## DEVELOPMENT AND PRODUCTION OF HIGH STRENGTH PIPELINE STEELS

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#### Abstract

In view of the ever-increasing pipeline length and operating pressure, the development of highstrength steels makes a significant contribution to pipeline project cost reduction. In the case of offshore pipelines, the operating pressure is not the most important but rather the ambient water Therefore, one of the design criteria for offshore pipelines is less the strength but pressure. more the collapse behaviour of the pipe. The pipe to be used in offshore pipeline construction should possess not only good materials properties but also good geometry to ensure good collapse strength. As the H<sub>2</sub>S content of the gas being transported increases, the requirements for HIC resistance of the pipe material increase. When an aqueous phase is present, CO<sub>2</sub>, H<sub>2</sub>S and chlorides are extremely corrosive. For applications in such corrosive environments, a pipe made of either all-corrosion resistant material or of a low-alloy steel pipe clad with a high-alloy corrosion resistant material is used. The initial part of the paper discusses the metallurgical principles and the development of large-diameter linepipe steels. In the second part the production results of different orders represent the state-of-the-art of production of longitudinally-welded large-diameter pipe. Projects of high strength line pipe, pipe for deepwater application, HIC resistant and clad pipe applications are presented. The paper concludes with the need of close collaboration between all parties involved to optimise the pipeline projects in terms of quality and costs.

#### Introduction

Over the past 30 years, severe demands have been placed on the pipe manufacturer for the development and processing of materials to linepipe. Generally, longitudinally welded largediameter linepipe is used for the transportation of oil and gas, because it offers the highest safety in pipeline operation and represents the most economic solution. From the point of view of pipeline economy, the pipe must respond favourably to laying in the field and permit high operating pressures for the pipeline. These requirements imply that the pipeline steel has to possess high strength and toughness and that the pipe has an optimised geometry.

The development of high strength steels is shown in Figure 1. In the seventies, the hot rolling and normalising was replaced by thermomechanical rolling. The latter process enables materials up to X70 to be produced from steels that are microalloyed with niobium and vanadium and have a reduced carbon content. An improved processing method, consisting of thermomechanical rolling plus subsequent accelerated cooling, emerged in the eighties. By this method, it has become possible to produce higher strength materials like X80, having a further reduced carbon content and thereby excellent field weldability. Additions of molybdenum, copper and nickel enable the strength to be raised to that of grade X100, when the steel is processed to plate by thermomechanical rolling plus modified accelerated cooling.



Figure 1: Development of high strength steels.

About 8 million tonnes of linepipe is produced every year. The major part of it consists of pipe in standard material grades, which are not addressed in detail in this paper. The big challenge to the linepipe manufacturer is posed by the projects that can only be executed by means of special efforts and measures; that means high strength, offshore, HIC resistance and clad pipe. High-strength pipe in grades X70 and X80 are currently being used in the construction of longdistance pipelines, and pipe in grades X90 and X100 are currently being evaluated. Having explored the major part of the reservoirs in shallow waters, the drilling activities and hence pipeline installations are being shifted into increasingly deep waters. For instance, an offshore pipeline is currently being installed at a depth of more than 2000 m. The pipe used in the construction of this pipeline has very little in common with the pipe used in the construction of onshore gas transmission pipelines. Additionally good resistance against sour gas is required for the pipes. In the case of highly corrosive fluids another manufacturing process, cladding of the pipe, has to be established. Pipe with such a combination of conflicting properties can only be produced when the metallurgical principles are properly understood and optimally utilised.

## **Microstructural Influences**

#### Ferritic and Bainitic Structures

Microstructural features such as dislocations, grain boundaries and precipitation, govern the mechanical properties of steels. In low-alloy steels, they develop in the course of transformation of the austenite during cooling, and the development depends on the cooling rate and cooling stop temperature.

Figure 2 shows, how the combination of the different types of microstructures contribute to increase mechanical strength and toughness of steels starting from normalized X60 grade, which was mainly used in the early seventies (1). The steel typically contains about 0.2% carbon, 1.55% manganese, 0.12% vanadium, 0.03% niobium and 0.02% nitrogen.



Figure 2: Microstructural effects for enhancing strength and toughness properties.

The thermomechanically processed X70 steel mentioned in the figure was microalloyed and contains only just 0.12% carbon. Thermomechanical rolling results in a significant reduction of the ferrite grain size. Grain refinement is the only method by which both strength and toughness can be improved simultaneously. The loss of strength resulting from reduced pearlite contents can be offset by precipitation hardening and dislocation hardening. Reduction of the pearlite content, grain refining, dislocation hardening and precipitation hardening contributed individually and in combination to the development of X70 steel with improved weldability and favourable ductile-brittle transition temperatures.

Further increases in strength and toughness, which led to the development of X80 steel, can only be attained by changing the microstructure of the steel matrix from ferrite-pearlite to ferrite-bainite. In comparison with the thermomechanically rolled X70 steel, the X80 steel has a further reduced carbon content, reduced grain size and an increased dislocation density. These two steel grades also differ in their precipitation characteristics.

Figure 3 shows typical microstructures of three types of linepipe steel. Banded ferrite and pearlite and coarse ferrite grain size (ASTM 7-8) are the characteristic features of

conventionally rolled and normalised X60 steels. The microstructure of TM rolled X70 steels is more uniform and the ferrite grains are finer (ASTM 10–11). The most uniform and extremely fine microstructure is attained by accelerated cooling that follows thermomechanical rolling, as shown for the X80 steel. The improved properties of this steel can be attributed to its ferritic-bainitic microstructure.



