

Development of High Strength Heavy Plate Optimised for Low-Temperature Toughness for Linepipe Applications

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Abstract
This paper gives an overview of the development strategy for high strength heavy plates and line pipes optimised for low temperature toughness for Salzgitter Mannesmann Grobblech and EUROPIPE. The complete material flow from steel to plate and pipe as well as all necessary R&D activities are available among the whole production chain which makes it possible to react directly to special customer demands.

INTRODUCTION

Salzgitter Mannesmann Grobblech GmbH (MGB), a member of the Salzgitter group, operates a 5.1-meter heavy plate rolling mill which is specialised on the production of plate for pipe. More than 90 percent of its more than 750,000 tons annual plate production is delivered to large diameter pipe mills all over the world. The most important and biggest customer is the German pipe producer EUROPIPE which is a joint venture of the two steel manufacturers Salzgitter and Dillinger Hütte. Forced by the market demand for high strength arctic linepipe steels MGB develops and improves ultra high strength heavy plates mainly for this customer. All important steps of this development take place integrated within companies owned or at least partly owned by the Salzgitter group: the steel is produced at Hüttenwerke Krupp Mannesmann (HKM), the plates are rolled at MGB, the pipes are manufactured at EUROPIPE, bends can be manufactured at MGB´s own pipe bending plant. Last but not least the whole research and development for this production chain is performed in cooperation with Salzgitter Mannesmann Forschung (SZMF) which is the development centre of the Salzgitter group.

The development of microalloyed steels for linepipe applications is a focus of research efforts at the SZMF. The research centre is equipped with the facilities to produce laboratory heats of up to 300 kg by vacuum induction melting and to perform thermomechanical rolling trials with accelerated cooling using two-high hot rolling mills. The typical weight of ingots used for heavy plate development is 100 kg. These ingots can then be divided into several coupons making it possible to investigate a broad range of compositions and processing parameters, in order to identify ideal rolling parameters for industrial scale rolling trials. An extensive database of results of rolling trials has been gathered, ranging up to the X120 strength level. The analysis of these data and of results of heavy plate production forms the basis for the optimisation and development of new processing strategies. This is complemented by numerical tools that are used for alloy design.

Since the beginning of the eighties of the 20^{th} century, plates, pipes and pipe bends of steel grade API X80 were developed and produced at Mannesmann and EUROPIPE. Today they are daily business for both companies. **Figure 1** gives an impression of the produced tonnage of heavy plates until 2007 in API Grade X80. In recent years, the complexity of requirements of line pipe materials has increased steadily with regard to toughness and weldability. For this reason, the X80 products were steadily improved by development along the entire process chain.

