

## ■ P R O C E S S I ■

# Development of high grade seamless pipes for deepwater application by metallurgical design

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*New solutions for the metallurgical design of high performance quenched and tempered (Q&T) seamless pipes of high grades from X65 up to X80 were found throughout a systematic work involving metallurgical modelling, laboratory tests, pilot and industrial trials. Both linepipes and risers for deepwater offshore fields such as Gulf of Mexico were considered. The target microstructure in the as-quenched condition has been identified as refined low-C bainite/martensite matrix (> 70%). This is promoted through the control of austenite grain growth during the heating stage (austenitization before quenching), proper alloy additions and through a very effective quenching. Promising low-alloy steels and suitable quenching and tempering conditions identified by metallurgical modelling were verified by laboratory heats (80 kg ingots) that were processed at a pilot scale and submitted to microstructural examination and mechanical testing. The best solutions were used in preliminary industrial trials, also utilised for a fine tuning. The Ni-Cr-Mo-Nb-V alloy system showed very interesting combinations of strength-toughness and field weldability, suitable for the production of heavy wall X65 grade linepipes for sour service and X80 top tension risers.*

**Key words:** Seamless pipe, linepipe, riser, strength, toughness, microstructure, quenching, tempering, metallurgical modelling

## INTRODUCTION

The technological evolution in the offshore sector exhibits a trend towards an increasing use of high strength steels (grade X65 to X80, and higher) both for risers and flowlines, although the service conditions and the performance required for the two systems are different. This trend is supported both by economical and technical reasons, because the development of deepwater oil and gas reserves is continuously facing the challenge of containing/reducing costs in all components. [1-2]

For instance, riser system costs are quite sensitive to water depth and there is a need to explore new technical solutions and reduce riser weight for ultra-deep water environments (greater than 2000 m). The use of high strength steels can decrease the wall thickness up to 30%, resulting in a more efficient design. Thinner wall risers mean reduced buoyancy requirements and less hydrodynamic loading on these components, with consequent improvement in riser response. For large field developments employing floating production facilities, with many heavy risers attached directly to the surface structure, payload limitations will receive higher consideration. Therefore, in this context the availability of higher-grade weldable steel risers, with a wall thickness to outside diameter ratio (WT/OD) adequate to the expected collapse performance is of engineering importance.

On the other hand, flowline wall thickness is increasing to provide sufficient resistance for the very high operating pressures. The trend in flowline specifications for deepwater

offshore fields is a consequence of both complex oil-gas field conditions, such as high pressure and high temperature (HPHT) and developments in design criteria (i.e. limit state design), welding and laying technologies. Often the requirements are close to the manufacturing limit of welded pipes, therefore seamless pipes that allow a higher (WT/OD) are preferred.

As a matter of fact pipe manufacturers are facing new challenges coming from new and/or more demanding material requirements, often related to specific performances and applications, including sour service which set limitations such as maximum material hardness ( $HV \leq 248$ ).

During the past years technologies have been developed in the field of quenched and tempered (Q&T) seamless pipe. In particular, the heat treatment capabilities of heavy wall pipes have been improved through the introduction of external and internal water quenching, which decreases the through-thickness temperature gradient.

Modern seamless pipes can combine high strength (grade X65) with good toughness properties and good girth weldability. For instance, in the case of pipes with  $WT = 15$  to  $34$  mm, a reasonable balance between customer specifications, processing capabilities and product properties was found for low-C low alloy steels with 1%Mn and optimized contents of Mo, Nb and V. Such Q&T seamless pipes, which also showed good resistance to strain aging and HIC, were successfully delivered for HPHT offshore production lines. [3] However, for pipe wall thickness greater than  $34$  mm, the required strength (grade X65) cannot be easily achieved maintaining the required toughness level. Similar difficulties are experienced in the case of  $WT = 15$ - $25$  mm for higher strength levels (e.g. grade X80). Therefore, new solutions which are outside of the conventional pattern for (micro)-alloying additions followed so far for Q&T seamless pipes, have to be found for high performance seamless pipes throughout a more systematic work.

In this paper, a description of the results of studies on high strength steel materials manufactured by Q&T processing is given. This work represents an on going development pro-

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gram on high performance Q&T seamless pipes for special deep water applications, involving metallurgical modeling, laboratory tests, pilot and industrial trials. The role of chemical composition and Q&T process conditions on microstructure and precipitation has been investigated, together with their effect on strength and toughness. These results have been exploited for the production of sour service grade API 5L X65/X70 for heavy wall flowlines (WT from 30 up to 42 mm) and X80 risers (WT = 16 to 25 mm) for deepwater offshore fields.

**METALLURGICAL BACKGROUND AND MODELLING**

Seamless pipes of medium O.D., i.e. up to 406 mm (16") are presently produced by a hot rolling process carried out in the following main stages: hot piercing, rolling at retained mandrel mill and sizing. Quenching and tempering treatments are performed on the pipes in order to refine the microstructure and obtain the required properties. [3,4]

A rational approach to the design and production of these materials requires the quantitative knowledge of the effects of steel chemistry and heat treatment variables on the microstructure and final mechanical properties. The influence of microalloying additions and Q&T practices on austenite refinement, phase transformation and response to heat treatments of low-C steels for seamless linepipes was investigated by dilatometry and pilot trials.

Also an integrated model, containing a thermal routine for simulating pipe quenching, based on the integration by finite differences of the general Fourier heat equation, coupled with a microstructural model, has been applied for the design of both the chemical composition and Q&T conditions of seamless pipes. The thermal-metallurgical model is able to calculate the fraction of microstructural constituents and hardness of a steel subjected to rapid continuous cooling after austenitisation (i.e. quenching). The calculation is carried out by an Artificial Neural Network (ANN) trained on a selected database of CCT diagrams of linepipe steels. [5,6] ANN is a powerful tool to obtain empirical non-linear models of complex phenomena whose analysis by ordinary statistical techniques or by modeling through fundamental physical process is not possible or very difficult. [7]

The program is able also to simulate a subsequent tempering treatment, predicting hardness (HV), yield strength (YS) and ultimate tensile strength (UTS) by an empirical approach. Different modules, each one describing an elementary process (e.g. austenitizing, quenching, tempering) can be managed by a user-friendly interface which allows to select the steel chemical composition, pipe diameter and wall thickness, and to set-up the process conditions. [5]

**Austenitizing**

The austenite grain size (AGS) depends on the austenitizing temperature and holding time, nature and size distribution of precipitates present in the as-rolled pipe. The more uniform the as-rolled microstructure, the easier it would be to homogenize the austenite.