Figure 3: Microstructures of normalised, thermomechanically treated and accelerated cooled steels

The basic morphological difference between the polygonal ferrite and bainite is illustrated in the following figures. Figure 4 shows electron microscope photographs of the grain structure of these two microstructural types. The effective grain size of bainitic microstructure cannot be established by optical microscopy, because, large and small angle grain boundaries are not discernible with an optical microscope. Therefore, the electron diffraction patterns of a sufficient number of test points on the screen must be systematically examined by means of dark field images (2). As shown in figure 5, a statistical mean grain size of less than 1  $\mu$ m can be obtained for the bainite, whereas the grain size of ferrite is a multiple of this value (3).



Figure 4: TEM microstructures of ferrite and bainite.

A further important difference is the substantially higher dislocation density in bainitic structures. Dislocation density measurements involve considerable experimental expenditure in the electron microscope, because in addition to counting out the dislocations, the foil thickness

must be determined at many points by means of convergent diffraction. The statistical evaluation of such dislocation density measurements can be seen in Figure 6.



Figure 5: Grain sizes of ferrite and bainite.



Figure 6: Dislocation densities of ferrite and bainite.

The results for the ferritic and bainitic microstructures are based on the measurements on largediameter pipe made of manganese-niobium steels. The thermomechanical rolling conditions were the same for both microstructures. The most important micro-structural differences between bainite and ferrite are mainly attributable to the lower temperature of formation of bainite.

Transformation into bainite can be effected by additions of boron or nickel and molybdenum to the steel. Figure 7 shows the continuous cooling transformation (CCT) diagram of a steel containing 0.08% carbon, 1.44% manganese, 2.31% nickel, 0.2% molybdenum, and 0.04% niobium, which after cooling in air contains about 50% bainite in the microstructure (4). But alloy additions made to obtain bainitic fractions increase the carbon equivalent, which may influence the steel's field weldability.

As can be seen in Figure 8, a microstructure consisting of 50% ferrite and 50% bainite can also be obtained with a normal manganese-niobium steel by subjecting it to accelerated cooling. Using a special water cooling system at the end of the thermomechanical rolling, the austenite passes more quickly through the ferritic range as shown in the CCT diagram, and the formation of pearlite is completely suppressed (5).



Figure 7: CCT-diagram of MnNiMoNb-steels.



Figure 8: CCT-diagram of accelerated cooled MnNb-steels.

## Effect of Microalloying Elements

In the following, the effect of niobium and titanium on the steel's microstructure is discussed (3). In case of thermomechanically rolled large-diameter pipe steels, the effect of the

microalloying elements on the mechanical properties is governed by their tendency to bind carbon and nitrogen. For the purposeful development of micro-alloyed steels, a thorough knowledge of this bonding behaviour is required.

According to the electron microscope photographs of extraction replicas shown in Figure 9, the lower final rolling temperature gives an increased number of fine niobium carbonitrides precipitates. In the case of the titanium carbonitrides, a change in size and quantity – compared with the higher final rolling temperature – is not established.



## **TEM (extraction replica)**

Figure 9: TEM microstructures of carbonitride precipitations.

For a number of micro-alloyed steels with varying niobium and titanium contents, the chemical composition of the carbonitride precipitates was determined by means of EDX-analyses and lattice spacing measurements. A substantial proportion of the niobium contained in manganese-niobium steels is used up in the upper austenite range in binding nitrogen. This means that in the lower austenite or ferrite range, only a small amount of niobium is available for precipitation.

In a manganese-niobium-titanium steel with a titanium to nitrogen ratio not below the stoichiometric value, the precipitation begins with titanium nitride containing very small amounts of niobium and carbon, even at high temperatures. Niobium can then precipitate, mainly in the form of carbides. The dissolution temperature of carbides is lowered significantly in the absence of residual nitrogen. Consequently, when the slab is reheated to the rolling temperature a substantial proportion of the niobium gets dissolved, while stable titanium nitrides remain un-dissolved. During thermomechanical rolling in the lower austenite region, fine particles with high niobium contents precipitate anew. This leads to a fine austenite grain size and thus contributes to the hardening of the ferrite. On the other hand, sufficient niobium is available for precipitation hardening through coherent precipitates in the ferrite.

From a large number of such particle analyses and the pertinent diffraction patterns, the chemical composition of the incoherent precipitates was determined. Figures 10 and 11 show the frequency distribution of the lattice constants of the carbonitride precipitates in manganese-niobium and manganese-niobium-titanium steels (6). A comparison of Figures 10 and 11 reveals that in the latter steel, as a result of titanium precipitation, a great proportion of the

niobium-carbonitrides exhibits very high carbon content which at low temperatures is available for the precipitation with the associated desirable effect.



Figure 10: Distribution of lattice constants of carbonitrides in MnNb-steels.



Figure 11: Distribution of lattice constants of carbonitrides in MnNbTi-steels.

These microstructural changes can be clearly studied using STEM techniques. Figure 12 gives an example of EDX analysis of the carbonitride precipitates in a manganese-niobium-titanium steel. The spectrum in the upper part of Figure 12 shows the metal contents of the cube-shaped carbonitrides, containing predominantly titanium, with small amounts of niobium. The lower part of this figure shows the metal contents of the spheroidal shaped carbonitrides, which consist almost exclusively of niobium.



Figure 12: EDX-analysis of carbonitride precipitations.