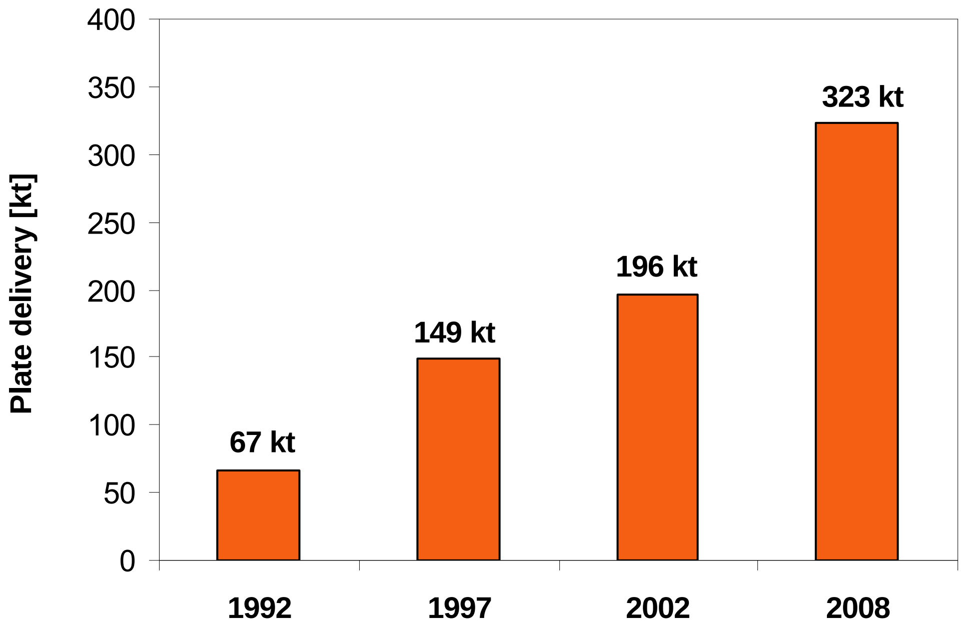

Figure 1: Development of X80 production at MGB GmbH

The use of high strength grades makes it possible to decrease the wall thickness of the pipe material or to increase the working pressure, thereby reducing the costs of pipeline construction and increasing the efficiency. For this reason, high strength grades are attractive especially for long-distance pipelines. Currently, there is a strong demand to develop remote gas fields in hostile environments such as arctic or permafrost regions. High deformability and low-temperature toughness are therefore critical requirements in order to ensure pipeline safety.

The strain-based design methodology has been developed as a guideline to ensure safe pipeline operation even under earth movement scenarios inducing longitudinal strains [1]. The heavy plate and pipe production processes for high strength steels has been optimised by producers in order to meet these strain-based design requirements [2]. The optimisation of the toughness has been another focus of development in order to ensure crack arrest at low temperatures. However, it has been shown that while existing criteria predict crack arrest reasonably well when compared with the results of full scale tests up to the X70 strength level, these do not appear safe in the case of high strength grades [3]. A DWTT total energy above around 800 J/cm^2 in combination with a high ratio of DWTT total energy and CVN energy was recently recommended as a design guideline for crack arrest [4]. However, more work is required to better understand the relationship between laboratory-scale and full scale tests of high strength grades.

Recently, a development program was carried out at the SZMF in cooperation with MGB with the aim to optimise the low-temperature toughness of the grade K65 (SMYS=555 MPa) by variation of the processing parameters and base metal composition. These consist of tensile test requirements in transverse and longitudinal direction, an average CVN energy at -40°C above 200 J and 85% minimum shear area in the DWT test at -20°C, as the pipe material is intended for use in an arctic environment. The carbon equivalent (IIW) was limited to a value of 0.45%. Laboratory heats were produced and thermomechanical rolling in combination with accelerated cooling was carried out at the SZMF, followed by production trials at MBG and EUROPIPE.

LABORATORY INVESTIGATIONS AT THE SZMF

Three 100 kg laboratory heats were produced by vacuum induction melting (**Table 1**). The criterion for the selection of the compositions was the maximum allowable carbon equivalent (IIW) of 0.45%. The levels of the additions of carbon, silicon and niobium were varied, while the other elements were held constant. In order to estimate the lower limit of the range of feasible reheating temperatures, thermodynamic calculations were carried out using Thermo-Calc [5] in combination with the TCFE5 database. In **Figure 2**, the volume fraction of phases is shown exemplarily for the composition Lab 1. Such calculations only predict the fraction of phases and their composition under equilibrium conditions. Due to segregation of elements during solidification, the alloy composition varies locally and the equilibrium is usually not reached. However, in the case of carbonitrides, it has been found that predictions correspond reasonably well with experimental observations [6]. In the example presented in **Figure 2**, the dissolution of Nb(C,N) is predicted at the indicated temperature. Due to the reduced carbon content of Lab 2, this temperature was shifted 20 K lower. In the case of Lab 3, the dissolution is predicted to take place 30 K higher.

	Composition [wt.%]						
	C	Si	Mn	others	Nb	Ti	CE_{IIW}
Lab 1	≥0.05	≤0.20	≥1.8	Cu, Ni	≤0.050	≤0.02	0.44
Lab 2	≤0.03	≥0.20	≥1.8	Cu, Ni	≥0.050	≤0.02	0.44
Lab 3	≥0.05	≥0.20	≥1.8	Cu, Ni	≥0.050	≤0.02	0.43

Table 1: Compositions of the investigated laboratory heats

In order to guarantee a high degree of dissolution of Nb(C,N) precipitates, the lower limit for the reheating temperatures was based on these calculations. Once the Nb(C,N) precipitates are dissolved, Ti(N,C) particles can inhibit grain coarsening because these are stable over the whole range of feasible reheating temperatures, as shown in **Figure 2**.