Laboratory tests and industrial trials have shown that to avoid the formation of coarse austenite grains (AGS > 25 μm) in low carbon steels (0.08-0.11%C) the heating temperature has to be lower than 900 °C for C-Mn steels; however, V-Nb steels and V-Ti steels can be safely austenitized up to 920 to 950 °C to dissolve V-rich precipitates, without problems due to the pinning effect of Nb (C, N) and Ti (C, N) fine particles which hinder grain boundary movement.

**Quenching**

Of concern in a quenching process of seamless pipes are the effects of through-thickness cooling rate gradients, induced

by surface water cooling, and the sequence of transformation and the resultant microstructure and hardness profile. A specific test program was performed with the main objective of measuring the phase transformation characteristics of austenite under continuous cooling conditions by dilatometry and metallography (construction of CCT diagrams). Mathematical modeling, which links the basic principles of heat transfer and microstructural phenomena was effectively applied in this field.

The volume fraction of microstructural constituents and hardness of as-quenched linepipes were predicted as a function of the local cooling rate, calculated by the thermal model, by means of ANN.

Input data of ANN are the chemical composition of the steel, the austenitizing temperature, the austenite grain size and the cooling rate (CR). The output contains information on the as-quenched state of the steel in terms of hardness and amount of microstructural constituents (e.g. ferrite, pearlite, bainite, martensite). The ANN was trained on a huge database of continuous cooling diagrams of Nb-V micro-alloyed linepipe steels (C=0.05-0.2%, Mn=0.5-2.0%), with possible Mo, Ni, and Cr alloy additions. Standard deviations of the regression lines between computed and experimental fractions were generally below 5%. The standard deviation for the hardness was 11 Vickers. In all cases the associated correlation coefficient was greater than 0.91. [5,6]

In order to check the prediction capability of ANN, new experimental CCT diagrams, not used for training, were determined on a Nb-V-microalloyed pipeline steel. Two diagrams were determined by austenitising the steel for 10 minutes at 930 and 1100 °C in order to obtain AGS of 9 and 40 μm, respectively. Hardness of the as-quenched samples were measured and the related microstructures analysed by optical microscope to evaluate the volume fractions of the microstructural constituents. Experimental data were compared to the predictions of the microstructural ANN model. The good results are presented in Figures 1 and 2.

Other experiments, including quenching of pipes by various industrial devices, were carried out. Both pipes instrumented by thermocouples and through-thickness hardness profiles on as-quenched pipes were employed to tune the heat transfer coefficient as a function of process conditions (e.g. water flow, etc.).

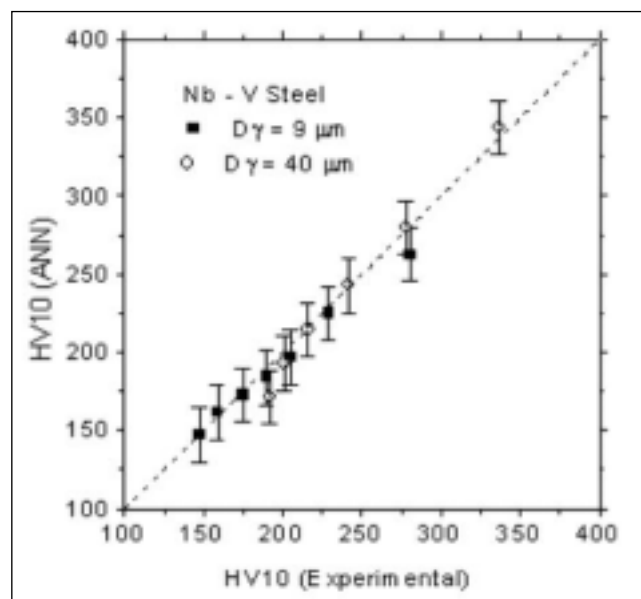
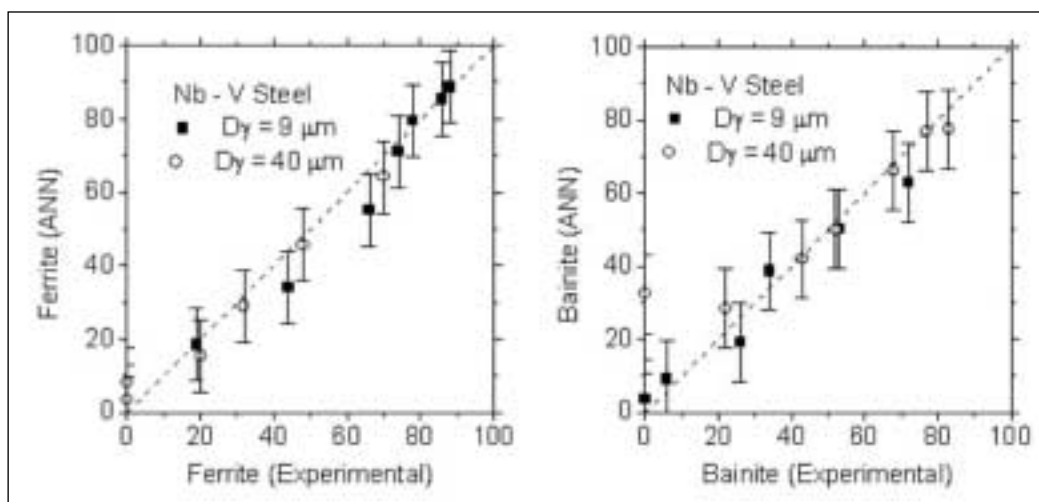


Fig. 1 – Comparison between the ANN predictions and the experimental data for hardness in a Nb-V linepipe steel.

Fig. 1 – Confronto tra previsioni con la rete artificiale neuronale e dati sperimentali sulla durezza di acciai al Nb-V per linepipe.

Fig. 2 – Comparison between the ANN predictions and the experimental data for ferrite and bainite volume fractions in a Nb-V linepipe steel.

Fig. 2 – Confronto tra previsioni con la rete artificiale neuronale e dati sperimentali sulla percentuale di ferrite e bainite in acciai al Nb-V per linepipe.