## Thermomechanical Rolling and Accelerated Cooling

To achieve a homogeneous fine-grained microstructure and hence improved strength, good toughness properties and high HIC resistance, compared to steels produced by conventional thermomechanical rolling, the accelerated cooling process is adopted in the plate mill.



Figure 13: Schematic illustration of thermomechanical rolling with and without accelerated cooling during the  $2^{nd}$  and  $3^{rd}$  rolling stage.

The metallurgical processes occurring during thermomechanical rolling in conjunction with accelerated cooling can be understood from the schematic diagram presented in Figure 13 (7), in which the most important rolling stages and rolling parameters to be controlled are shown.

The cooling system used here can be put into operation twice during rolling. Cooling operation 1 enhances the grain refinement of ferrite, whereas cooling operation 2 prevents the formation

of pearlite during cooling, thereby improving the homogeneity of the final microstructure. Important variables of the cooling operations are cooling rate and cooling stop temperature. This special cooling system permits cooling to be carried out after the second and third stages of rolling since it is quite compact and is installed close to the rolling stand.

The essential rolling parameters of the thermomechanical process are:

- the slab reheating temperature (SRT) for dissolution of the precipitated carbonitrides,
- the roughing phase for producing a fine, polygonal austenitic grain by means of recrystallisation,
- the final rolling temperature (FRT), which must be maintained within the range of the non-recrystallising austenite, and
- the degree of final deformation (FD) in this temperature range.

If accelerated cooling is employed, the following additional parameters are considered:

- the cooling rate, and
- the cooling stop temperature (CST).

Figure 14 shows the effect of accelerated cooling that follows the first and the second finish rolling operations on microstructure. A few pearlite islands are present in the mid-wall region of the thermomechanically rolled plate. As a result of the two-stage cooling, not only is the ferrite grain size further refined but also the pearlite is replaced with bainite. The microstructure of the accelerated-cooled material gives the impression of homogeneity. The homogeneity of this microstructure has also improved the strength and toughness properties of the accelerated cooled material.





Figure 14: Effect of accelerated cooling on the microstructure of TM-steels.

## **High Strength Linepipes**

Over the past two decades, extensive work has been carried out to develop high-strength steels in grades X80 and X100 to assist customers in their endeavour to reduce weight and pipelaying costs.

The developments in alloy design since 1984 can be seen in the results (8) on pipe produced commercially for the Megal II, CSSR and Ruhrgas projects (Table I).

A manganese-niobium-titanium steel, additionally alloyed with copper and nickel, was used in the production of the 13.6mm wall pipe for the Megal II order in 1984. Subsequent optimization of production parameters enabled the CSSR order to be executed using a manganese-niobium-titanium steel without the additions of copper and nickel. This has simultaneously led to reduction in the carbon equivalent of the steel used.

Order	Pipe geo- metry	с	Si	Mn	Р	S	AI	Cu	Cr	Ni	Мо	Nb	ті	N	ııw	РСМ
Megal II	44" x 13.6mm	.081	.42	1.89	0.011	.0016	.038	.18	.04	.18	.01	.044	.018	.0052	.430	.206
CSSR	56" x 15.6mm	.085	.38	1.85	0.014	.0017	.030	.03	.05	.03	.01	.044	.019	.0060	.409	.197
Ruhrgas	48" x 18.3mm	.09	.40	1.94	0.018	.0011	.038	.03	.05	.03	.01	.043	.017	.0040	.435	.213

Table I Chemical composition of GRS 550 / X80 pipe

In 1992, 48" diameter pipe was produced for a Ruhrgas pipeline project in Germany with 250km length requiring GRS 550 (X80). Also pipes of 48" diameter with 19.3mm wall thickness were included. Since the strength decreases as the wall thickness increases, it was necessary to raise the carbon and manganese levels marginally. The concentrations of all other elements remained unchanged.

Table II shows the mean values of the mechanical properties measured on the pipes produced for the three orders. The measured tensile and the impact energy values conformed fully to the specification requirements in all cases. The standard deviation for the yield and tensile strength values is very low. The impact energies measured on Charpy V-notch impact specimens at  $-20^{\circ}$ C are very high, resulting in an average value of about 180J. The 85% shear transition temperatures determined in the drop weight tear (DWT) tests are far below  $-20^{\circ}$ C.

Pipe Order geo-		Yield strength MPa		Tensile strength MPa		Elongation 5d (%)		CVN, -20 °C (Joule)	
	metry	х	S	х	s	ТХ	S	x	S
Megal II	44" x 13.6mm	603	21	737	22	22	2	182	30
CSSR	56" x 15.6mm	607	18	716	10	23	2	183	29
Ruhrgas	48" x 18.3mm	592	14	729	16	21	1	176	32

Table II Mechanical properties of GRS 550 / X80 pipe

DWTT (-20°C) SA >85%

Figure 15 gives an idea of the Ruhrgas pipeline project (9). After welding, non destructive examination and coating, the girth welded segments were lowered onto the prepared trench bottom.

One of the latest projects using X80 was a UK pipeline for BG Transco in 2000. The results of the tensile tests, performed in the context of certification of the pipe, are shown in figure 16. All values conform to the requirements of X80. The standard deviations are 15MPa for yield strength, 13MPa for tensile strength, 0.02 for yield to tensile ratio and 1.8% for elongation.



Figure 15: Pipe laying of the Ruhrgas Schlüchtern-Werne-Project.



**Base Material - Tensile Test** 

Figure 16: Distribution of mechanical properties of 48"diam. X80 linepipe with 15.1 mm wt.

