# ADOPTION OF COMMERCIAL PRODUCTION OF X65 PLATE STEEL FOR DEEP-WATER GAS PIPELINES AT AZOVSTAL IRON & STEEL WORKS AND WELSPUN PIPE COMPANY

Yu. I. Matrosov<sup>1</sup>, O. V. Nosochenco<sup>2</sup>, O. A. Bagmet<sup>2</sup>, P. Mittal<sup>3</sup>

<sup>1</sup>I.P. Bardin Central Iron & Steel Research Institute, Moscow, Russia;

<sup>2</sup>Azovstal Iron & Steel Works, Mariupol, Ukraine;

<sup>3</sup>Welspun Pipe Co, India.

Keywords: controlled rolling, steel for deepwater pipes, microalloying with niobium, chemical composition, and mechanical properties.

### Abstract

This paper describes the metallurgical approach used for the production of 160 miles of 36" OD X-65 linepipe in thicknesses up to 31 mm (1.25 inch) which was installed in several hundred feet of water in the Gulf of Mexico. The project was conceived and executed by GulfTerra (now Enterprise Products) and is named Cameron Highway Offshore Project. At the time of completion it represented the longest offshore pipeline in the Gulf of Mexico. A low carbon niobium microalloyed steel was produced and rolled at Azov Stal. Careful attention to continuous casting and rolling conditions resulted in plate and pipe that had uniform microstructure throughout the thickness and excellent notch toughness.

## Introduction

Azovstal Iron & Steel Works jointly with I.P. Bardin Research Institute and Welspun Pipe Co implemented commercial production of high-quality low-alloy plate steel for production of API 5L Grade X65 PSL2 pipes, intended for construction of deep-water gas pipeline by Gulf Terra (now Enterprise Products) in the Gulf of Mexico. This paper presents the research results for this large commercial lot of rolled steel plates (~70 thousand tons) and pipes manufactured as described herein for the offshore project.

The specification for the rolled steel plates, in sizes of  $24 \times 1826 \times 12375$ ,  $27 \times 1816 \times 12375$  and  $31 \times 1806 \times 12375$  mm, included the following requirements for mechanical properties:

- YS = 448 550 MPa;
- UTS = 530 645 MPa;
- $YS/UTS \le 0.88;$
- $El(2'') \ge 28\%;$
- $HV_{10} \le 230;$
- $A_{V-30}{}^{\circ}{}_{C} \ge 130 \text{ J}$  (average for three specimens), individual values  $\ge 100 \text{ J}$ , which is equivalent to KCV- $_{30}{}^{\circ}{}_{C} \ge 162 \text{ J/cm}^2$  and  $\ge 125 \text{ J/cm}^2$ , respectively;
- Shear area on Charpy V-notch specimens at  $-30^{\circ}C \ge 90$  %;
- Shear area on DWTT specimens at -10  $^{\circ}C \ge 85$  %;
- CTOD  $\geq 0.38$  mm;
- $CE \le 0.38$  %;  $PCM \le 0.20$  %;

• Centerline segregation ranking for concast slabs < 2.0 on Mannesmann Scale (or OST 14-4-73).

An analysis of the specified requirements shows that the steel must have high impact toughness in Charpy V-notch specimens that is almost two times the minimum values for the same characteristic for standard X65 steels (KCV  $\ge$  88.5 J/cm<sup>2</sup>, which is equivalent to A<sub>V</sub>  $\ge$  70.8 J), now applied in Russia for manufacturing onshore gas line pipes designed for pressure 7.4 MPa.

Special attention was given to reducing centerline segregates in the concast slab, whose centerline unsoundness and centerline cracking number shall not exceed 2 by reference to charts in OST 14-4-73 "Method of macrostructure examination in the billet (slab) manufactured by continuous casting". To ensure high weldability and cracking resistance in pipes, especially in pipe welded offshore, carbon equivalent  $CE \le 0.38$  % and parameter PCM  $\le 0.20$  % were specified.