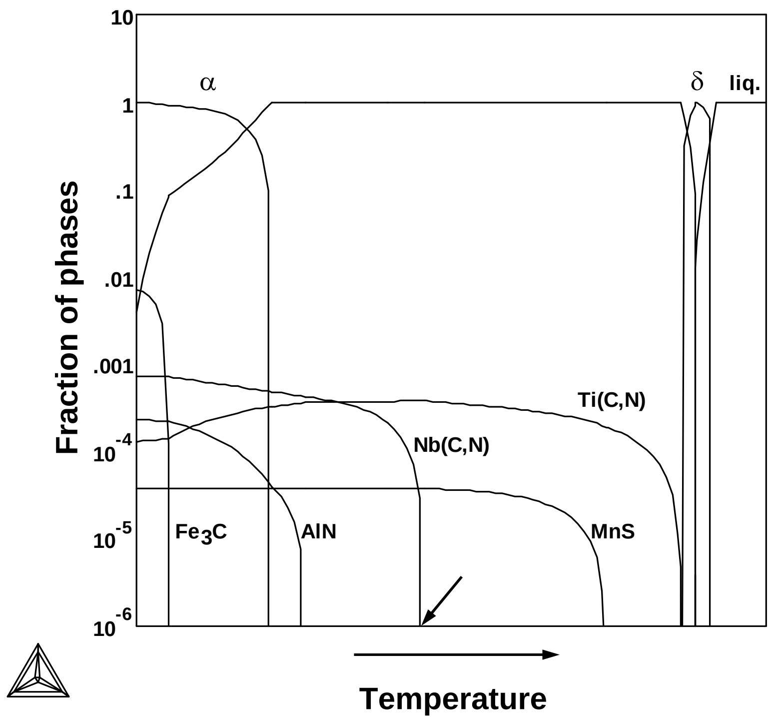

Figure 2: Fraction of phases as a function of temperature calculated for the composition Lab 1

Reheating at excessively high temperatures leads to grain coarsening [7], which can have a detrimental effect on the toughness of the product, because the grain size of the final product increases as well. On the other hand, it has been shown that increasing the reheating temperature can lead to an increase in the strength of the heavy plate [8]. Finding the right balance between the dissolution of Nb(C,N) and austenite grain coarsening during reheating is therefore important in order to achieve the desired combination of mechanical properties.

In one set of laboratory rolling trials on the heats Lab 1 and Lab 2, the reheating temperature (TR), the final rolling temperature (FRT) and the cooling stop temperature (TCS) were varied in order to investigate the effect on mechanical properties. TR-1 was selected slightly above the temperature of dissolution of Nb(C,N) and TR-2 was 100 K higher. The final rolling temperature was decreased from FRT-1 to FRT-2 by around 20 K. Both were selected well above the expected temperature of the onset of the austenite to ferrite transformation in order to ensure a fully austenitic structure prior to accelerated cooling. The cooling stop temperature decreased from TCS-1 to TCS-2 by roughly 50 K. In a second set of rolling trials, the effect of the final rolling temperature was investigated using the heat Lab 3. The first final rolling temperature used in the second trial was FRT-2, which was subsequently lowered by 20 K (FRT-3) and 40 K (FRT-4).

The plates were rolled on a two-high rolling mill to a final wall thickness of 27 mm followed by accelerated cooling. Materials testing comprised tensile tests of round bar specimens, Charpy impact tests and instrumented pressed-notch (PN) Battelle drop-weight-tear tests at -20°C in transverse direction using specimens with the full wall thickness. In the drop-weight-tear tests, the load-displacement curve was recorded in order to evaluate the energy consumed for fracture of the specimen. Apart from the shear area, the total energy was used as a measure of the potential for crack arrest. Which parameter best describes the potential for crack arrest is currently not completely understood, but a high total energy has been recommended [4].

Results of the laboratory rolling trials

The microstructure that was achieved in the rolling trials was in all cases predominantly bainitic with minor volume fractions of ferrite. An example of the microstructure is shown in **Figure 3**.