Table III contains the results obtained on commercially produced 36" diameter pipe with 32.0mm wall thickness in API grade X80. The manganese-niobium-titanium steel used here has a sufficiently high ratio of titanium to nitrogen and is additionally alloyed with molybdenum. The low carbon equivalent ensures good field weldability. The elongation values (A2") are particularly high. The Charpy V-notch impact energy measured at  $-40^{\circ}$ C is over 200J and the shear area of DWTT specimens tested at  $-20^{\circ}$ C is greater than 85%. The forming and welding operations carried out on this high strength steel did not cause any problems.

Table III Chemical and mechanical properties of 36" diam. X80 pipe with 32mm wt

				Mea	in chen	nical co	mposi	ion [v	vt.%]					
С	Si	Mn	Р	S	AI	Cu	Cr	Ni	Мо	Nb	Ti	N	IIW	РСМ
.07	.27	1.86	.015	.0010	.036	.02	.03	.02	.15	.040	.023	.0057	.419	.186
Specimen R0.5 Rm Rt/Rm A2" Charpy V-notch (1/1), trappy or rot - 40°C DWTT, -20°C							20C							
orier (and	ntat. type)	MP	a N	IPa	%	%	J		J	J	Ave J	r. S.	A.	S.A. %
Trans (flat b	sverse bar)	55	9 6	85	82	2 47 222 219 231 224 85				5	90			
Trans (round	sverse I bar)	<b>57</b>	9 6	74	86	46								

#### Development to X100

Only improvements in the existing technology were involved in the production of grade X100 plate. As a result, the production window in thermomechanical rolling and accelerated cooling is quite narrow. Heat treatment of plate or pipe is obviously not advisable. As can be seen in figure 17, three different approaches are generally possible with respect to the selection of chemical composition and cooling conditions (10).



Figure 17: Modification of steel chemistry and cooling parameters to achieve the strength level of X100.

Approach A, which involves a relatively high carbon content, has the disadvantage that the crack arrest toughness requirements to prevent long-running cracks, may not be fulfilled. Moreover, this approach is also detrimental, e.g. to field weldability.

Approach B, which is practically the same as direct quenching with fast cooling rates down to a very low cooling-stop temperature, results in the formation of uncontrolled fractions of martensite in the microstructure, which have a detrimental effect on toughness properties of base metal and lead additionally to the softening in the heat affected zone. This effect cannot be adequately compensated for by extremely low carbon contents, without adversely affecting

productivity. Moreover, it is very difficult to produce pipe with adequate uniformity of strength properties. This difficulty cannot be attributed solely to the Bauschinger effect associated with the variation in local deformation occurring during the heavy straightening operation which is needed in the case of thin section plate, because it distorts heavily during direct quenching.

Experience gained meanwhile indicates that Approach C is the best choice. This approach enables the desired property profile to be achieved through an optimized two-stage rolling process in conjunction with a reduced carbon content, a relatively high carbon equivalent and optimized cooling conditions. The special potential of the existing rolling and cooling facilities contributes significantly to the success of this approach.

Approach C, which involves a low carbon content, ensures excellent toughness as well as fully satisfactory field weldability, despite the relatively high carbon equivalent. The chemical composition should therefore be considered acceptable for the purpose of current standardization.

Large-diameter grade X100 pipes were produced on a trial basis for the third and burth time. The pipes are intended for use as test pipes to determine the behaviour of grade X100 material in full-scale burst tests (Figure 18), which are conducted as part of an ECSC-funded research project (11).



Figure 18: Full-scale burst tests on X100.

The mechanical properties determined on the pipes are quite appreciable, although they do not fully conform to the requirements currently specified for lower-strength material grades. The current requirements particularly for yield to tensile ratio and elongation, which are applicable to material grades up to X80, are too severe to be fulfilled for grade X100 on a statistical basis. This difficulty is attributable to the basic finding that as the strength increases, the yield to tensile ratio increases and the elongation decreases. The X100 material produced responds favourably to manual and mechanised field welding, a finding which can be attributed to its reduced carbon content.

For reasons of technical feasibility and cost-effective production, it is necessary in the context of grade X100 to reassess and redefine some of the requirements for the mechanical properties, considering the expected service conditions for the pipe.

## **Linepipes for Deepwater Application**

A further new challenge to the pipe manufacturer and the pipelaying contractor is the offshore pipeline in deeper waters. A consortium has been carrying out a feasibility study since 1993, for a gas transmission pipeline from Oman to India in 3500m deep water.

Table IV shows the most important requirements for the pipe (12). To prevent collapse of the pipeline under an ambient external pressure of about 350 bar, the linepipe to be used has to meet severe requirements. The extreme pipe geometry was characterized by a wall thickness of 41mm and 28" diameter.

Table IV Requirements for Oman-India gas-transmission pipeline

Length of subr	marine line:	1200 km
Max. water de	pth	3500 m
Max. pipe size	2	610 mm I.D. x 41 mm W.T.
Material:		X70 non- <del>s</del> our
Yield strength,	,	482 - 586 MPa <i>Longand</i>
Tensile streng	10°C:	565 - 793 MPa∫ <i>transverse</i>
CVN base, -	10°C:	200 / 150 J
CVN weld, -	10°C:	100 / 75 J
DWT, -	10°C:	min. 85% SA
CTOD weld, -	10°C:	min. 0.15 mm
Out - of - round	dess:	£4 mm

Mechanical strength and geometry of the pipe are the two most important factors that affect the collapse strength. High strength values, which need to be uniform over the pipe circumference, reduce the susceptibility of pipe to collapsing. The material for the pipe is grade X70 (non-sour). For offshore pipelines, the pipe is often required to meet the requirement for yield strength also in the longitudinal direction. The toughness requirements are quite high at >200J average and >150J individual for the base material and >100J average and >75J individual for the weld. DWT test and CTOD test are also specified. Out-of-roundness of the pipe has a detrimental effect on collapse strength. Hence, the requirement for out-of-roundness reads: as low as possible, but 4mm maximum.

