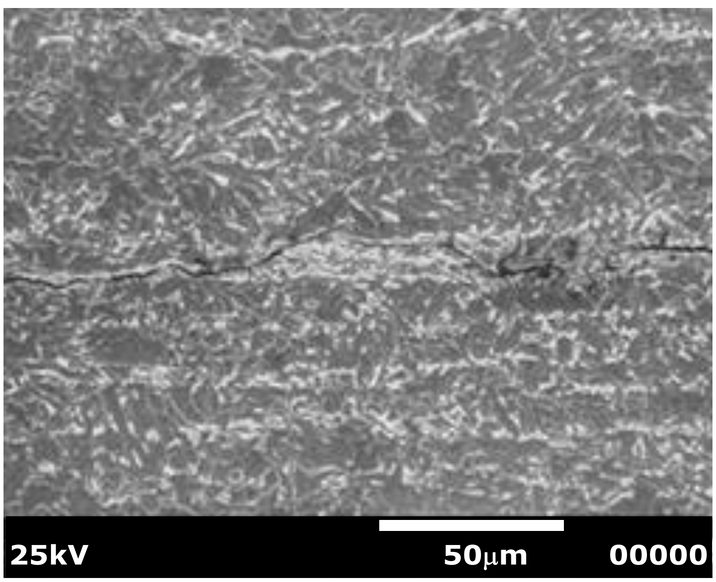


Figure 2. Cracking in the center segregation zone.

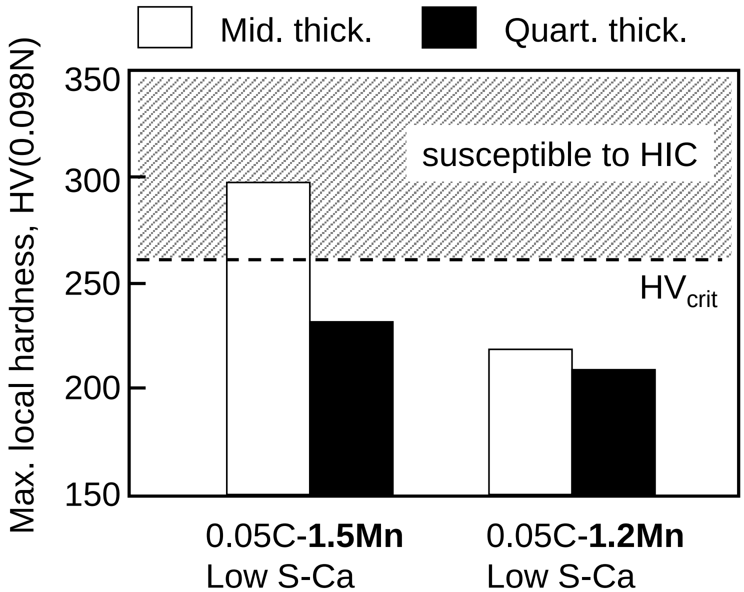


Figure 3. Effect of Mn content on maximum local zone hardness.

Effect of Accelerated Cooling Conditions

Recently, steel plates for sour linepipes were produced by the controlled rolling and accelerated cooling process. Crack resistance properties were improved by fine bainitic microstructures rather than ferrite-pearlite microstructures. However, the microstructure of the controlled rolled and accelerated cooled steel is quite sensitive to plate rolling and cooling conditions, with crack resistance changed accordingly. The effect of microstructures, obtained by different cooling conditions, on crack resistant properties of Grade X65 linepipes was therefore investigated.

The chemical composition of the steels employed is shown in Table I. As discussed previously, very low sulfur content and Ca treated steel with a limited content of C and Mn was used. Steel plates were produced by applying the controlled rolling and accelerated cooling process. In order to obtain different microstructures, the temperature at the start of accelerated cooling was changed, while rolling finishing temperature and other conditions were almost the same. Figure 4 shows the microstructures of X65 sour linepipes produced with different AC start temperatures for plate rolling. When the AC start temperature was near the transformation temperature, Ar_3, the microstructure obtained was almost fully bainite, as shown in Figure 4(a). On the other hand, ferrite-bainite or ferrite-pearlite microstructures were obtained when the AC start temperature was below Ar_3, Figure 4(b) and (c). Results of HIC tests for Grade X65 linepipes with different AC start temperatures are shown in Figure 5. CLR in the HIC test was higher for the linepipe with lower AC start temperature. If the AC start temperature is lower than Ar_3, polygonal ferrite forms and this causes concentration of alloying element in the austenite phase that will subsequently transform to bainite hardened by the richer alloying composition. Then, a mixed ferrite and bainite microstructure can be obtained after accelerated cooling. In this case, the difference in hardness of the ferrite and bainite phases is large and cracking can easily propagate along the phase boundaries. On the other hand, when the AC start temperature was near the Ar_3 or above, almost no cracking was found. Therefore, homogeneous bainitic microstructures are essential for improving crack resistance and the starting temperature for the accelerated cooling process should be carefully controlled for high strength sour linepipes.

