EFFECTS OF MICROALLOYING AND HOT ROLLING PARAMETERS ON TOUGHNESS AND YIELD STRENGTH OF API X80 GRADE STEEL STRIPS

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Abstract

Recently API X80 grade has been increasingly used for UOE pipes and, furthermore, API X100 and X120 grades are being developed. However, the usage of hot rolled API X80 grade for spiral-welded pipes has been limited. This is partly due to metallurgical barriers which are the anisotropy of mechanical properties and the strength drop after pipe forming. Manufacturing conditions and metallurgical parameters causing these barriers have been reviewed. The effect of both size and distribution of MA constituent on the Charpy impact upper shelf energy has been investigated. In addition, the development of API X80 hot rolled strip and commercial production results from a 12,000 tonne API X80 grade order for a linepipe project have been summarised.

Introduction

Higher strength grades of hot rolled steel strip are now being used for pipeline construction to obtain high gas transport efficiency. The use of high strength steel strip allows higher operating pressures and gas transmission rates, Figure 1 [1,2]. These higher grades require both increased strength and enhanced toughness by adding more microalloying elements without deteriorating weldability, to ensure the safety and economy of the pipeline. It is therefore important to optimise chemical composition and processing parameters to satisfy the requirements of the entire production route, Figure 2.







Figure 2. Production route of high strength steels for linepipe application.

To manufacture higher strength steels it is necessary to add more alloys, which potentially results in inferior weldability and toughness. An increased operating pressure in a pipeline system requires higher toughness values to achieve crack arrest when running ductile fracture occurs. This results in the API/ISO Standards for higher strength grades requiring higher values of Charpy energy. Metallurgically, unfortunately this requirement is contradictory to the nature of steel since higher strength usually results in lower toughness as shown in Figure 3. HSLA steels have firstly overcome this paradox and therefore have been widely used in high strength steel applications. Although no revolutionary development has been pushing the technological limit of steel manufacturing processes, as seen in the IT industry, deeper scientific understanding of metallurgical phenomena has become possible. It sould be noted that technology transfer from laboratory to mill is considered to be another technical problem in steel research, which is mainly due to the size difference. Small scale laboratory output cannot be directly implemented in the commercial production mill. Several thousand kilometer long pipelines made with higher strength grade steels have been successfully constructed in the world by using the mill as laboratory-like facilities, i.e. intensive metallurgical analysis and investigation on the mill, not using samples melted and processed in the lab. Practically speaking, 20 tonnes of slabs rolled as plates or strip with cooling water and oxide scale on the surface cannot be rolled in the same condition from head to tail or from surface to centre line of the stock. Statistical controls in the material processing that are based on and stick to metallurgical principles should be introduced in the mill in order to guarantee the target mechanical properties.

In the manufacturing of UOE pipes, an expansion process increasing yield strengths by straining of the pipe body is beneficial to the final dimension and also the mechanical properties. Meanwhile, it is more challenging to obtain the desired strength and toughness in the pipes due to anisotropy of mechanical properties originating from the crystallographic texture in hot rolled strip [3]. This is because more reduction (one-directional hot rolling) is applied in the strip manufacturing process, using a tandem hot rolling facility, compared with plate rolling. The mechanical anisotropy, illustrated in Figure 4, shows the diagonal direction which is coincident with the hoop direction in the pipe body having poor toughness and lower yield strength. A crystallographic texture is traditionally believed to be responsible for this phenomenon but the temperature dependency of Charpy energy and the relationship between these properties before and after pipe making have not been clearly explained yet.



(self-arrestability)

* Strength vs. Toughness (CVN)

Grades	CVN (API/ISO) J	CVN [J, -15 °C]	YS [MPa]
API X70	84 / 27	500	490
API X80	133 / 27	150-370	560

Figure 3. A comparison of toughness requirements for API X70 and X80.



Figure 4. Anisotropy of yield strength and Charpy energy of strip.

One important empirical result indicates that the microstructure, including effective grain size and the distribution of precipitates, plays a key role in the mechanical anisotropy of properties. For example, the occurrence of cleavage observed on the fracture surface of a Charpy specimen has a strong dependency on hard phases contained within the microstructure. The resulting lower energy values are associated with brittle behaviour, while ductile fracture occurs by microvoid coalescence. The effective grain size and the second phase distribution, for acicular ferritic/bainitic microstructures, frequently shown in high grade API steels, cannot be well defined, which leads to the difficulty of understanding the basics of toughness behaviour. It is noted in Figure 4 that the anisotropy of toughness is weakened as temperature increases and at extremely low temperatures below -100 °C no toughness anisotropy has been observed.