## **Chemical Composition**

The basic chemical composition of the steel corresponded to formula 0.07%C-1.6%Mn-0.075% Nb, follow in the 07G2B specification, and complying with the present day concept for high performance niobium-containing steels for large diameter gas line pipes for critical applications. These are characterized by lower carbon content, manufactured using thermomechanical controlled process (TMCP) [1, 2].

Steel was made in 350-t converters and, after ladle treatment; it was cast by slab concasters. Over 200 heats were made. Histograms of frequency distribution of primary elements in X65 steel are shown in Figure 1. Descriptive mathematical statistics for the test heat compositions shows rather little deviations from the average values for the basic chemical elements. In general, the steel was low carbon ( $\leq 0.08$  %), low sulfur ( $\leq 0.004$  %) and had an economical content of alloying elements. Major alloying elements were manganese, niobium and silicon. Economical alloying and low carbon content ensured moderate carbon equivalent CE and low Pcm parameter, which did not go above 0.36 and 0.17 %, respectively (Figure 2).



Figure 1. Bar diagrams for frequency distribution of chemical contents in X65 heats for deep-water gas pipeline.



Figure 2. Bar diagrams for frequency distribution of weldability parameters *CE* and *P*cm in X65 heats for deep-water gas pipeline.

### **Metallurgical Considerations and Analysis**

Concast slabs were rolled on a two-stand 3-6 metre wide reversing plate mill using TMCP practices. These included heating to 1180 °C, roughing at 1000–980 °C to 3-3.5 final gauge followed by finishing rolling to 24, 27 and 31 mm thicknesses in the temperature range 730–690 °C. Then plates were then subjected to thermodiffusion treatment by slow pile cooling at temperature from 450 to 100 °C. This operation was used to remove diffusible hydrogen from the steel, to release internal stresses and to improve structural conditions in the centerline zone of the plates, which was beneficial for plate soundness and plasticity [3].

Assessment of centerline chemical non-uniformity of the cast slabs by OST 14-4-73 and the Mannesmann scale was performed on slab cross-sections after deep etching at 60-80 °C in 50 percent water solution of hydrochloric acid. The microstructure of hot rolled plates was examined by optical microscope and scanning electron microscope. Using image analyzer SIAMS-600, quantitative evaluation of rolled plates microstructure with determination of grain size and volume fraction of structural components was performed. Determination of the fracture appearance shear area of Charpy specimens impact tested at -30 °C was carried out using scanning electron microscopy. Chemical analysis of nonmetallic inclusions on the fracture surface of CVN specimens was carried out using energy dispersive X-ray spectrometer in the scanning electron microscope. Tensile properties were determined on flat ASTM A370 test specimens, with gauge length 2" (51 mm). CTOD tests at 0 °C were performed to assess resistance to cracking. Crack tip opening displacement value,  $\delta_m$ , was determined according to BS 7448.

Tables I and II show the chemical compositions of typical heats and the combination of the mechanical properties of plates made from those heats, in thicknesses 24, 27 and 31 mm. Figure 3 shows the histograms of frequency distribution of mechanical testing for 24, 27 and 31 mm plates. It is seen that the mechanical properties of the X65 steel plates fully meet the order requirements. CTOD-test results for more than 40 plates demonstrated that crack tip opening displacement  $\delta_m$  was between 0.38-0.9 mm, at -10°C which is indicative of adequately high crack resistance.