Simulations carried out by the model and industrial trials showed that linepipes with wall thickness less than 16 mm can be effectively quenched passing continuously through water jets produced by nozzles arranged in a series of rings forming a tunnel. In the case of heavy wall pipes, external and internal quenching is needed to reduce hardness gradients and make more homogeneous the as-quenched structure. This type of quenching is carried out by dipping the pipe in a tank containing stirred water. During quenching the pipe is under rotation and an internal water jet is used, too.

**Tempering**

The tempering conditions, mainly temperature and the presence of elements able to give precipitation hardening and to slow the recovery/recrystallization process, are the controlling factors for the combination of strength and toughness, for a given as-quenched microstructure.

The integrated model system is also able to simulate a subsequent tempering treatment of the microstructure after quenching. In this case an empirical approach was used, which permits the estimation of the final hardness taking into account also the effect of secondary hardening phenomena due to precipitation of second phases in steels containing V and/or Mo. The yield strength (YS) and the ultimate tensile strength (UTS) of the Q&T material were estimated from hardness using empirical equations. [6]

A comparison of calculated and experimental strength values of as-quenched industrial specimens submitted to tempering under very well controlled conditions in the laboratory are shown in Table I.

**Sensitivity Analysis**

A sensitivity analysis was performed on a reference steel,

Table I – Comparison between predicted and experimental strength for a Nb-V linepipe steel submitted to tempering at various temperatures for 60 min after industrial quenching (WT = 22 mm).

Tabella I – Confronto tra resistenza meccanica misurata e quella calcolata per tubi in acciai al Nb-V per linepipe rinvenuti a varie temperature per 60 min dopo tempra industriale. (spessore 22 mm).

Tempering Temperature (°C)	HV		YS (MPa)		UTS (MPa)	
	Calc	Exp	Calc	Exp	Calc	Exp
620	229	229	617	605	695	706
640	219	225	585	576	670	681
660	210	221	558	586	647	665
680	202	217	530	561	624	637
700	194	209	503	516	603	615

Table II – Chemical composition (mass %) of the reference steel used in the sensitivity analysis.

Tabella II – Analisi chimica (% in massa) dell'acciaio di riferimento usato per l'analisi di sensibilità.

CMn	Si	Ni	Cr	Mo	P	S	Cu	V	Al	Nb
0.12	1.2	0.3	0.1	0.15	0.07	0.01	0.004	0.15	0.05	0.025

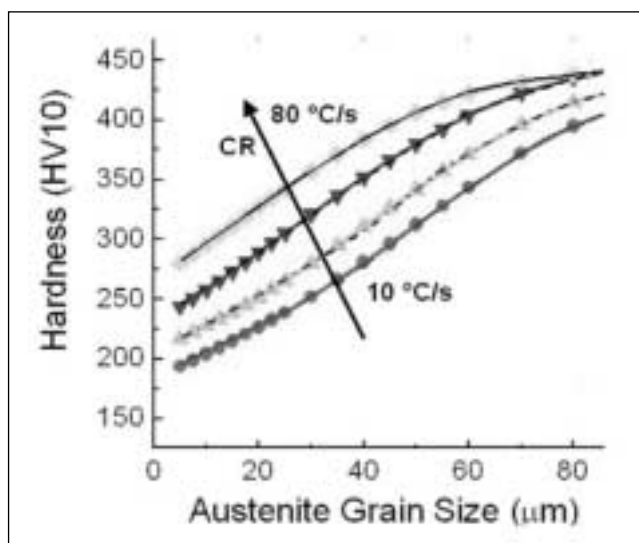


Fig. 3 – Calculated effect of AGS and cooling rate during quenching on hardness.

Fig. 3 – Effetto della dimensione media del grano austenitico e della velocità di raffreddamento durante tempra sulla durezza. Valori calcolati da modello.

having the chemical composition shown in Table II, in order to identify the role of chemical composition and process conditions on the phase transformation and response to tempering.

The well known effect of the austenite grain size in enhancing the hardenability of the steel clearly appears from Fig. 3. Hardness increases monotonically with AGS and with coo-

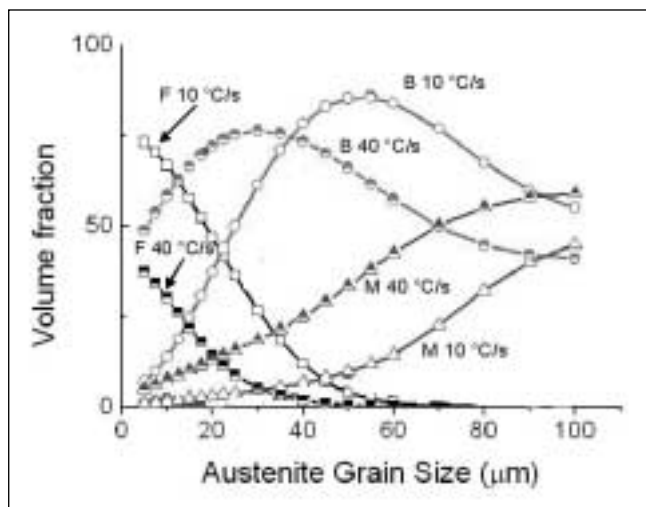


Fig. 4 – Calculated effect of AGS and cooling rate during quenching on microstructural constituents.

Fig. 4 – Effetto della dimensione media del grano austenitico e della velocità di raffreddamento durante tempra sulle frazioni dei costituenti microstrutturali. Valori calcolati da modello.

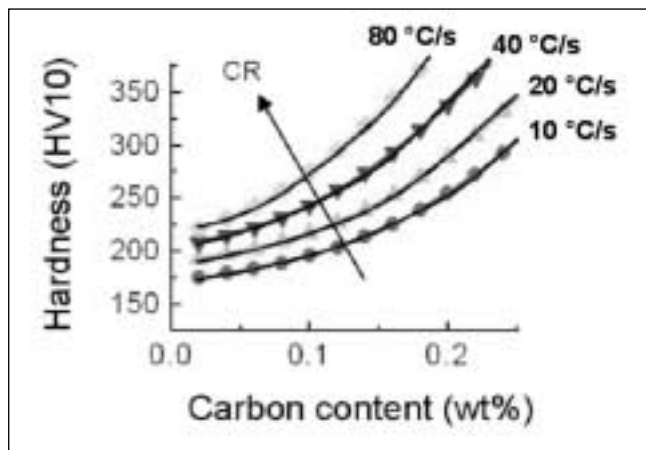


Fig. 5 – Calculated effect of carbon content and cooling rate during quenching on hardness.

Fig. 5 – Effetto del tenore di carbonio e della velocità di raffreddamento durante tempra sulla durezza. Valori calcolati da modello.