The addition of microalloying elements, such as Nb, Ti, Mo and V which precipitate in austenite (γ) as carbides, nitrides, or carbonitrides are essential additions for API grade steels. The controlled rolling of microalloyed steels is performed to optimise metallurgical phenomena such as Nb dissolution in the reheating furnace, austenite conditioning, pancaking during finishing and finally phase transformation on the runout table. Even nano-precipitation at low temperature (below 600 °C) has to be considered after coiling. Effects of alloying elements can be summarised as follows [4]. Mo enhances strength and toughness by readily promoting the formation of low-temperature transformation phases and grain refinement. Cr enhances strength as it promotes low-temperature transformation phases but deteriorates low-temperature toughness due to coarse grain size. No is effective for enhancing both strength and toughness as it efficiently refines grains and raises the volume fraction of secondary phases. Considering only the effect of primary alloy addition, it can be concluded that high strength attained with increased alloying decreases toughness, Figure 5. Although it is known that a ductile ferrite matrix will give a Charpy energy level of 500 J, the microstructural parameters which affect DBTT (Ductile Brittle Tansition Temperature) and Charpy energy plateau (CVN), respectively, are not clearly, quantitively understood yet.



• High strength needs more alloying ⇒ decrease in toughness

Figure 5. General alloying effect on the strength and toughness.

A schematic thermal history and the major metallurgical events occuring during strip manufacturing processes are shown in Figure 6. It is notable that the temperature is the most crucial property controlling factor. Alloys added to the steels, acting as a promoters/suppressors of phase transformation and precipitation, can be utilised as originally intended to determine the final microstructure, by mill-line temperature control. One of the difficult metallurgical issues is the kinetics of Nb(C,N) precipitation in deformed γ in roughing stands in Nb microalloyed steels. It is complicated to understand because several parameters are acting at the same time, such as alloying elements, temperature and deformation. It is known that the strain-induced precipitation of NbC in γ is delayed in Nb-Ti microalloyed steels compared with that in Nb-microalloyed steels because of incorporation of some of the Nb atoms in the undissolved (Ti,Nb)(C,N) particles during the solution treatment, Figure 7.



Figure 6. An overall thermal history during strip manufacturing processes.



Figure 7. Dissolution behaviour of precipitates during reheating (a), and resulting AGS (Austenite Grain Size) (b).

It has been reported that (Ti,Nb)N precipitates are cuboidal, having a size of 45 to 300 nm, and (Ti,Nb,V)C or (Nb,V)C precipitates are spherical or irregular shaped, being 20 to 45 nm in size. Nb-rich precipitates of 30 to 150 nm tend to nucleate on Ti-rich precipitates or on grain boundaries, and fine NbC of less than 5 nm precipitate at low temperatures, as for example those encountered during coiling.

As explained before, optimisation of microstructure and precipitation behaviour is necessary to improve the strength and toughness of high grade pipeline steels. Appropriate design of the chemical composition together with thermo-mechanical controlled processing (TMCP) contribute to achieving effective microstructures and textures, and through them the desired mechanical properties. In this paper some key metallurgical points are summarised and discussed that were obtained during the development and commercialisation of API X80 hot rolled strip for spiral-welded pipes, at POSCO, mainly considering the effects of hot rolling conditions on mechanical properties.

Hot Rolling Condition

With the pipeline requirement, as seen in Table I, higher Charpy energies for securing safe transport of 1,300 psig max. (pounds per square inch gauge) gas had to be obtained. Thus, based on experience, the microstructure of API X80 steel was designed to have an acicular ferrite matrix, carbide-free cells of bainite with an isle-like dispersed martensite/austenite (MA) constituent. The addition of Nb, Mn, Ni, and Mo is effective for the formation of acicular ferrite to obtain high toughness as well as high strength. The carbon content is kept at about 0.07% or less. Chemical composition of the high grade linepipe steels were $\leq 0.07\%$ C – $\leq 1.85\%$ Mn – $\leq 0.085\%$ Nb < 0.025%Ti – $\leq 0.35\%$ Mo – $\leq 0.6\%$ Cr + Ni + V (wt%), and several heats were mill-trialled to determine the final chemistry and hot rolling conditions.

WT(mm)	Charpy E (J) @ -15 ^o C (Coil)	Remarks
10.85	240	-
	181	Each heat
14.45	130	-
	99	Each heat

Table I. Requirement of CVN for API X80 Grade Steels

Hot rolled strip with thicknesses of 10.85 and 14.45 mm were manufactured in oxygen converters and then cast to slabs with a final thickness of 250 mm. The slab reheating temperature was above 1200 °C in order to bring niobium carbides or nitrides into solution.