Heat	C	Mn	Si	S	Р	Al	Ti	Nb	Ν	$CE^1$	$PCM^2$
1	0.06	1.70	0.26	0.004	0.011	0.020	0.008	0.079	0.009	0.35	0.16
2	0.07	1.62	0.26	0.003	0.010	0.032	0.012	0.076	0.006	0.34	0.16
3	0.07	1.55	0.24	0.003	0.011	0.031	0.010	0.083	0.007	0.34	0.16
4	0.07	1.59	0.18	0.003	0.010	0.040	0.011	0.073	0.005	0.34	0.16
5	0.07	1.62	0.21	0.003	0.012	0.029	0.008	0.077	0.006	0.35	0.16
6	0.06	1.65	0.20	0.004	0.008	0.036	0.012	0.075	0.007	0.34	0.15
$ ^{1}CE = C + \frac{Mn}{6} + \frac{Cr + Mo + V}{5} + \frac{Cu + Ni}{15} ^{2}PCM = C + \frac{Ni}{60} + \frac{Si}{30} + \frac{Mn + Cr + Cu}{20} + \frac{Mo}{15} + \frac{V}{10} + +5B $											

Table I. Chemical composition of X65 heats

Table II. Mechanical properties of 24, 27 and 31 mm plates, of X65 Steel

								Shear area	ı		0 T 0 T
Plate		TS,	YS,	YS/	El	AV	CVN	-30 °C	-10 °C	HV10	CTOD
thickness,	Heat	MPa	MPa	UTS	(2"),	(-30°C),	(-30°C)	(Charpy)	(DWTT)		(0 °C)
mm					%	J	J/cm <sup>2</sup>				$\delta_{m}, mm$
24	1	565	493	0.87	29	278	348	100/100	95/95	188	0.55
	2	565	492	0.87	30	225	281	100/100	95/95	187	0.50
27	3	565	499	0.88	29	217	271	100/100	95/95	189	0.49
	4	550	488	0.88	30	236	295	100/100	95/95	188	0.49
31	5	540	469	0.87	28	146	185	100/100	95/95	187	0.40
	6	551	483	0.88	32	167	209	100/100	95/95	189	0.39



of 24, 27 and 31 mm X65 steel plates for deep-water gas pipeline.

From impact testing of CVN specimens high toughness values at -30 °C were obtained without any brittle fracture. Investigation of impact test specimens' fracture appearance showed the presence of delaminations representative of TMCP with rolling finishing temperatures in the  $\gamma$ + $\alpha$ -region. By microfractographic examination using scanning electron microscope it was established that, as a result of TMCP, regular fibro-banded fracture appearance, favorable for steel cold resistance, occurred in plates, with the surface consisting of alternating transcrystalline ductile fracture appearance and transcrystalline quasi-brittle fracture appearance (Figure 4). All DWTT specimens tested at -10 °C produced brittle fracture appearance less than 5 percent.



Figure 4. Typical fracture appearance of CVN specimens of X65 steel plates tested at -30 °C: 1 — transcrystalline ductile fracture; 2 — transcrystalline quasi-brittle fracture; 3 — globular nonmetallic inclusions.

High impact toughness of the steel was achieved by the use of low content of carbon (0.06-0.08 %) and sulfur (0.003-0.004 %), low level of contamination from nonmetallic inclusions and globular shape of such inclusions as a result of calcium treatment. This prevented occurrence of lamellar, MnS inclusions elongated in the rolling direction. Compound sulfides of globular shape, in contrast to lamellar MnS, are not stress concentrators and do not have any negative effect on impact toughness values. Figure 4 shows globular sulfides located on the fracture surface. X-ray spectrum, Figure 5, shows a complex composition of this sulfide that, in addition to Mn and S, also contains Ca and Al.



Figure 5. X-ray spectrum of globular nonmetallic inclusions found on the fractured CVN specimens' surface (see Figure 4).