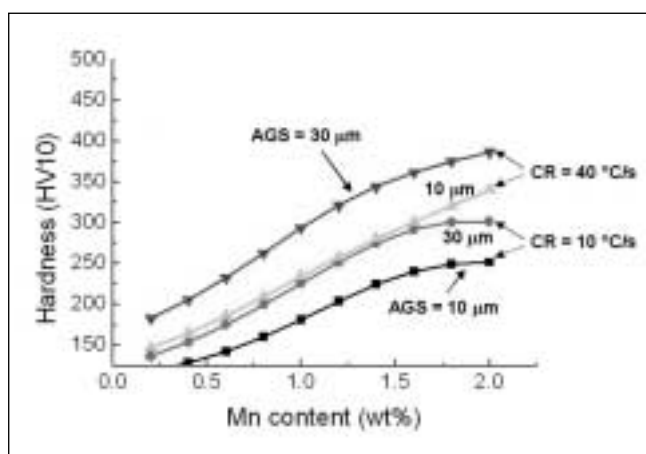


Fig. 6 – Calculated effect of manganese content and cooling rate during quenching on hardness.

Fig. 6 – Effetto del tenore di manganese e della velocità di raffreddamento durante tempra sulla durezza. Valori calcolati da modello.

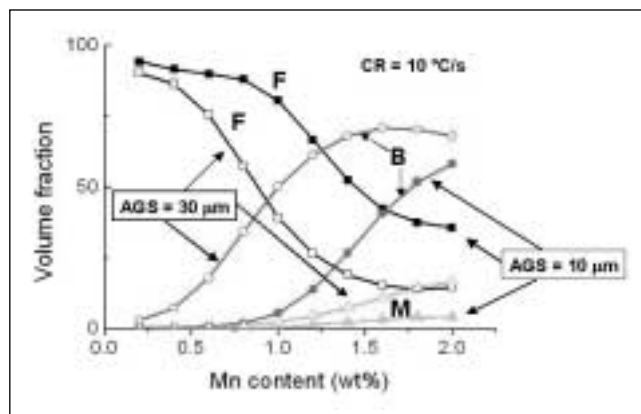


Fig. 7 – Calculated effect of manganese content and AGS during cooling at 10 °C/s on microstructural constituents.

Fig. 7 – Effetto del tenore di manganese e della dimensione media del grano austenitico sulle frazioni dei costituenti microstrutturali formati per velocità di raffreddamento di 10 °C/s. Valori calcolati da modello.

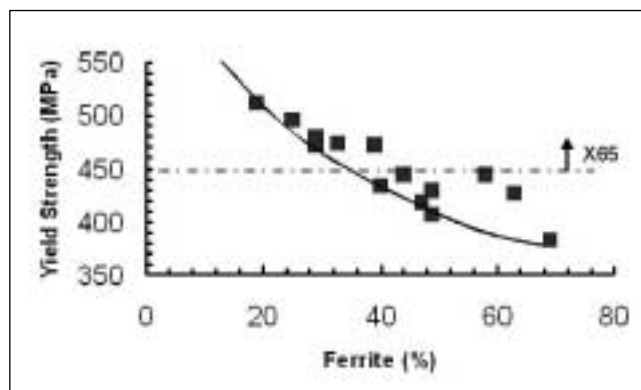


Fig. 8 – Calculated effect of ferrite amount on YS of Q&T materials (tempering at 660 °C for 60 min).

Fig. 8 – Effetto della percentuale di ferrite sulla tensione di snervamento di materiali temprati e rinvenuti a 660 °C per 60 min. Valori calcolati da modello.

ling rate, showing a saturation characterized by a stabilization in the amounts of bainite and martensite in the as-quenched microstructure with coarse grain (Fig.4). At the same time, as expected, ferrite appears to be present at low cooling rates and small AGS and to decrease as both cooling rate and AGS increase. One can observe that the bainite fraction goes through a maximum due to competition with ferrite at small AGS and martensite in the large grain region (Fig.4).

It can be noticed that the ANN has been extrapolated in the range of grain sizes from 50 to 100 mm with respect to the training interval for AGS. Apparently this has not caused any saturation effect in the output parameters.

Also the increase of carbon content (Fig.5) has shown a similar effect on hardness as that exhibited by AGS, in agreement, also in this case, with the experience.

In the third example the effect of Mn is considered for different values of AGS and cooling rate (Fig.6). The hardening effect of Mn is associated with the promotion of bainitic structures and this behavior is favored by large grains (Fig.7).

The application of the mathematical model indicates that to attain the required yield strength for grade X65 in the case of 40 mm (i.e. CR of 12-15 °C/s) it is necessary to have a fraction of polygonal ferrite below 30% (Fig.8).

The model is a very powerful and easy-to-use tool for designing the industrial Q&T processes taking into account all

the relevant process parameters. It has shown a very good reliability in reproducing the experimental data not only on the microstructure, but also on mechanical properties. For Q&T seamless linepipes, it is able to quantify both the through-thickness thermal gradients during the quenching process together with the related local microstructures and hardness, and the effect of subsequent tempering treatments. The model is an effective tool in defining the optimum Q&T treatments for a given steel to match the required tensile properties of the final product. However, no information on toughness is available from modelling. Therefore, a specific experimental activity has been designed and carried out to assess the effect of microstructure and precipitation on strength-toughness combination, starting from a promising chemical composition identified by metallurgical modelling.

HEAVY WALL HIGH STRENGTH FLOWLINES

Pilot Trials

A series of laboratory heats, with designed changes in the content of Mo, Ni, Cr, V and C, with respect to a base steel composition, were vacuum cast as 80 kg ingots. The carbon equivalent, Ceq (IIW) was in the range 0.33% to 0.43% and maximum Pcm parameter was 0.25%.

The ingots were hot rolled by a pilot mill simulating the typical thermo-mechanical process of heavy wall seamless pipes (40 mm final thickness). All ingots were instrumented with thermocouples and the evolution of temperature during hot rolling was recorded.

The hot rolled materials were quenched in stirred water and tempered under strictly controlled parameters (Table III), being each piece instrumented by a thermocouple embedded at mid-thickness.

	Min	Max
Austenitizing Temperature (°C)	920	1020
Cooling rate during quenching (°C/s)	10	25
Tempering Temperature (°C)	630	690

Table III – Range of laboratory heat treatment conditions.