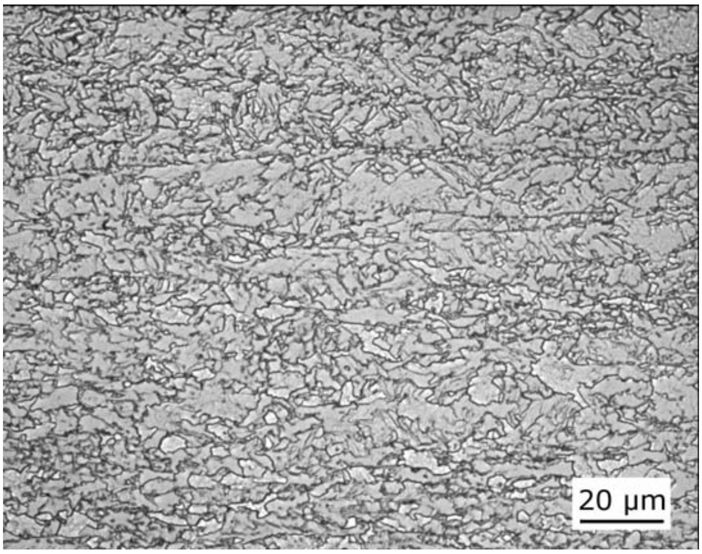

Figure 3: Example of the microstructure obtained by thermomechanical rolling and accelerated cooling of the heat Lab 1

The round bar tensile tests of the plates in transverse direction rolled using Lab 1 und Lab 2 (see **Figure 4**) showed that the tensile requirements were not fully fulfilled in the first set of trials. An increase in the reheating temperature from TR-1 to TR-2 leads to significant increase of the yield and tensile strength to a level above the tensile requirements for Lab 1, for which the increase was more pronounced compared to Lab 2. A decrease of the final rolling temperature from FRT-1 to FRT-2 led to a slight drop of the yield and tensile strength for both compositions. An increase in the cooling stop temperature from TCS-1 to TCS-2 did not have a severe affect on the tensile properties. This is related to the fact that the cooling stop temperature was well above the martensite-start-temperature in both cases.

The Charpy impact tests (see **Figure 5**) showed that an increase of the reheating temperature leads to a lower impact energy for both compositions, although the impact energy is still above the requirements shown in **Table 3** even at -60°C. The effect of the final rolling temperature on the impact energy could not be clarified conclusively in this set of experiments. A drop in the impact energy for both compositions was observed after an increase of the cooling stop temperature from TCS-1 to TCS-2.

Battelle drop-weight-tear tests were carried out in transverse direction using pressed-notch specimens with 27 mm wall thickness. The effect of the variation of the processing parameters on the shear area and total energy in the drop-weight-tear test at -20°C is shown in **Figure 6** for Lab 2. The best results with a shear area above 90% and total energy around 11000 J were achieved at a low reheating temperature (TR-1) in combination with a low final rolling temperature (FRT-2) and a low cooling stop temperature (TCS-1), while an increase of the reheating temperature (TR-2) caused a significant drop in the shear area and total energy. Increasing the final rolling temperature from FRT-2 to FRT-1 reduced both the shear area and total energy, while leading to a considerably higher scatter of values. Increasing the cooling stop temperature appears to have a slightly less negative influence.

Examples of fracture surfaces of two drop-weight-tear test specimens of Lab 2 taken from plates that varied only in their cooling stop temperature are shown in **Figure 7**. It appears that the increase of the cooling stop temperature has an effect on the fracture appearance. The reduction of the total energy after cooling to the higher cooling stop temperature may be linked to the formation of separations.