Table I. Chemical Composition of Grade X65 Linepipe (wt.%)

C	Si	Mn	P	S	Nb	Ca	Others	Ceq	Pcm	Ar3
0.05	0.20	1.23	0.006	0.0006	0.04	0.0018	Cu, Ni, V, Ti	0.30	0.13	722

Ceq = C+Mn/6+(Cu+Ni)/15+(Cr+Mo+V)/5

Pcm = C+Si/30+Mn/20+Cu/20+Ni/60+Cr/20+Mo/15+V/10+5B

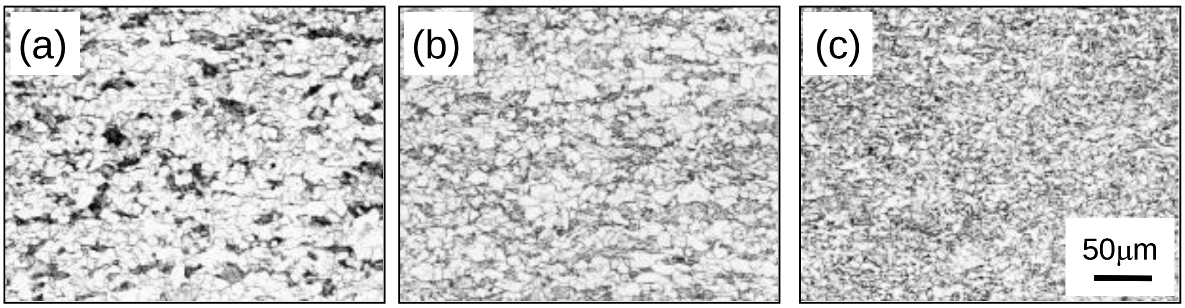


Figure 4. Microstructure of X65 linepipe accelerated cooled from different temperatures; (a) 760 °C, (b) 730 °C and (c) 690 °C.

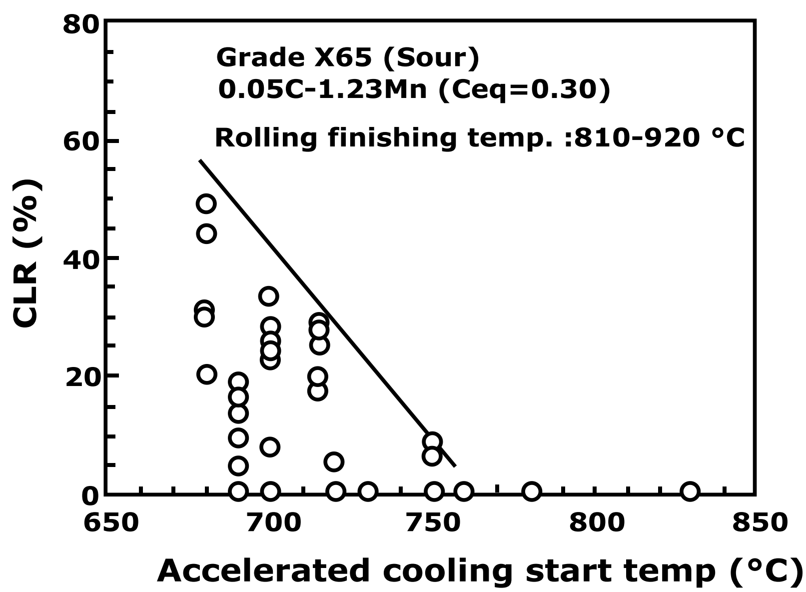


Figure 5. Relationship between accelerated cooling start temperature and CLR.

Improvement of Toughness by Accelerated Cooling Process

Effect of Cooling Rate on Microstructure

Grain refinement is the most effective measure for improving the toughness and strength balance of steels. The bainitic microstructure transformed from austenite by rapid cooling can be strongly affected by cooling rate.

The effects of cooling rate on microstructure and resulting strength and toughness of 0.05%C-1.3%Mn-Nb-V containing linepipe steels were investigated by using laboratory scale equipment. The cooling rate was varied from 1 °C/s to 50 °C/s by controlling the amount of water used for cooling. The thickness of the hot rolled plates was 20 mm.

Figure 6 shows the relationship between tensile strength and impact toughness of the accelerated cooled plates. Impact toughness was evaluated by the fracture surface appearance transition temperature (FATT) in the Charpy impact test. The plates produced with cooling rates higher than 20 °C/s show a better relationship between strength and toughness, these show a lower FATT for higher strength than the plates with a lower cooling rate of about 10 °C/s.

Figure 7 shows the effect of cooling rate on ferrite or bainite grain sizes. Grain size was measured by the intercept length of grains from SEM micrographs. Typical SEM micrographs of the plates produced with cooling rates of 10 °C/s and 30 °C/s are shown in Figure 8. Throughout the cooling rate range investigated, grain size becomes finer with increasing cooling rate, as has been reported elsewhere [13,14]. However, in the cooling rate range where uniform bainitic microstructures can be obtained, above about 20 °C/s, the effect of cooling rate becomes smaller compared with the lower cooling rate range.

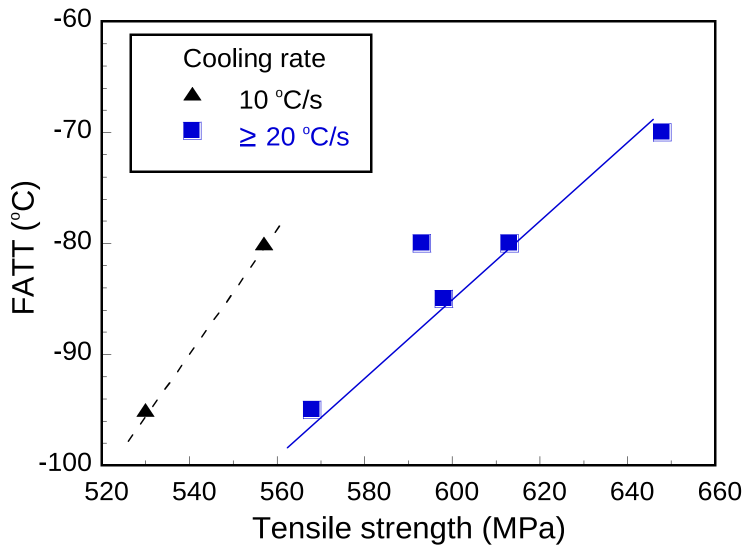


Figure 6. Relationship between tensile strength and Charpy impact toughness (FATT) of the steel plates accelerated cooled with different cooling rates.