Tabella III – Intervallo esplorato per le condizioni di trattamento termico in laboratorio.

The Q&T materials were examined by light and scanning electron microscopy. Microstructures were observed on sections after 2%-nitric etching. Islands of high carbon martensite with retained austenite (MA constituent) were revealed by selective etching. [8]

The austenite grain boundaries were revealed by etching in a saturated aqueous picric acid solution containing a few drops of teopol and HCl.

The average austenite grain size (AGS) was measured according with ASTM E112.

Tensile and Charpy V-notch testing was conducted on transverse specimens. Charpy-V transition curves were determined together with the fracture appearance transition temperature (50% FATT).

Effect of Alloy Design on Microstructure

It was confirmed, as suggested by model simulations that in order to achieve the required yield strength level after tempering it is a pre-requisite to maintain the fraction of polygonal ferrite well below 30% in the as-quenched material (Fig.9).

A judicious addition of Mo, Ni and Cr allows to develop after quenching a microstructure containing a suitable combination of constituents such as fine bainite (B > 75%), poly-

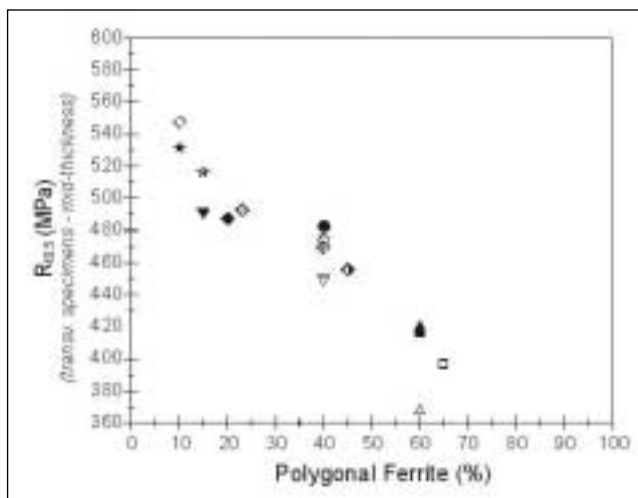


Fig. 9 – Strength vs polygonal ferrite amount for various laboratory Q&T steels.

Fig. 9 – Resistenza meccanica in funzione della percentuale di ferrite poligonale per vari acciai di laboratorio.

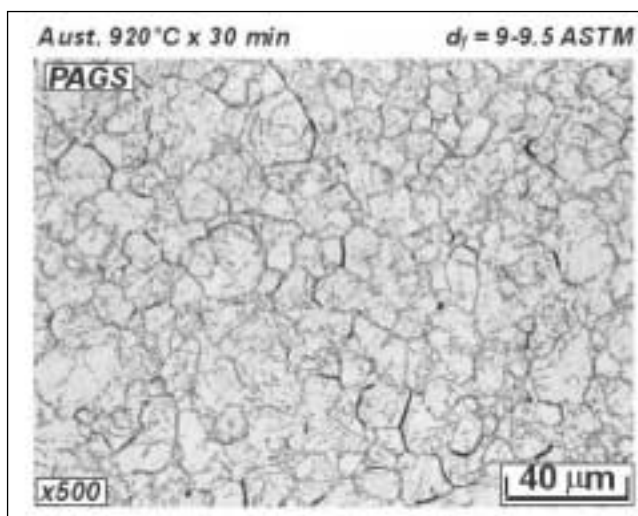


Fig. 10 – Mo-Ni-Nb-V Steel. AGS obtained with austenitizing temperature of 920°C and quenching with CR = 12°C/s. Prior austenite grain boundaries were revealed by selective etching by saturated picric solution.

Fig. 10 – Acciaio al Mo-Ni-Nb-V. Grano austenitico ottenuto dopo trattamento a 920 °C e tempra con velocità pari a 12°C/s. I bordi dei grani austenitici primitivi sono stati evidenziati mediante attacco metallografico selettivo mediante soluzione acquosa satura di acido picrico.

gonal ferrite (PF < 25%) and fine islands of MA constituent, uniformly dispersed in the matrix. This predominantly bainitic structure was found to exhibit good toughness values especially when the AGS was fine (< 15 mm; > ASTM No.9) and homogeneous.

An example of the typical AGS (ASTM No.9.2) of Mo-Ni-Nb-V steel, quenched from 920 °C, is reported in Fig.10. The addition of Nb slows down grain growth and helps to maintain relatively fine and homogeneous the AGS during austenitization.

In the case of fine AGS, the increase of the cooling rate from 12 °C/s to 20 °C/s, refines the microstructure (Fig.11).

Therefore, even better strength-toughness combinations are expected for linepipes with thickness smaller than 40 mm. Concerning the microstructure evolution during tempering, a better spheroidisation of cementite and a more extended transformation of MA islands into ferrite and carbides, was observed with increasing tempering temperature (Fig.12).