#### **Metallographic Examination**

Metallographic examination of X65 plates showed that the structure in all thicknesses was a combination of fine ferrite grain with the average grain size Number 12 according to GOST 5639 and pearlite in the amount of 5-7 percent according to GOST 3443 (Figure 6). Significant grain refining was achieved by microalloying by higher amount of niobium. This element acts to restrain austenite grain growth during heating, retards recrystallization processes during rolling and increases the initial temperature of recrystallization thus extending the temperature region of non-recrystallized austenite existence during TMCP [4,5]. As a result, finishing rolling of the steel was performed below the temperature of deformed austenite recrystallization. Austenite deformation at temperatures below the recrystallization temperature leads to formation of elongated austenite grains thus increasing

the specific effective surface area. The increased specific surface area, occurrence of strain bands, twin grain boundaries and other lattice defects result in the increased rate of the new phase nucleation at polymorphic  $\gamma \rightarrow \alpha$ -transformation, which contributes to fine dispersion of the final structure. The ultimate formation of fine the grained structure took place during finishing rolling as a result of dynamic  $\gamma \rightarrow \alpha$ -transformation due to plate strain below temperature A<sub>r3</sub>.



Figure 6. Typical microstructure of the base metal (a) and of the centerline zone (b) of X65 steel plates for deep-water gas pipelines: F — ferrite; P — pearlite; B — bainite.

To improve weldability and to produce structures with only slight centerline chemical and structural non-uniformity, X65 steel was made with 1.5 times less carbon content compared to the accepted values for conventional pipe steels 10G2FB. Centerline segregation in X65 slabs was subtle: the rating for centerline unsoundness was in most cases 1.0, for centerline cracks 0-0.5 and for centerline chemical non-uniformity 0-1.5 by OST 14-4-73 (Figure 7a).



Figure 7. Comparison analysis of the centerline segregation in X65 slab cross-section (a) and in 10G2FB (b) and 17G1S-U (c) slabs cross-sections.

Comparison of the results obtained from the assessment of X65 slabs centerline chemical segregation with the results presented in Paper [6] for pipe steel with higher carbon contents 10G2FB and 17G1S-U indicates that reducing lower carbon in steel from 0.19 percent (17G1S-U) to 0.06 percent (X65), the segregation rating decreases from 4 to 1.5 by OST 14-4-73 and from 5 to 2 by Mannesmann scale (Figure 7, Table III) under similar casting conditions.

Steel	С	Mn	S	OST 14-4-73	Mannesmann scale
X65*	0.06	1.70	0.004	1.5	2
10G2FB	0.11	1.69	0.004	2	<3
17G1S-U	0.19	0.43	0.012	4	5

Table III. Comparative analysis of centerline chemical segregation ranking for X65 slabs and 10G2FB and 17G1S-U slabs

\* Chemical composition of X65 steel corresponds to Heat 1 in Table I.

The lower segregation intensity in the X-65 slabs had a positive effect on plate microstructure. The microstructure of the plate centerline zone differed insignificantly from the base metal microstructure and was ferrite-pearlite with individual small areas of low carbon bainite (see Figure 6b). The average microhardness in the centerline zone was  $Hv_{1.96}=2050$  MPa, and that of the base metal at 1/3 thickness was  $Hv_{1.96}=1860$  MPa. In performing quantitative assessment the structural non-uniformity coefficient K(Hv<sub>1.96</sub>) was used defined as the ratio between the centerline zone microhardness and the base metal microhardness whose value for X65 plates was 1.10. In papers [6, 7] it is demonstrated that for conventional pipe steels with higher carbon the structural non-uniformity coefficient K(Hv<sub>1.96</sub>) is much higher. In Figure 8 it is seen that by reducing carbon from 0.19 percent to 0.06 percent the structural non-uniformity coefficient is reduced from 1.79 to 1.10. Low values of the structural non-uniformity coefficient of the tested steel are suggestive of high uniformity of the plate throughout the entire thickness.



Figure 8. Carbon content effect on structural non-uniformity coefficient  $K(HV_{1.96})$  in pipe steels.

The minimalized centerline chemical and structural non-uniformity in the X65 steel are attributable to its low carbon content. It is known that a decrease of carbon below 0.09 percent brings about noticeably decreased segregation of heavily segregating elements, such as carbon, manganese, sulfur, phosphorus and others [6, 7]. Such a decrease of segregation during solidification in continuous casting of steel with carbon < 0.09 percent can be explained by the absence of peritectic reaction, shorter crystallization times and greater residence time in the  $\delta$ -ferrite field.