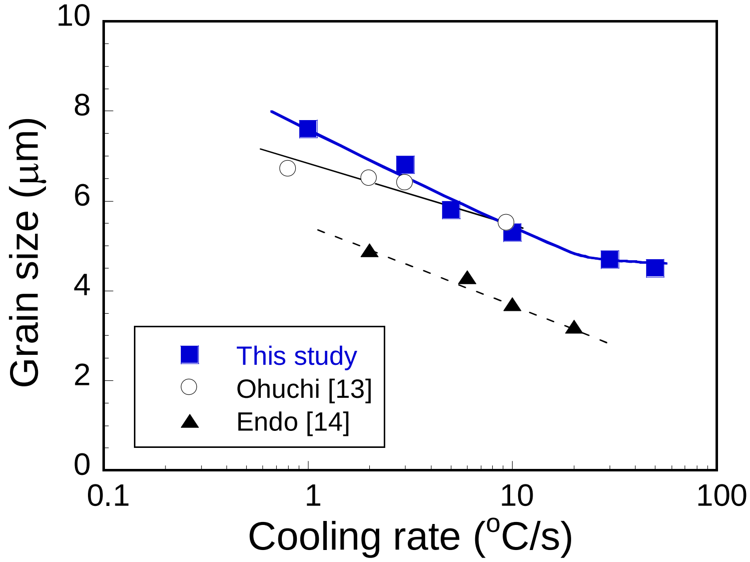
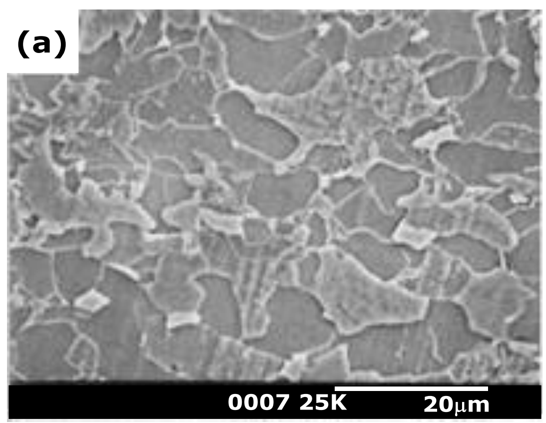


Figure 7. Effect of cooling rate on grain size of accelerated cooled plates.



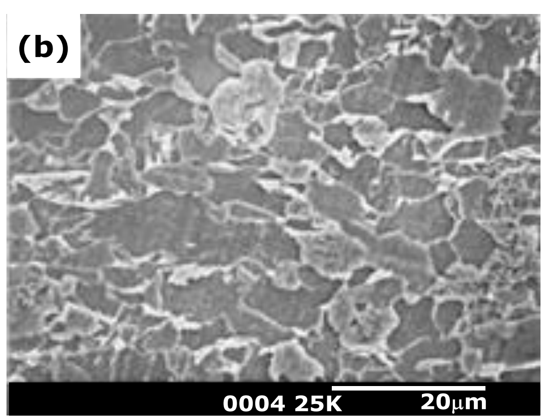


Figure 8. SEM micrographs of the plates accelerated cooled with cooling rates of;
(a) 10 °C/s and (b) 30 °C/s.

Rolling Conditions and Base Metal Toughness

Fine grained and homogeneous bainitic microstructures are obtained by applying accelerated cooling with higher cooling rates, as demonstrated in the previous section. Base metal toughness, which is usually evaluated by the drop weight tear testing (DWTT), can also be improved by higher cooling rates.

Figure 9 shows DWTT shear area transition curves for Grade X65 sour linepipes with wall thicknesses of 22.6 and 25.2 mm with different cooling rates. Steels with leaner chemistry were used for the pipe produced with higher cooling rates so that the same strength was obtained for both pipes. The linepipe steel with the higher cooling rate shows a lower transition temperature.

The toughness of linepipe steels is also strongly affected by rolling conditions. Figure 10 shows the effect of percent reduction during controlled rolling on DWTT shear area transition temperature (85%SATT). The transition temperature decreases with increasing reduction in controlled rolling. This tendency usually causes difficulty in obtaining higher toughness for heavy wall pipes when a conventional accelerated cooling rate is applied. However, base metal toughness can be improved by increasing the cooling rate in the accelerated cooling process and superior toughness can be achieved even for heavy wall pipes, such as 52 mm thick pipes as shown in Figure 10.

The effect of finish-rolling temperature, FRT, on base metal toughness was investigated in high strength sour linepipes that were produced from plates with different rolling conditions. Figure 11 shows the relationship between 85% shear area transition temperature and FRT. Base metal toughness can be improved by using a lower FRT. However, lower FRT leads to lower start temperatures for accelerated cooling. This results in deterioration of crack resistance in the HIC test, as indicated in Figure 5. Therefore, FRT needs to be carefully chosen for balancing base metal toughness and HIC crack resistance.

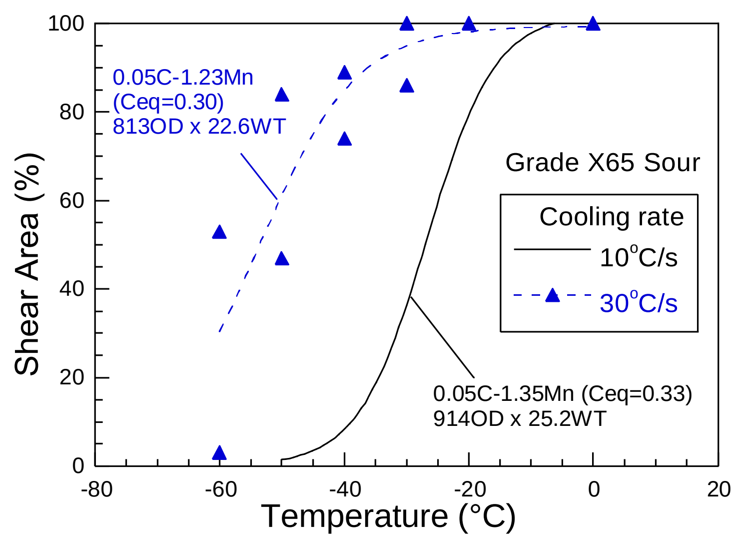


Figure 9. DWTT shear area transition curves of Grade X65 linepipes produced with different cooling rates in accelerated cooling process.