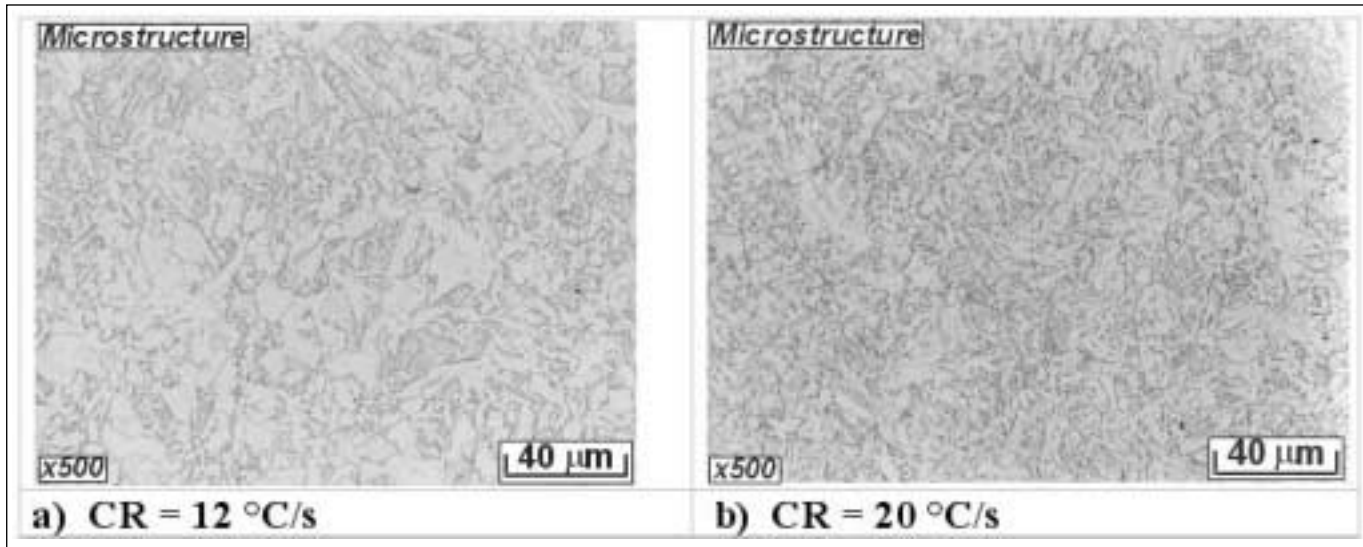


Fig. 11 – Mo-Ni-Nb-V Steel. Microstructure obtained after austenitizing at 920°C and quenching with CR=12°C/s and 20 °C/S, respectively. The as-quenched microstructure was revealed by 2% Nital etching. a) CR = 12 °C/s b) CR = 20 °C/s.

Fig. 11 – Acciaio al Mo-Ni-Nb-V. Microstruttura ottenuta dopo austenitizzazione a 920°C e tempra con velocità di raffreddamento di 12°C/s e 20 °C/S, rispettivamente. La microstruttura grezza di tempra è stata evidenziata con attacco al nital 2%.

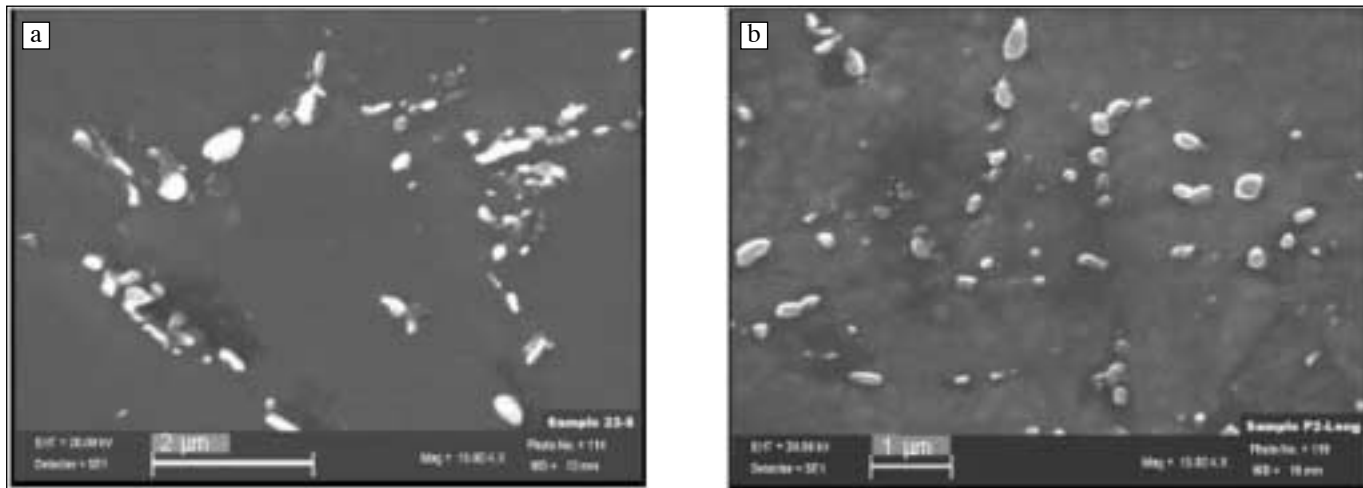


Fig. 12 – Cementite morphology after Q&T (SEM image). a) Low Tempering Temperature b) High Tempering Temperature.

Fig. 12 – Morfologia della cementite dopo tempra e rinvenimento (immagini al microscopio elettronico a scansione).

### Strength and Toughness Properties

The general pattern of strength/toughness properties as a function of (micro)-alloy design is reported in Fig.13. The Mo-Ni-Nb-V composition gives excellent strength/toughness combination: yield strength well above 460 MPa (grade X65); 50%FATT as low as – 85°C.

With reference to the same base composition and Q&T conditions of Fig.13:

- The increase of C content up to 0.13% gives strengthening (DYS = + 60 MPa), but is detrimental to toughness and weldability.
- The V-free version (Mo-Ni-Nb steel) allows to reduce FATT, but at expense of strength (DYS = – 35 MPa).
- Addition of Cr produces further improvement in toughness (50% FATT below – 100°C).

Therefore the Mo-Ni-Cr-Nb-V-steel resulted to be the most promising for the production of heavy wall linepipes.

The laboratory materials exhibit the general trend of increasing yield to tensile (Y/T) ratio when yield strength increases. For the required strength levels (YS > 450 MPa) the Y/T values are in the range 0.83 to 0.87, being the highest ratios related to a predominantly bainitic structure.

The alloy design based on the Mo-Ni-Cr-Nb-V version exhi-

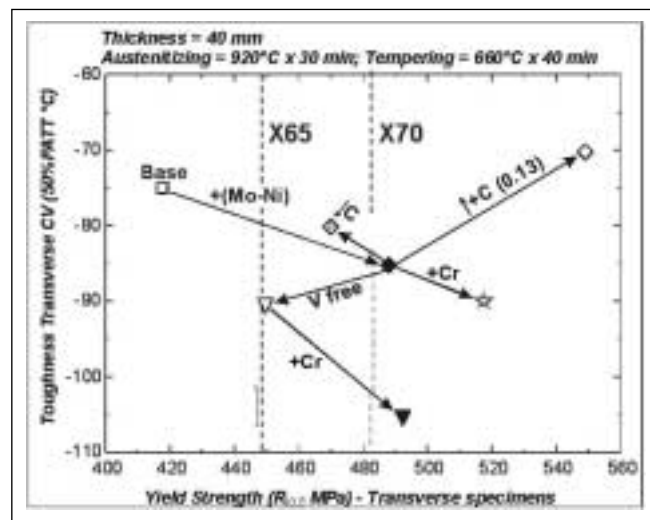


Fig. 13 – Toughness vs yield strength for various laboratory Q&T steels.