An exclusive contract was awarded in 1995 to produce the first 1000m of pipe for weldability trials. Forming pipe with such an unfavourable diameter-to-thickness ratio places severe demands on the pipe forming equipment, e.g. the crimping press, Uing press, Oing press and mechanical expander. Extensive laboratory work involving finite element analysis was conducted to determine the parameters needed for forming the pipe and the loads occurring on the forming equipment. Figure 19 shows the chemical composition and the macrosection of the linepipes produced for this contract.

0.1011												
	С	Si	Mn	Р	S	AI	Cu	Cr	Ni			
base	0.09	0.25	1.69	0.011	8 *)	0.044	0.02	0.03	0.22			
weld	0.07	0.29	1.40	0.012	41 *)	0.022	0.03	0.04	0.15			
	Мо	V	Ti	Nb	N	В	CE	PCM				
base	0.01	0.08	0.003	0.05	36 *)	1 *)	0.41	0.20				
weld	0.20	0.05	0.022	0.03	56 *)	36 *)	0.38	0.20				

Chemical composition (wt%): Base material and weld metal

\*): ppm



Figure 19: Chemical composition and macrosection of Oman-to-India-X70 pipes, 28" diam. with 41.0mm wt.

The variation in pipe strength, the pipe geometry and the Bauschinger effect caused by pipe forming operation all have an effect on the collapse strength. A further aspect is the ovality that should be less than 4 mm; results of the produced linepipes are shown in figure 20.



Figure 20: Production results of Oman-to-India X70 pipes ovality at pipe ends.

Some collapse tests on linepipe were performed to investigate the collapse behaviour (Figure 21). The results are given in Table V. The values shown as calculated collapse pressures were determined by an analytical procedure (Figure 22). The calculated values are greatly dependent on the compressive yield strength, which can be determined experimentally, and on the initial ovality.

As can be seen, there is little difference in buckling pressure between pipes of different strength for a given level of ovality, except in one case. The effect of compressive yield strength of the material on the buckling pressure is quite significant in the case of pipes with a thicker wall (13).



Figure 21: Photograph showing buckled linepipe section.

Pipe	Measured collapse pressure (bar)	Calculated collapse pressure (bar)	Ovality ሁ (%)	Compress · yield strength σ <sub>c0.2</sub> (MPa)	Tensile yield strength Rt0.5 (MPa)
36 dia X 17.5mm wt (X65)	31.0	30.8	0.5	400	470
36 dia X 31.0mm wt (X65)	147	154	0.4	425	490
28 dia X 35.6mm wt (X65)	311	359	0.5	410	485
28 dia X 42.0mm wt (X65)	421	427	0.7	425	490
28 dia X 41.0mm wt (X70coating.simul.)	484	475	1.4	525	540
28 dia X 44.0mm wt (X70)	468	459	0.6	435	550

Table V Results of collapse tests



Figure 22: Simulation of collapse behaviour with FEA.

Furthermore, it is remarkable that the 28" diameter pipe with 41.0mm wall thickness subjected to a simulated coating thermal cycle has a higher collapse pressure than does the pipe of the

same diameter not subjected to the simulated thermal cycle, but with a higher wall thickness of 44.0mm. This result can also be explained in terms of the Bauschinger effect. As a result of the thermal treatment (at 200°C) prior to testing, the Bauschinger effect on the mechanical strength of the pipe material was almost completely eliminated.

## Linepipes for Sour Service

The development of sour service grades for linepipe with resistance to hydrogen induced cracking (HIC) over the past 10 years has been considerably influenced by the requirements of the market. In the following, the state-of-the-art of linepipe production is illustrated using the data on commercially produced pipe.

The new linepipe steels for sour service are characterized by extremely good toughness properties and high resistance to HIC. These properties have been achieved by means of lean chemistry and highest steel cleanness. This ensures that the steels have a low hardenability and the formation of inclusions and precipitates detrimental to HIC is avoided. Figure 23 gives an idea of the efforts of steel-makers to cope with these requirements.



Figure 23: Production steps of steels with high cleanness requirements for sour service.

To meet the requirements for other properties, such as strength, weldability and fabricability, the steel composition and the process parameters for the steel making and plate rolling are important. It is also important to mention that each production step has to adhere closely to parameters, such as time regime for each step, specially established for the production of HIC resistant steels. The accelerated cooling process in the plate mill leads to a more homogeneous microstructure resulting in improved HIC resistance. The banded ferritic-perlitic microstructure is then replaced by a ferritic-bainitic structure (14).

Table VI shows the results on 260,000 tonne of production pipe for the construction of an offshore gas transmission pipeline (15). In the table the mechanical properties are described by means of average values and standard deviations. The requirements for grade X65 pipe were comfortably met. The important requirements for the chemical composition of an HIC resistant steel are low concentrations of carbon, manganese and sulphur.