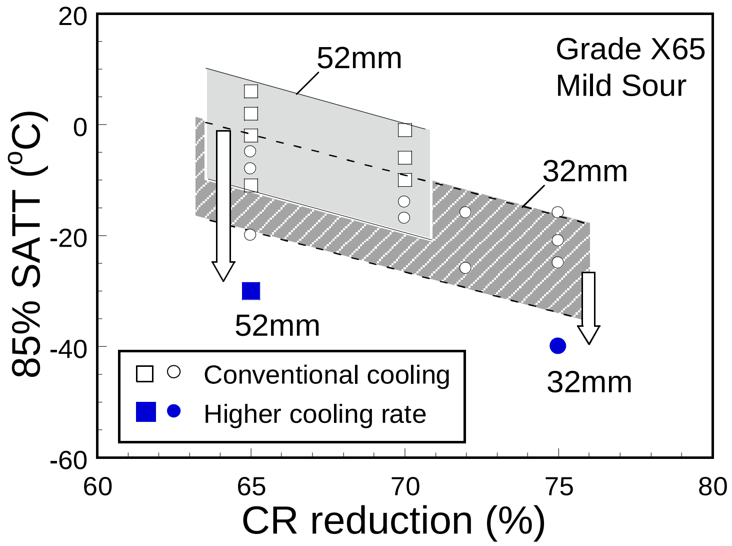


Figure 10. Relationship between DWTT shear area transition temperature and reduction in controlled rolling.

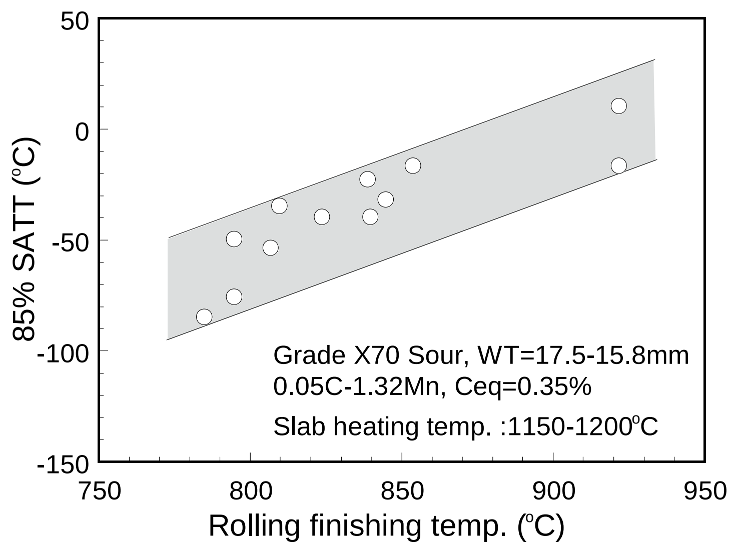


Figure 11. Relationship between DWTT shear area transition temperature and finish-rolling temperature.

Plate Manufacturing Technology for Sour Linepipe by Heat-treatment On-line Process (HOP)

Concept of New TMCP Process Applying On-line Heat-treatment

In order to produce high strength and high performance linepipe steels, the heat-treatment on-line process (HOP) was developed [15]. The combination of accelerated cooling and subsequent heating enables novel metallurgical control that cannot be achieved by the conventional TMCP.

One example of the temperature profile, when steel plate is manufactured by applying HOP, is shown in Figure 12, together with the conventional TMCP. This process is described as follows.

After controlled rolling is finished, steel plate is accelerated cooled from the temperature above the ferrite transformation start temperature (Ar_3) down to the middle of the temperature range of bainite transformation and immediately after accelerated cooling, a reheating treatment is applied. Metallurgical behaviors of the steel by this process are:

- Bainite transformation during accelerated cooling with higher cooling rate and the presence of untransformed austenite at the cooling stop temperature, taking advantage of the incomplete transformation phenomenon of bainite;
- During the reheating after accelerated cooling, ferrite transformation from untransformed austenite occurs. At the same time, precipitation of nanometer-sized carbides occurs at the austenite/ferrite interface;
- Microstructure is homogenized by tempering, and fine carbides are also formed in the bainite phase during the heat-treatment.

The purpose of this new process is to utilize precipitation strengthening that cannot be obtained in conventional TMCP but still with high productivity, which means without applying off-line tempering or slow cooling.

Characteristics of the steel plate produced by the on-line heat-treatment process are listed as follows: (i) Homogeneous hardness distribution in the through thickness direction, (ii) Small scatter of mechanical properties in the plate, (iii) Precipitation strengthening by fine carbides, (iv) Reduction of MA (martensite-austenite constituent). These metallurgical features are very suitable for high strength steels for sour resistance. Details of the metallurgical and mechanical characteristics are to be explained in the next section.