To determine slab reheating temperatures, Nb precipitation behaviour was analysed with respect to reheating temperatures, Figure 8, and also chemical composition because these precipitates control AGS (Austenite Grain Size) in the slab. Abnormally large grains in the slab can be a starting point of poor toughness in the final product because they are difficult to refine after the reheating process. As a result, reheating temperature should be high enough to secure the dissolution of the alloying element but not too high, in order to prevent abnormal grain growth.



Figure 8. Nb precipitates in a 0.05% Nb steel at reheating temperatures which control AGS (Austenite Grain Size) during reheating processes.



Figure 9. Through thickness grain refinement variation with the amount of rolling reduction and mean flow stress changes, showing non-recrystallisation in the roughing mill.

In Figure 9 the through thickness grain size changes, with the amount of reduction in the roughing mill, and mean flow stresses calculated using the roll force from the hot rolling mill are shown. These micrographs and the changes of mean flow stresses imply that reduction and temperature are keys to the control of microstructure, in conjunction with Nb precipitation. It is important because the final acicular ferritic or bainitic microstructure is strongly dependent on the accumulated strains in the non-recrystallised austenite phase (pancaked) in the roughing mill.

Figure 10 shows very fine (Nb,V)C precipitates which are believed to be formed after coiling, at lower temperatures. The table rows represent the sampling location corresponding to head (top row), middle (centre row) and tail (bottom row) of a strip. It can be seen that precipitation is stronger in the mid-section of a strip, because of slower cooling rates, compared to the outer or inner windings of the coiled strip.



- Homogeneous precipitation in ferrite region
- Round shape (Nb,V)C
- Size <3 nm

Precipitated

Alloys (Total)	Ti (0.018)	V (0.051)	Nb (0.052)
Precipitated	0.019	0.0029	0.031
	0.019	0.0120	0.046
	0.019	0.0026	0.029

*Nb exists, both as precipitates and as solid solution

Figure 10. Nb rich precipitates formed after coiling processes.

Mechanical Properties

Table II lists the mechanical properties of manufactured strips for a trial coil and 42" OD pipes, respectively, showing values higher than the API specification. The yield strength of the coiled strip is measured at 30 degrees to the rolling direction near the tail end, corresponding to the hoop direction of the pipes. Other mechanical properties such as Charpy and DWTT have also been measured along the 30 degree direction of the strip. Flattened tensile specimens were prepared from a coupon taken from ring samples. Yield and tensile strength in the transverse and longitudinal directions are always greater compared to that at the 30 degree to the rolling direction. This anisotropy of mechanical properties is undesirable, which is thought to be one of the difficulties of designing spiral welded pipelines.

WT	Coil (MPa), 30°		Pipe (MPa), Hoop direction	
(mm)	YS	TS	YS	TS
10.85	676	838	595 (Avg.) (599/587/597)	756 (Avg.) (761/747/758)
14.45	597	760	580 (Avg.) (580/587/574)	760 (Avg.) (760/758/761)

Table II. Mechanical Properties of Strip Coil and Pipes

Charpy Impact Energy

Charpy energy is of importance for designing pipelines to control fracture behaviour especially during high pressure gas transport. Charpy toughness values at different temperatures, as a function of specimen orientation, are displayed in Figure 11, showing a similar dependency as seen in tensile properties. The Charpy impact energies at 20~45 degrees to the rolling direction are decreased despite the low yield strength. This anisotropy originates from the major components of textures, which are {332}<113> and {113}<110> orientations, and the density of {001} cleavage planes in the Charpy specimen, which is highest at 30 degrees to the rolling direction compared with that of other orientations. Therefore, the highest susceptibility to cleavage fracture, i.e. increased ductile-brittle transition temperature, can be seen in the 30 degree direction [5].

Figure 12 shows photos of fractured Charpy specimens and fracture surfaces of longitudinal (L), transverse (C) and 30 degree (30°) samples tested at -80 °C. The shape of fractured specimens is an indication of the amount of plastic deformation experienced by the Charpy specimen when impacted by the striker of the impact machine. Lateral expansion or deformation on opposite side to the notch (striking face) is observed in L and C direction samples while no plastic deformation is observed in the 30 degree sample. It can be clearly seen in the SEM photos that cleavage fracture is dominant in the 30 degree sample.



Angle to R.D. (°)

Figure 11. Changes of Charpy impact energy with hammer striking direction and temperature.