	N F	lecha prope	anical erties			Mean		Star	ndard		
-	Transve strength			485			14				
-	Tensile Rm [MPa	streng a]	th			572					
Ì	Yield-to∙ Rt0.5 /Rrr	-tensile [%]	e ratio		85 1.94			1.94	1		
1	Elongation A2"					50.4			1.78	3	
(     	CVN toughness, -20°C Weld metal [J] HAZ [J] Base metal [J]					84     25       310     65       336     38					
I	DWTT [%SA]					93			3.3		
	С	Si	Mn	Ρ	S	AI	V	Nb N IIW P			PCM
Mean comp	.04	.31	1.34	.012	.000	.038	.07	.044	.004	.286	.127

Table VI Production results on 36" diam. X65 linepipe for sour service 260,000 tonnes with 28.4mm wt

As this table reveals these requirements have been strictly observed in the chemical composition of the pipe used for this X65 offshore project. Besides the low carbon and manganese contents the steel features microalloying additions of vanadium and niobium, which were made to meet the requirements for the mechanical properties. The HIC test results on this order are given in table VII. Under the standard test conditions according to NACE (pH = 3, 1bar  $H_2S$ ) the specified acceptance criteria have been reliably fulfilled.

		1110 000010000		<u></u>	
	Sp	pecification	Resultonpipe		
Pipe	Test condition	Acceptance criteria	Base	Weld	
36" O.D. 28.4 mm X65 Sour service	рНЗ	CTR: _ CLR£10.0 CSR£ 2.0	CLR£10.0 CSR£ 0.5	CLR€10.0 CSR€ 0.5	

Table VII Typical HIC test results on sour service pipe

To find a correlation between laboratory tests and real corrosion behaviour of the component, a series of full-scale tests was carried out. The laboratory tests were performed in pH 5 and pH 3 solutions for 92h. The full-scale tests were performed in the pH3 solution, the test duration being 90 days. Both HIC-susceptible and HIC-resistant steels were included in the test. Table VIII shows the results of these tests. Two significant results can be read from the table. First, the ranking of the materials based on the results of the laboratory tests is confirmed by the full-scale tests. Second, the HIC resistant material did not suffer any internal cracking and surface blistering in the full-scale test, although the test solution used was very aggressive.

Table VIII Laborator	y HIC results versus	s full-scale test res	sults (solution pH3)
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Toot	Results						
Test	HIC-susceptible steel	HIC-resistant steel					
Laboratory HIC test 96 h	CLR = 75.7% CTR = 12.4% CSR = 3.7%	CLR = 5.7% CTR = 0.4% CSR = 0.0%					
Full-scale test 90 days (2160 h)	Heavy cracking and blistering	No cracks No blisters					

A series of tests was carried out, prior to the manufacture of linepipe for several orders, to optimise the weld metal composition such that its hardening behaviour is comparable to that of the plate material whilst maintaining the required toughness. This has been achieved by using a specially developed, low carbon, titanium-boron alloyed wire with the remaining composition similar to the parent material. An additional factor that was necessary to fulfill the toughness requirements was the limitation of the nitrogen content of the weld metal. For some big orders, the toughness requirements for weld metal centre and heat-affected zone at -30°C were 37J minimum on individual specimens and 45 J minimum for the mean of three specimens. Despite these exacting testing conditions, all orders could be executed, comfortably fulfilling the specification requirements. Molybdenum-free titanium-boron-alloyed weld metals as well as titanium-boron-free manganese-nickel-molybdenum weld metals have been used, as required by the customers.

To make the step from a heavy wall grade X65 pipe on to a heavy wall grade X70 pipe (30mm W.T.), two different approaches have been developed and applied. The idea of increasing carbon and manganese contents to improve the strength of the steel was abandoned, because these elements segregate and enhance centreline segregation, thereby reducing the HIC resistance of the steel. The Carbon content was 0.04%, the manganese content 1.35%.

The first approach to increasing the strength of the steel is aimed at the distribution and type of microstructural constituents and at achieving additional solid solution hardening. The classical composition of the niobium vanadium-type steel, used for grade X65 pipe, was modified by adding or increasing the concentrations of copper, nickel, chromium and molybdenum in the steel analysis (Table IX). This concept results in a carbon equivalent according to IIW of 0.39.

Also shown in Table IX is the other approach, which is based on increasing the niobium concentration and adding titanium to the steel (16). The niobium content is increased to have higher amounts of niobium in solid solution in the austenite-region as it retards the austenite to ferrite transformation and to increase the strengthening by precipitation hardening. Titanium was added to bind nitrogen thereby preventing the precipitation of niobium carbonitride and making niobium more effective for increasing the strength. This approach leads to an carbon equivalent according to IIW of 0.32.

Mechanical properties	NbTi 0.08Nb 0.025Ti	NbV 0.08V 0.04Nb CuNiMo~ 0.2
Yield strengthR₀.₅[MPa] at RT Transverse Longitudinal	502 504	509 519
Tensile strength R <sup>m</sup> MPa] at RT Transverse Longitudinal	582 566	595 582
Yield-to-tensile ratio Rt0.5/Rm[%] Transverse Longitudinal	86 89	86 89
Elongation A5 [%] at RT Transverse Longitudinal	25 27	24 25
CVN toughness at -30°C Base metal [J] HAZ [J] Weld metal [J]	470 95 140	480 135 160
DWTT [%SA] Base metal at -10°C Base metal at -20°C	92 82	97 91

Table IX Mechanical properties of X70 steels for sour service

It should be noted that both the approaches (referred to as NbV and NbTi type in the tables) were applied in combination with an improved accelerated cooling process after final rolling to produce HIC resistant plates with 30 mm wall thickness in grade X70.