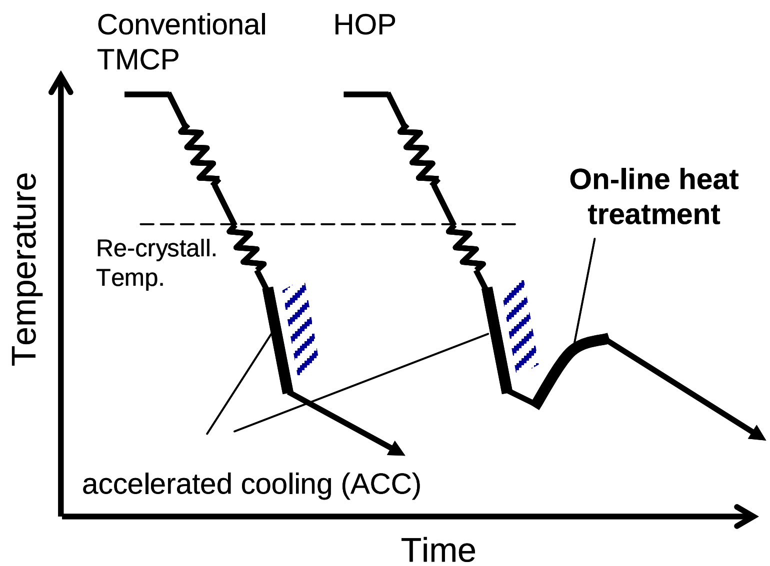


Figure 12. Schematic temperature profiles in plate production process.

<u>Mechanical and Metallurgical Properties of the Steel Produced by the On-line Heat-treatment Process</u>

A 0.05%C-1.25%Mn-0.1%Mo-0.04%Nb-0.045%V steel ingot was prepared in a laboratory furnace. Then, plate rolling was conducted on laboratory equipment including rolling, water cooling and heating devices. Plates were produced by applying the on-line heat-treatment process and the conventional TMCP.

In the laboratory rolling, accelerated cooling (ACC) was applied from 820 °C to 500 °C. In the conventional TMCP, the plate was cooled down to room temperature by air-cooling after ACC. On the other hand, in the HOP process heating was applied after accelerated cooling. The re-heating temperature was 650 °C. After the laboratory plate manufacture, round bar tensile specimens were taken from the mid-thickness of the plate. Samples for microstructural observation were polished and etched in 3% Nital; two-stage electrical etching was applied. The microstructures of the steel were observed by optical microscopy and scanning electron microscopy (SEM). SEM examination was conducted paying attention to the martensite-austenite constituent (MA). The morphology of precipitates was also investigated by transmission electron microscopy (TEM). Thin film samples were taken from the middle thickness portion of the plates. Chemical compositions of precipitates were analyzed by energy dispersive X-ray spectrometry (EDS).

Optical micrographs and SEM micrographs of the steels etched in 3% Nital produced by conventional TMCP and HOP are shown in Figures 13 and 14, respectively.

The steel submitted to conventional TMCP consisted of bainitic ferrite and a second phase, such as cementite or MA and the second phase was observed along the grain boundaries. On the other hand, bainitic ferrite, polygonal ferrite and second phases were observed in the steel produced by the HOP process. A second phase was observed along grain boundaries similar to conventional

TMCP steel. However, the second phase seen in the HOP process steel was finer than that in conventional TMCP steel. In order to distinguish the cementite and MA phases, two-stage electrical etching was applied for both steels.

In Figure 15, only MA is seen as the second phase since the cementite was dissolved by electrical etching. MA was not observed in the steel produced by the HOP process, this means that the second phase observed along the grain boundary in Figure 14 was cementite. On the other hand, MA was observed in the conventional TMCP steel. It is considered that the second phase in the conventional TMCP steel consisted of cementite and MA.

Tensile test results of these steels are shown in Table II. Although the chemical compositions are the same for both steels, tensile strength (TS) and yield strength (YS) of the HOP steel were higher than those of the conventional TMCP steel by about 50 MPa and 80 MPa, respectively. The yield to tensile ratio (YR) was higher for the steel produced by HOP treatment.

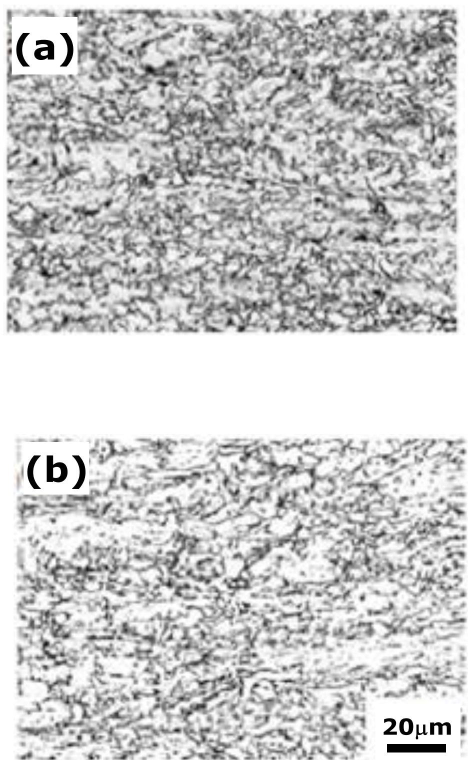


Figure 13. Optical micrographs of the steels etched in 3%Nital;
(a) HOP process, (b) conventional TMCP.