Figure 12. Fracture surface showing lateral expansion by plastic deformation (L-Longitudinal & C-Transverse) and brittle fracture without plastic deformation (30 degree). Charpy impact test was performed at -80 °C.

The Charpy impact energies of high strength linepipe steels can be affected by microstructural factors, such as initial centreline segregation and abnormal grains found in slabs. The AP (Ammonium Persulphate) method has been used to reveal centerline segregation in the slabs, which then allowed adjustment of the soft reduction condition during the continuous casting process. Abnormal grains, on the other hand, survive and increase the effective thickness and length of the pancaked austenite grains, generated during rolling in the non-recrystallisation regime, and remain as a source of anisotropy and poor toughness. When measured, the effect can be seen in the range of length and thickness of the pancaked austenite grains, $3\sim35 \,\mu\text{m}$ and $2\sim10 \,\mu\text{m}$, respectively [6,7].

Effects of MA on Toughness

It is believed that the addition of Ni has a beneficial effect of improving toughness. This effect has been confirmed in this work, as shown in Figure 13. Ni additions up to 0.2% improve Charpy impact toughness however no big difference has been observed by increasing the Ni addition from 0.2 to 0.4%. To obtain a higher impact toughness all chemical elements should be balanced and optimised to minimise carbides and, more importantly, nitrides. The microalloy carbides and nitrides can be a source of crack initiation, resulting in poor toughness, so that the addition of Ti and V, for example, should be minimised. The effect of alloying elements, however, cannot fully explain the toughness of a steel composition. Hard phases spotted in the matrix result in poor toughness due to their lack of ductility. Small islands of martensite or retained austenite (MA constituent) can accelerate cracking and are frequently observed in an API X80 grade alloying system. Cracks can be initiated at and be propagated along the interface of MA and matrix as shown in Figure 14. The formation of MA is known to be strongly dependent on the amount of Mo and Cr added.



Figure 13. Effect of Ni addition on CVN.



Figure 14. Relationship between MA volume fraction and Charpy impact energies.

Drop Weight Tear Test (DWTT)

Figure 15 shows changes of shear area, SA (%), measured from DWT tests at various temperatures, using 16.9 mm thick API X80 grade produced in mill trials. As observed in Charpy impact tests, similar anisotropic properties of SA can be seen, that is the lowest value of SA occurs at 30 degrees to the rolling direction, Figure 15 (a). A general relationship between the degree of reduction in the non-recrystallisation zone and 85% Shear Appearance Transition Temperature (SATT) can be derived from mill trial results, Figure 15 (c).



Figure 15. Variation in DWTT Shear Area (%) with temperature and sampling direction; (a) fractured surfaces, (b) the general relationship between % reduction in the nonrecrystallisation zone and (c) 85% Shear Appearance Transition Temperature (SATT).

Commercial Production of API X80

The optimisation of chemical compositions and hot rolling conditions, by reviewing mill trials, has been done for commercial production. To achieve the high toughness level requirement of API X80, mill-line temperatures, such as reheating, roughing delivery and coiling temperatures, have to be finely tuned. Cooling water and rolling speed were adjusted to control roughing delivery temperature. Fine grained microstructures improve the ductile-brittle transition temperature, but do not appear to influence the upper shelf energy.

Figure 16 shows the Charpy energy distribution at -20 °C from commercial production of 12,000 metric tonnes of hot rolled strip. Produced coils with lower toughness than the requirement were rejected, however this concerned only a few coils. The microstructure of a sample coil is shown in this figure and reveals a bainitic microstructure.



Figure 16. Distribution of Charpy energy measured at -20 °C for commercially produced API X80 grade with a thickness of 10.85 mm and typical steel microstructure at different scales.

For the construction of the Southeast Supply Header pipeline, Figure 17, 27,000 pipes were manufactured and supplied from a pipe mill. In conclusion, 300 miles of API X80 grade 42" spiral pipeline has been successfully constructed.



Figure 17. South East Supply Header pipeline. (source: www.spectraenergy.com/Operations)

Summary

API X80 grade hot rolled steel strip with thicknesses of 10.85 and 11.45 mm has been developed. This API X80 grade satisfies the API requirements for commercial applications and has been successfully applied in a pipeline project. The DWTT shear area fraction at -20 °C was 100%, and the Charpy impact energies at -20 °C were more than 240 J in the strip. A combination of high strength and good toughness was obtained by controlling hot rolling parameters to obtain an optimised microstructure of acicular ferritic/bainitic matrix with a reduced MA content.

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