These plates were formed by the U-ing - O-ing - Expanding process into 30" diameter pipes. Following the pipe expansion, samples were taken to determine their mechanical properties and corrosion behaviour.

Table IX also contains the mean values of the mechanical properties for the two different approaches. As can be seen from the data the requirement for a shear area of 85 % minimum in the DWT test  $-10^{\circ}$ C are met by both variants. The Charpy-V-notch impact energy values measured at  $-30^{\circ}$ C are as expected for a low-carbon steel, at above 450J. In the weld seam and the heat affected zone impact energy values in the range of 100 to 160J were measured. In general, the niobium vanadium approach gave more favourable strength values so that the strength requirements for grade X70 can be met in both transverse and longitudinal directions.

The HIC tests were performed in accordance with NACE standard TM0282-96, solution A (pH =3) on base material and weld seam. Both approaches fulfilled the requirements for HIC resistance as can be seen in table X. The niobium vanadium approach was again more favourable.

Specification I	Requirements	NbT	Гi	NbV		
Test Condition	Acceptance Criteria	Base Meta	Weld Meta	Base Meta	Weld Metal	
pH3 1bar H₂S	CTR <u>≤</u> 5% CLR <u>≤</u> 15% CSR ≤ 1,5%	≤ 3% ≤ 8% ≤ 1%	≤ 5% ≤ 7% ≤ 1%	≤ 2% ≤ 7% ≤ 1%	≤ 4% ≤ 6% ≤ 1%	

Table X HIC test results of X70 pipe
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With the steadily increasing demands it becomes more difficult to fulfill the standard requirements for HIC requirements according to the above mentioned standard test conditions.

In those cases where these requirements can not be consistently achieved, fit-for-purpose testing methods are currently emerging.

The example given below concerns an inquiry for a linepipe of 42" diameter with 31.5mm wall thickness in grade X65 or, if practicable, in grade X70 and HIC requirements. A lean chemical composition that is typically used for the production of linepipe intended for sour service could not be used here because of the heavy wall. The chemical composition was optimized mainly to fulfill the requirements for mechanical properties. Attention was paid to each production step with a view to improving HIC resistance of the steel. Different HIC test variants were tried to work out a procedure that is close to the predicted service conditions and that is not difficult to implement. Figure 24 shows the behaviour of the 31.5mm thick grade X65 material, produced on a trial basis, in the various HIC test variants. The HIC index shown on the Y-axis is a measure of the extent of cracking. The higher the value of the index, the larger is the extent of cracking. The pH of the test solution was 3. The first row beneath the bars indicates the types of specimen used. Standard HIC specimens and large plate specimens were tested. The plate specimens were hydrogen charged by placing a glass tube on the specimen and filling it with the test solution. The second row beneath the bars shows the number of specimen sides exposed to the test solution. Only one-sided hydrogen charging was used in the case of plate specimens. The third row shows the partial pressure of H<sub>2</sub>S in the  $H_2S + N_2$  gas mixture with which the test solution was saturated at atmospheric pressure. The figure clearly demonstrates the effect of test conditions on the HIC index.



Figure 24: HIC behaviour of X65 steel designed for "slightly sour service".

To be on the safer side, the order was executed in grade X65 and the HIC tests were carried out using 1-side exposure in pH3-solution, but with a  $H_2S$  partial pressure of 0.1 bar.

About 200,000 tonne of pipe was produced for this order. The pipe's diameter produced was 42" with 29.8mm or 37.9mm wall thickness. Table XI shows the mean chemical composition and the mean mechanical properties determined on the 37.9mm wall thickness pipe.

	Mechanical properties			Mea	an		Sta	anda	arddevia	tion		
	Yield strengthR <sup>₀.5</sup> [MPa] Transverse Longitudinal				504 514				21 16			
٦	Tensile strength R <sup>m</sup> [MPa] Transverse Longitudinal				607 596				18 16			
	Yield-to-tensile ratio R <sup>10.5</sup> /R <sup>m</sup> [%] Transverse Longitudinal				83 87				2.1 1.6			
E	ElongationA2" [%] Transverse				22.1 22.8				1.27 1.67			
(	CVN toughnessat -30°C Weld metal [J] HAZ [J] Base metal [J]				11 7 26	0 6 6				37 37 35		
C Si Mn		Mn		Р	S		AI		Cu	Cr		
Mean comp.	.078	.20	1.60	.0	80	.00	08	.005		.13	-	
	Ni	Mo	N	)	Т	ï		Ν		IIW	PCN	
Mean comp.	.27	-	.03	5	.017 .00		004		.37	.18		

Table XI: Production results on 42" diam. X65 linepipe intended for "slightly sour service" with 37.9 mm wt

Compared to a conventional grade X65 material intended for sour service, the present steel had a higher carbon content and a higher manganese content to ensure that the steel should attain the required mechanical properties, particularly the toughness, at the heavy wall involved. The steel was made to the standard sour service practice, including vacuum degassing. It was desulphurised to a very low sulphur level of 8 ppm average and calcium-treated. This practice was adopted to ensure that the steel met the requirements for HIC resistance.

As can be seen from the mean values and the standard deviations given in Table XI, the pipe could be produced with a high statistical confidence level, despite its heavy wall. Both the transverse and longitudinal tensile properties of the pipe are comfortably above those required for grade X65. The Charpy V-notch impact energy values measured on the base material at  $-30^{\circ}$ C are in excess of 250J.