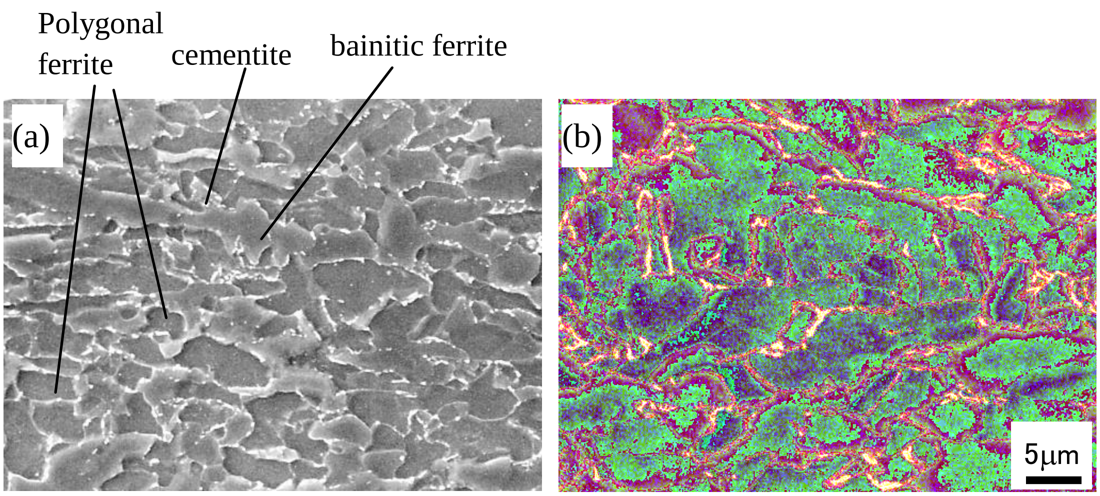


Figure 14. SEM micrographs of the steels etched in 3% Nital;
(a) HOP process, (b) conventional TMCP.

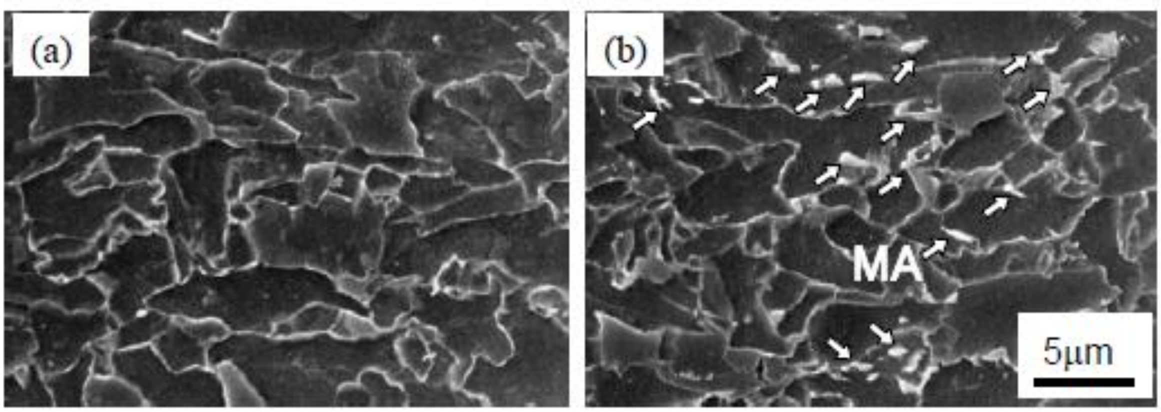


Figure 15. SEM micrographs of the steels etched by two-stage electrolytic etching;
(a) HOP process, (b) conventional TMCP.

TEM micrographs of the steels produced by HOP and conventional TMCP are shown in Figure 16. Large amount of carbides, which were 5 nm or less in diameter, were formed in the steel produced by the HOP process. Two kinds of precipitate morphology, random precipitation and row precipitation, were observed. EDS spectra of the precipitates observed in the steel by the new HOP process are shown in Figure 17. It is considered that the fine precipitates are complex carbides that consist of Nb, Ti, V and Mo. On the other hand, in the conventional TMCP steel, fine precipitates were hardly formed and large particles of (Nb, Ti)(C, N), which were not dissolved during slab reheating, were observed.

Table II. Tensile Properties of the Steels Studied

Process	**YS (MPa)**	**TS (MPa)**	**YR (%)**
HOP	608	660	92
Conventional TMCP	525	611	86

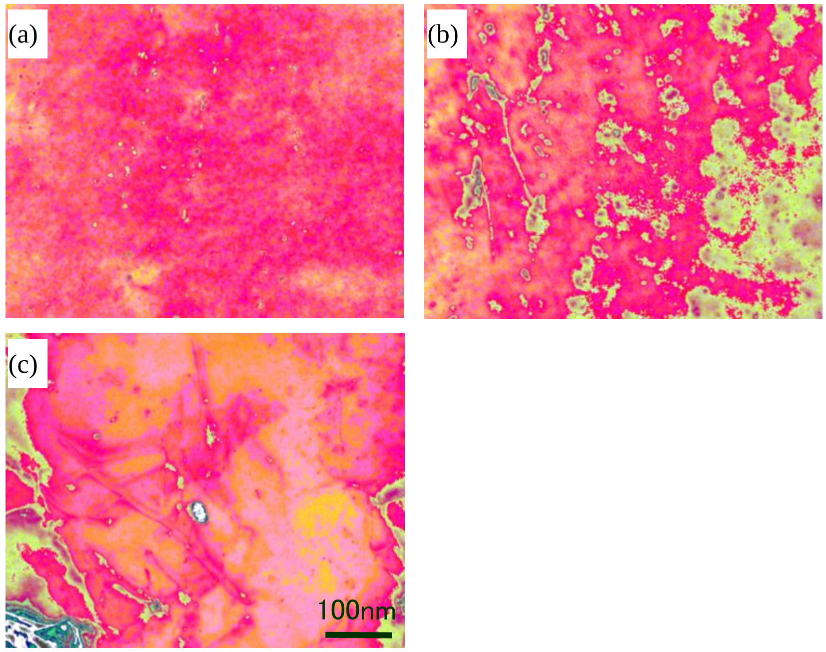


Figure 16. TEM micrographs of the steels; (a) HOP, random precipitates, (b) HOP, precipitate rows, (c) conventional TMCP, relatively large precipitates.