Fig. 13 – Tenacità in relazione alla tensione di snervamento per vari acciai di laboratorio temprati e rinvenuti.

bits the best results in terms of low values of Y/T ratio. Concerning toughness, when the as-quenched microstructure is fully bainitic and the volume fraction of MA is very low, the 50% FATT of Q&T material is practically independent of the tempering temperature, as expected. The increase of tempering temperature is effective in improving toughness if significant amounts of MA constituent, in the form of large islands, are present in the as-quench material. In this case, usually, the rising of tempering temperature from 630°C to 680°C leads to a decrease in yield strength (DYS = - 20 MPa) and a slight improvement of toughness in terms of 50% FATT (DFATT = - 10°C).

**Industrial Production and Pipe Qualification**

Heavy wall seamless pipes of medium diameter (OD = 219 to 323 mm) and WT = 30 to 40 mm were produced at Tenaris works by the seamless process, using the steel chemistry range and heat treatment conditions identified as promising by the metallurgical design. Water quenching was carried out by dipping the rotating pipe in a tank containing stirred water. Also an internal water jet is used to increase heat transfer at the inner surface.

All pipes were manufactured according to specific customer requirements for production risers and flowlines. In particular, in addition to weldability requirements, in terms of carbon equivalent, Ceq (IIW) and Pcm parameter, suitable tensile properties at room and at 130 °C shall be guaranteed (minimum yield strength = 448 MPa).

The characterization of selected pipes from the production was carried out by extensive metallography and mechanical testing, which included hardness measurements, longitudinal and transverse tensile testing, Charpy-V impact testing, crack tip opening displacement (CTOD) testing.

The industrial production confirmed the effect of main process parameters and metallurgical factors on microstructure and strength-toughness combination, as outlined by the laboratory experiments, and allowed to identify the actions for a fine tuning to develop a good combination of strength and toughness.

All hardness values on Q&T pipes were below 248 HV10. The mechanical properties of various industrial materials in terms of 50% FATT and yield strength are summarized in Fig.14.

The materials produced using the Mo-Ni-Cr-Nb-V steel, have significantly improved toughness for a given yield strength level between 460 and 530 MPa, compared to the conventional chemistry without Ni. The productions indicate that suitable toughness levels (i.e. 50% FATT < - 50°C) have been achieved. Also high CTOD values at - 10 °C (> 1.1 mm) were measured on the Q&T seamless pipes. Best results were attributed to the following aspects:

- strict control of process parameters during heating in order to develop uniform and small PAGS;
- effective quenching to promote higher volume fractions of bainite and a more refined final microstructure.

Suitable strength levels of pipes for flowlines and produc-

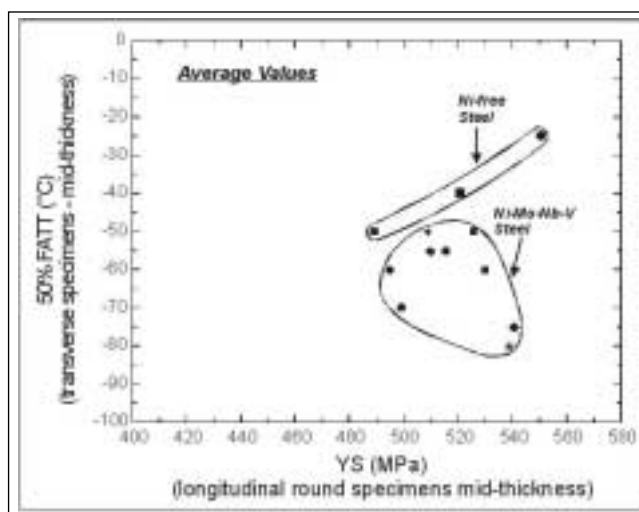


Fig. 14 – Toughness vs yield strength for heavy wall seamless pipes.

Fig. 14 – Tenacità in relazione alla tensione di snervamento per tubi senza saldatura di grosso spessore, per linepipe.

tion risers were also achieved at 130 °C.

Some pipes were girth welded by GMAW using low (0.6 kJ/mm) and high (3 kJ/mm) heat inputs. CTOD was also performed in the HAZ of girth welds performed by GMAW. CTOD specimens were located at both the Transformed-HAZ (THAZ) and Visible-HAZ boundary (VHAZ). Post-test validity checks were carried out by specimen sectioning followed by fractographic and metallographic evaluation. The CTOD results at 4 °C were quite good both for the THAZ (CGHAZ), with values of 0.6 - 1 mm and the VHAZ with values of 1 - 1.5 mm. At - 10 °C, CTOD remained always greater than 0.3 mm.

**HIGH STRENGTH RISERS (WT = 15 to 25 mm)**

Due to the higher cooling rates during quenching, these pipes can develop a predominantly bainitic-martensitic structure more easily. The tuning of Mn, Mo, Cr, V and Ni additions was done on the basis of the results from the metallurgical model and pilot trials (Table I).

A steel with carbon equivalent, Ceq° (IIW), in the range 0.37% to 0.40% and maximum Pcm parameter below 0.2% was selected.

The sizes and mechanical properties of the risers produced are shown in Table IV.

Hardness near the internal surface is usually higher because a high-pressure water jet blows the steam generated inside the pipe during the immersion in the water tank, promoting a higher heat transfer coefficient, thus increasing the severity of quenching. However, it is important to mention that suitable hardness can be obtained adjusting the tempering

Item	OD (mm)	WT (mm)	Ceq° (IIW)	HV max	Longitudinal *				Transverse **			
					YS (MPa)	UTS (MPa)	EI (%)	FAT T (°C)	YS (MPa)	UTS (MPa)	EI (%)	FAT T (°C)
1	323.9	16	0.38	243	589	677	42	- 60	596	672	25.4	- 50
2	298.5	22	0.40	248	594	678	41	- 50	602	681	25.0	- 40

\* full strip specimen; \*\* round specimen

Table IV – Sizes and average mechanical properties of the produced risers.

Tabella IV – Dimensioni dei riser prodotti e relative proprietà meccaniche.

treatment parameters. As all the hardness values were below 250 HV10, these risers can be used for sour service applications.

An interesting feature found in these pipes is that there is not a significant difference between the longitudinal and the transverse tensile properties at room temperature. This is due to the high isotropy which is typical of seamless pipes. The materials exhibited an absorbed energy value above 140 J up to  $-60^{\circ}\text{C}$ . This is a consequence of the microstructure promoted after external/internal quenching and tempering. Also high CTOD values ( $> 1.2\text{ mm}$ ) were measured at  $-20^{\circ}\text{C}$  on these high strength Q&T seamless pipes. Weldability trials proved that the selected chemical composition combined with proper welding procedure assures hardness values lower than 280 HV and satisfactory toughness levels both in the weld metal and heat affect zone.