Considering that diffusion mobility of chemical elements in  $\delta$ -ferrite exceeds by two orders of magnitude the rate of diffusion in austenite, the longer existence of slabs in the  $\delta$ -ferrite region results in more homogeneous re-distribution of elements over the entire slab volume [8]. Higher uniformity of elements distribution in slab decreases the probability of occurrence of structural non-uniformity in the centerline zone of plate.

Azovstal X65 steel plates were formed by roll bending and cold expending into pipe at Welspun Pipe Co. in Dahej, India. The dimensions of the pipe were 20" OD by 30.99 mm thickness. Figure 9 demonstrates the influence of the pipe making process on the base metal mechanical properties of Azovstal X65 plates. It can be seen that an increase in both YS and UTS values, occurs without significant lowering of elongation.



Figure 9. Influence of pipe making process on the mechanical properties of the X65 steel.

### Conclusions

1. Commercial production of X65 plates, in thicknesses up to 31 mm, for deep-water gas pipeline was started at Azovstal; the steel is characterized by lower carbon content and economical alloying (absence of vanadium and molybdenum) that resulted in low values of CE ( $\leq 0.37$  %) and PCM ( $\leq 0.17$  %).

2. Achieved values of mechanical properties for X65 rolled plates, in thicknesses 24, 27 and 31 mm, were as follows: YS = 450-520 MPa; UTS = 530-600 MPa; YS/UTS = 0.82-0.88; El (2") =28-38 %; KCV<sub>-30</sub> $^{\circ}_{\rm C}$  = 162-375 J/cm<sup>2</sup>. Ductile fracture in Charpy V-notch specimens at - 30 $^{\circ}_{\rm C}$  was 100% and on DWTT specimens at -10  $^{\circ}_{\rm C}$  it was  $\geq$  95 %.

3. Centerline segregate non-uniformity of X65 slabs was barely visible: centerline unsoundness was 1.0-1.5, centerline cracking was 0-0.5 and centerline chemical non-uniformity was 0-1.5 to OST 14-4-73. The microstructure of plates manufactured from the new steel was uniform, which is demonstrated by the low value of structural non-uniformity coefficient for plates K(HV<sub>1.96</sub>) that was equal to 1.10.

## References

- 1. Heisterkamp F. et al., *Nb-containing low alloy steels (*M.: "JV INTERMET INGINEERING", 1999), 94.
- 2. Hulka K. and Gray J. M. "High temperature processing of line-pipe steels" (Paper presented at the International Symposium Niobium 2001 held in Orlando, Florida, USA. December 2-5, 2001).
- 3. Novik V.I. et al., "Manufacture of rolled plates with improved continuity" *Steel*, 5 (1991), 63-66.
- Palmiere E. J. et al., "The Influence of Niobium Supersaturation in Austenite on The Static Recrystallization Behavior of Low Carbon Microalloyed Steels" *Metal Trans.*, 27A (4) (1996), 951.
- 5. Kwon O. and DeArdo A. J. "Interactions Between Recrystallization and Precipitation in Hot-Deformed Microalloyed Steels" *Acta Met.*, 39 (1991), 529.
- 6. Matrosov Yu. I. and Nosochenko A.O. "Study of chemical and structural segregation in concast slabs and plates for large diameter gas line pipes" ("Advanced rolled steel plates for large diameter gas line pipes and special application steel structures", Collected presentations, M.: Metallurgizdat, 2004), 70-75.
- 7. Nosochenko A.O. et al., "The properties of pearlite-free X65 X70 steel, 0.03C-1.5Mn-0.09Nb type" *Metallurg*, 12 (2003), 30-33.
- 8. Ogibayashi S. et al., "Influence of Roll Bending on Centerline Segregation in Continuously Cast Slabs" *ISIJ Intern.* V. 31, 12 (1991), 1408-1415.