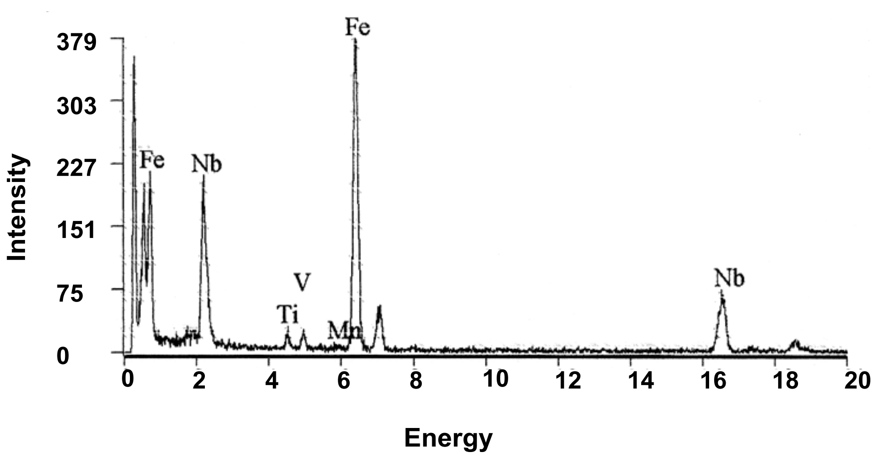


Figure 17. EDS spectrum of the precipitates.

Mill Trial Production Results of Grade X70 Sour Resistant Linepipe

Trial production of X70 linepipe for heavy sour service was carried out. The chemical composition is shown in Table III. Steel plate of 19 mm thickness was produced applying the heat-treatment on-line process (HOP). Figure 18 shows the hardness distribution along the plate width of the X70 grade sour linepipe steel, showing the hardness of the surface and quarter thickness positions. It is shown that hardness in the surface and quarter positions are almost the same, moreover, a very flat hardness distribution across the plate width was obtained. This is because of the homogeneous microstructure, which is produced by the homogeneous temperature distribution in the HOP heating process. Accordingly, scatter of the material properties such as strength and toughness can be reduced in the plate.

The microstructure of the trial X70 steel observed by SEM is shown in Figure 19. Microstructures consisted of bainitic ferrite, polygonal ferrite and cementite, while MA was not observed. TEM observation revealed precipitation of a large amount of nanometer-sized carbides, as shown in Figure 20. It is confirmed that precipitation strengthening can be sufficiently obtained even in mill production by applying HOP. Then, pipes with 914.4 mm diameter were manufactured by the UOE process. Mechanical properties and HIC resistance of the trial X70 linepipes are shown in Table IV. The trial pipes had sufficient strength for X70 Grade and showed excellent resistance to HIC in NACE sour environment.

Table III. Chemical Composition of Trial X70 Sour Linepipe (wt.%)

Grade	C	Si	Mn	P	S	Others	Pcm
X70	0.05	0.28	1.13	0.014	0.0005	Mo, Ni, Cr, Nb, Ca	0.14

Pcm = C+Si/30+Mn/20+Cu/20+Ni/60+Cr/20+Mo/15+V/10+5B

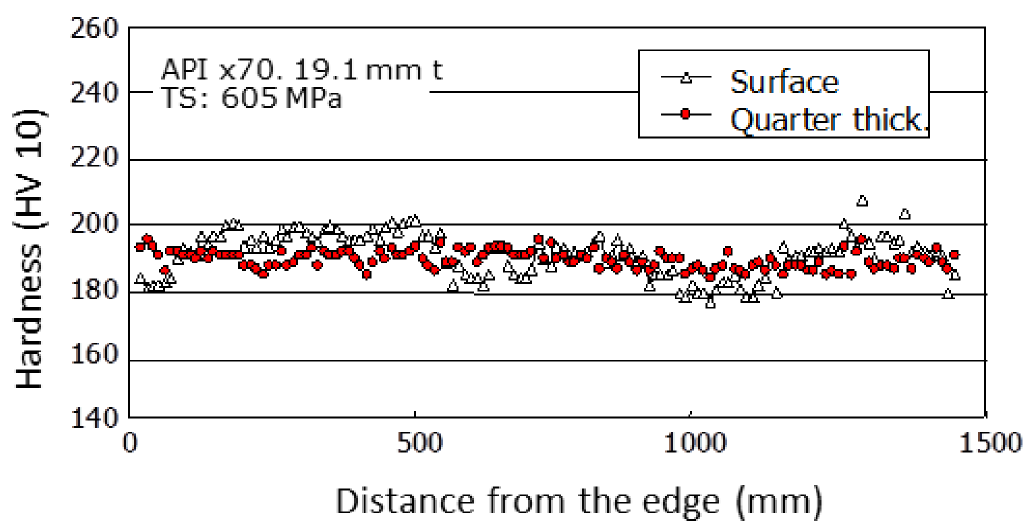


Figure 18. Hardness distributions across the width of X70 sour linepipe steel plate.

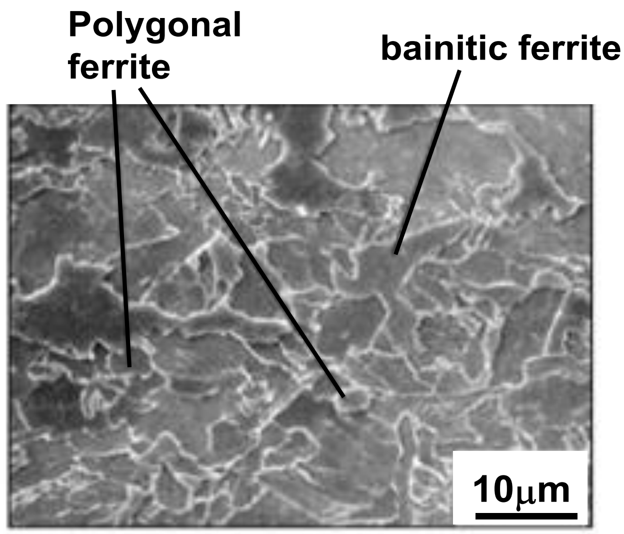


Figure 19. SEM micrograph of X70 sour linepipe produced by the HOP process.