### CONCLUSIONS

New chemistries and optimized Q&T conditions were identified for the production of seamless pipes for both flowlines with thickness up to 42 mm and risers of grade X80 and 15 to 25 mm WT, through an extensive characterization of laboratory and industrial materials. A number of selected alloy systems have been systematically investigated. Main conclusions are:

- The as-quenched microstructure plays a primary role. The best toughness values are related to a predominantly bainitic microstructure after quenching (bainite  $> 70\%$ ) combined with a homogeneous and fine distribution of MA constituent. This is promoted through the control of austenite grain growth during the heating stage and an effective quenching.

- The Mo-Ni-Cr-Nb-V alloy system showed the best combination of strength-toughness and weldability.
- The tempering temperature has a secondary role. However, higher tempering temperature leads always to a slightly lower yield strength and, in the case of coarse MA islands and cementite particles, a slightly improved toughness.
- The CTOD results in the HAZ of GMAW girth joints were good.

### REFERENCES

1. M. Cook, OPT USA 2002, Houston, 16-17 Sept. 2002.
2. U. Eigbe, OPT USA 2002, Houston, 16-17 Sept. 2002.4.
3. E. Anelli et al., 11th International Offshore and Polar Engineering Conference and 2nd International Deep Ocean Technology Symposium, Stavanger, Norway, June 17-22, 2001.
4. J.C. Gonzalez, M. Tivelli, H. Quintanilla, G. Cumino, Proc. 40th Mechanical Working and Steel Processing Conference, ISS, 1998, pp 621-626.
5. P.E. Di Nunzio, E. Anelli and G. Cumino, EUROMAT 2001 Int. Conf., Rimini, Italy, 10-14 June 2001, paper no. 546.
6. E. Anelli, M.C. Cesile, P.E. Di Nunzio, Int. Conf. "Modeling Control and optimization in Ferrous and non Ferrous industry", ed. F. Kongoli, B.G. Thomas, K. Sawamiphakdi, ISS & TMS, Chicago, 9-12 Nov. 2003, pp. 453-466.
7. Special Issue "Application of Neural Network Analysis in Materials Science", ISIJ Int., Vol.39, No.10, 1999, pp. 965-1105.
8. D. Colleluori, Pract. Met., Vol.20, 1983, pp. 546-553.

### A B S T R A C T

#### SVILUPPO METALLURGICO DI TUBI SENZA SALDATURA DI GRADO ELEVATO PER APPLICAZIONI IN ACQUE PROFONDE

**PAROLE CHIAVE:** Tubi senza saldatura, linepipe, riser, resistenza meccanica, tenacità, microstruttura, tempra, rinvenimento, modelli metallurgici

Attraverso un'indagine sistematica condotta impiegando sia la modellistica metallurgica sia sperimentazioni di laboratorio e prove su impianto pilota e in scala industriale, sono state identificate nuove soluzioni per la progettazione metallurgica di tubi senza saldatura temprati e rinvenuti (Q&T) ad elevate prestazioni ed alto grado (da X65 a X80). Un approccio razionale alla progettazione e alla produzione di questi materiali ha richiesto la conoscenza quantitativa degli effetti dell'analisi chimica dell'acciaio e dei parametri di trattamento termico sulla microstruttura e sulle proprietà meccaniche finali. L'influenza degli elementi di lega e delle condizioni di tempra e di rinvenimento sull'affinamento dell'austenite, sulle trasformazioni di fase e sulla risposta ai trattamenti termici è stata studiata mediante apposite sperimentazioni su acciai a basso tenore di carbonio impiegati nella fabbricazione di tubi senza saldatura per linepipe e riser. Inoltre, è stato validato un modello matematico integrato, costituito da un modulo termico per la simulazione numerica della tempra in acqua dei tubi basato sulla soluzione dell'equazione di Fourier del trasporto di calore e da un modello microstrutturale. Questo è stato applicato alla progettazione sia della composizione dell'acciaio sia del trattamento termico dei tubi senza saldatura. Il modello termico-metallurgico è in grado di calcolare la frazione dei costituenti microstrutturali e la durezza di tubi in acciai sottoposti ad un raffreddamento continuo rapido dopo austenitizzazione (tempra). Il calcolo della trasformazione di fase viene effettuato mediante una rete neurale artificiale (ANN), addestrata

su un database costituito da una selezione di diagrammi di trasformazione di fase in raffreddamento continuo (CCT) di acciai per linepipe. Il programma consente di simulare anche il successivo trattamento di rinvenimento, prevedendo, mediante un approccio empirico, durezza (HV), tensione di snervamento (YS) e carico unitario a rottura (UTS). Vari moduli, ognuno in grado di descrivere un processo elementare (ad es. austenitizzazione, tempra, rinvenimento), sono gestiti da un'interfaccia-utente di uso immediato che consente di selezionare l'analisi chimica, il diametro e lo spessore del tubo e di stabilire le condizioni di trattamento termico. L'applicazione della modellistica matematica ha mostrato che per ottenere la tensione di snervamento desiderata per il grado X65, nel caso di tubi di spessore 40 mm, è necessario sviluppare una percentuale di ferrite poligonale inferiore al 30% in volume. La microstruttura ottimale dopo tempra, costituita prevalentemente da bainite-martensite a basso carbonio, viene promossa da un controllo delle dimensioni dei grani austenitici durante l'austenitizzazione che precede la tempra, da aggiunte calibrate di elementi di lega e da un raffreddamento molto efficace. Attraverso l'applicazione del modello metallurgico sono state definite alcune composizioni promettenti di acciai bassolegati e le condizioni di tempra e rinvenimento più idonee. Le previsioni sono state verificate con colate di laboratorio (lingotti da 80 kg) processate su scala pilota, esaminate metallograficamente e valutate in termini di proprietà meccaniche con particolare attenzione alla tenacità. I materiali più promettenti sono stati impiegati per prove su scala industriale e per la calibrazione fine dei modelli matematici. Acciai al Ni-Cr-Mo-Nb-V hanno mostrato combinazioni di resistenza meccanica e tenacità molto interessanti per la produzione di tubi di grado API 5L X65/X70 per flowline di grosso spessore (da 30 a 40 mm) e riser (di spessore da 16 a 25 mm) di grado fino a X80, per lo sfruttamento di giacimenti in acque profonde.