The HIC specimens tested to the fit-for-purpose procedure showed no ultrasonic indications, thereby complying with the specification.

Thus, the steel chemistry selected and the steelmaking practice adopted have proven the right approach to execute this special order successfully.

### **Clad Linepipe**

The use of high alloy materials for the linepipe often represents the only solution to prevent corrosion in situations where inhibition and gas processing is not practicable. Clad pipe combines the excellent corrosion behaviour of high-alloy austenitic materials such as Incoloy 825 and the high strength of carbon steels to an economic solution. Therefore a technology of making clad pipe from clad plate, which can be produced by sandwich-type rolling or by rolling of explosion-clad slabs, was developed. In both cases, a metallurgical bond between the substrate and the cladding is ensured.

In rolling a clad slab into plate, the thermomechanical rolling parameters adopted play an important role in achieving the strength properties of the substrate material and the corrosion resistance of the clad material (17). Thermomechanical rolling coupled with accelerated cooling is performed in two temperature ranges. The temperature of the first rolling stage is in a range just above the austenite recrystallisation regime. Heavy rolling passes, leading to a total reduction of 60% are essential for achieving a fine grained austenitic structure. The finish rolling is performed above Ar<sub>3</sub> temperature (about 800°C) in the austenite non-recrystallisation range to develop a fine grained transformation product. Additional niobium retards the recrystallisation of the austenite. The temperature range between 800 and 500°C must be passed through very fast with the help of accelerated cooling to avoid the precipitation of any phases that sensitise the cladding material and to achieve high strength and toughness properties for the base material. Figure 25 gives a rough summary of some details of the The two-pass weld is performed by the multi-wire submerged-arc longitudinal seam weld. welding process. Dilution of the inside weld from high-alloy cladding material is prevented by adopting a special groove geometry and controlling the heat input.

Pipe material						
Base material	Microalloyed C-Mn steel (X 65)					
Cladding material	Incoloy 825					

Details of welding							
Be sequ	ence	Welding process	Welding consu - mables				
Pass	1 and 2	Multi- wire SAW	Mn- NiMo or Mn-Mo (wire)				
no.	3	Electro - slag welding	Inconel 625 (strip)				



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Figure 25: Welding consumables and typical build up of the longitudinal weld seam in clad linepipe (24" diam. with 20.5 mm wt).

Table XII shows production results on Incoloy 825 clad X65 linepipe (24" diameter x 20.5mm wall thickness). The substrate steel (X65) has a very good toughness and a low transition temperature in the DWT test. The pipe was welded from inside using the electro-slag strip welding process which leads to very little dilution from the parent material. The niobium content of the substrate pipe was increased to 0.08% to ensure adequate strength values after forming and welding.

Table XII: Chemical composition and mechanical properties of large diameter clad linepipe X65 / Incoloy 825 (24" diam. with 20.5 mm wt)

and cladding material (B), wt %										
	С	Si	Mn	Cu	Ni	Cr	Мо	V	Nb	Ti
A*)	0.03	0.11	1.50	0.05	0.27	0.06	0.01	0.00	0.08	0.01
B	0.01	0.24	0.81	1.40	39.8	23.4	3.40	0.05	0.02	0.73
*) DOM = 0.40%										

#### a) Chemical compositions of base material (A) and cladding material (B), wt %

\*) PCM = 0.16%

# b) mechanical properties of base material (transverse specimens)

Yield strength (R <sub>p</sub> )	490MPa
Tensile strength (R <sub>m</sub> )	590MPa
Yield-to-tensile ratio $(R_p/R_m)$	83%
Elongation (A <sub>2"</sub> )	42%
Toughnes (CVN, -30°C or -22°F)	330 J
DWT (85% SA)	< -20°C
Shear stress (ASTMA 265)	460 MPa

### Conclusion

The predicted growth in energy consumption in the coming decades necessitates severe efforts for transporting large amounts of natural gas to the end user. Large-diameter pipelines are the best and safest means of transport. This paper gives an overview of the current requirements of high strength steels, heavy wall pipe for deep sea application, HIC-resistant materials and clad pipe and the associated developments. The technical possibilities were described. Also for the future additional substantial improvements can be expected.

The high cost of natural gas forces the pipeline operator to explore all the possibilities of reducing the cost of a pipeline project in the future. The pipe manufacturer can assist by supplying high quality pipe. The effect of pipe quality on project costs will be more substantial when the pipeline is constructed to the limit state design. For an offshore pipeline in the North Sea, the project team could reduce the project costs especially for transportation in several stages, by optimising the pipe diameter, giving a cost reduction of 5%, increasing the material grade, giving a cost reduction of 4%, and using of limit state design, giving a cost reduction of 6%.

Finally, the pipe manufacturer contributes to reducing operational costs of a pipeline over its life by investigating the fatigue, corrosion and ageing behaviour of pipe and pipe materials. These properties have a significant bearing on the integrity of a pipeline and consequently to the operating costs. These properties are currently being extensively studied. The knowledge gained from these studies can be made available to assist the pipeline operator when planning a new pipeline project or when estimating the residual life of an ageing pipeline.

For reasons of technical feasibility and cost-effective production, it is necessary in the future to reassess and redefine some of the materials requirements for pipe, considering the expected service conditions for the pipe. Close co-operation between the pipeline designer, operator, laying contractor and the pipe manufacturer is necessary for finding good solutions in modern pipeline construction.

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