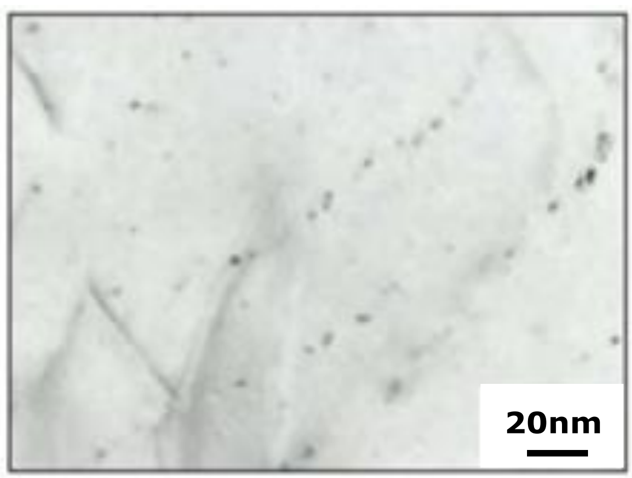


Figure 20. TEM micrograph of X70 sour linepipe produced by the HOP process.

Table IV. Trial Production Results of X70 Sour Linepipes by Applying HOP

Grade	**Pipe No.**	**Tensile Properties** *1				**Impact properties**	**DWTT**	**HIC** *2	
		YS (MPa)	**TS (MPa)**	**EL (%)**	**Y/T (%)**	**vE (J) at –10 °C**	**SA% at 0 °C**	**CLR (%)**	
								90°	**180°**
X70	1	531	613	23	87	373	100	0, 0, 0	0, 0, 0
	2	523	600	22	87	343	100	0, 0, 0	0, 0, 0

* 1 ISO rectangular
* 2 NACE TM0284-solution A

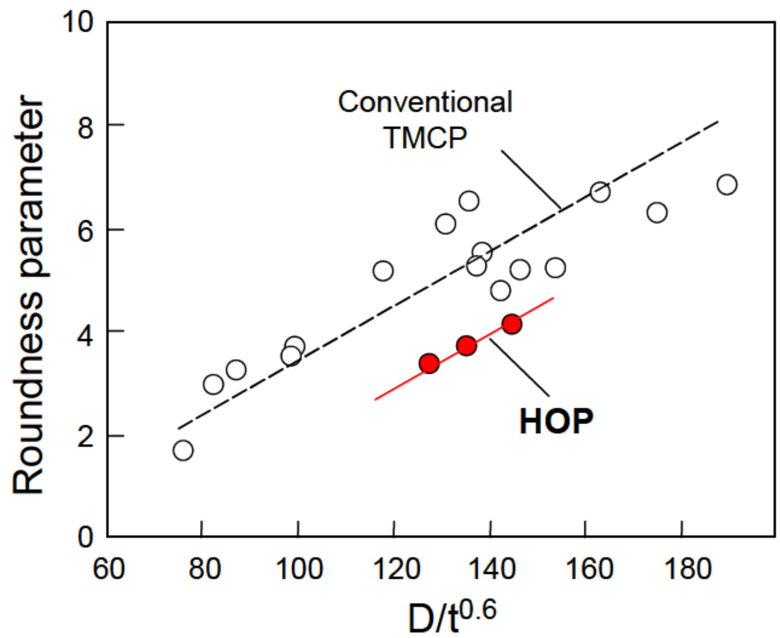


Figure 21. Relationship between roundness and $D/t^{0.6}$ for linepipes with different manufacturing processes.

The HOP process has been applied to mass production of Grade X60 to X70 sour linepipes. Figure 21 shows the relationship between pipe roundness parameter which is obtained by statistical out-of-roundness data from different pipeline projects, a lower value of the roundness parameter means better shape, ie. lower out-of-roundness and pipe dimension. Roundness of the pipe deteriorates as $D/t^{0.6}$ increases, however, roundness of the pipe improved by applying HOP. Homogeneous material properties in the plate thickness direction, as well as the plate width direction, as shown in Figure 18, are achieved by the HOP process, giving great benefit in uniformity of pipe forming, resulting in excellent pipe roundness.

Conclusions

The effect of steel composition and plate rolling conditions on HIC crack resistance and mechanical properties of recent high strength sour linepipes were investigated. The key points for improving crack resistance and toughness of heavy gauge steels are; (i) reducing sulfur content with Ca addition, (ii) balancing DWTT toughness and HIC resistance properties by lowering the finish-rolling temperature while preventing ferrite formation before the start of accelerated cooling, (iii) a higher cooling rate during the accelerated cooling process for achieving a fine bainitic microstructure through the thickness.

Grade X70 linepipes for sour environments with superior toughness were produced by applying higher cooling rates. The on-line heat-treatment process (HOP) was applied for producing Grade X70 linepipe for sour service. It was shown that nanometer-sized fine precipitates were formed while formation of MA constituent was prevented by the HOP™ process and uniform material properties through thickness and along the plate width were achieved. Trial production of grade X70 linepipe for severe sour service was carried out. The trial pipes had sufficient strength, toughness and excellent resistance to HIC in NACE sour environment.

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