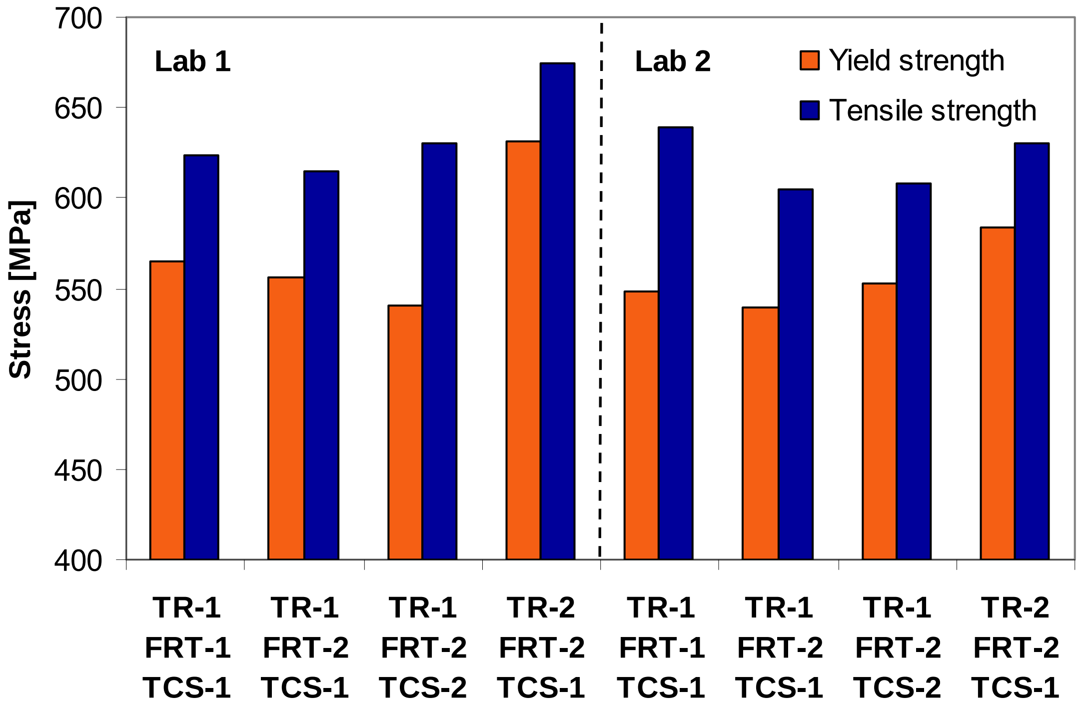

Figure 4: Effect of the variation of processing parameters on the tensile test results of the laboratory heats Lab 1 and Lab 2

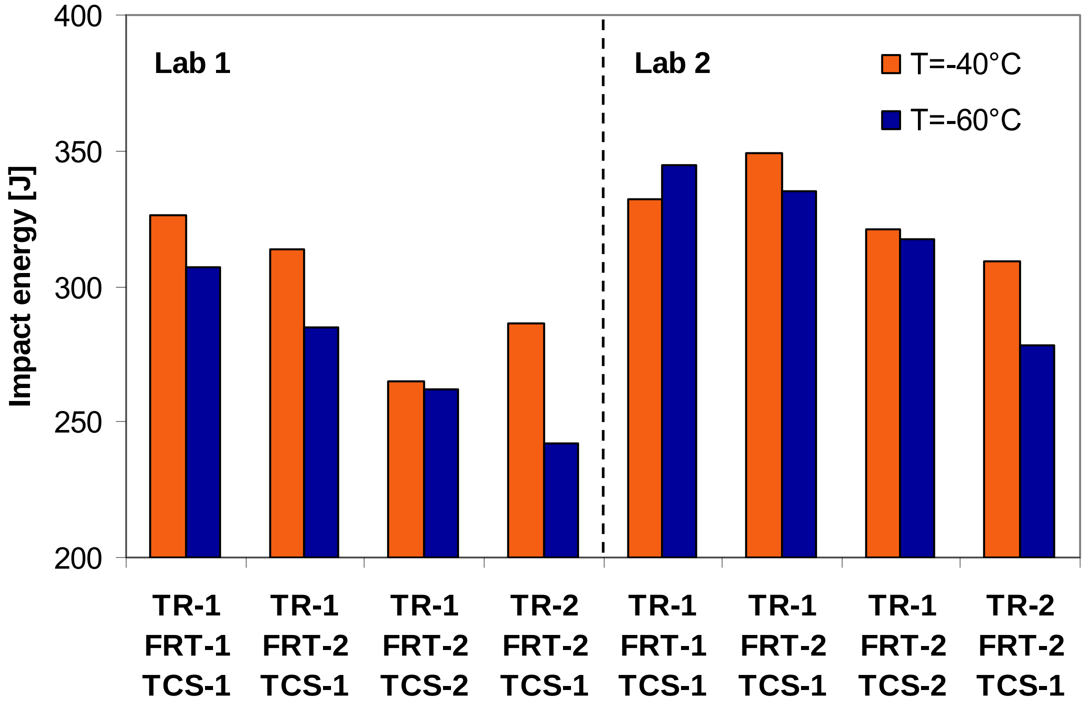

Figure 5: Effect of the variation of processing parameters on the Charpy test results of the laboratory heats Lab1 and Lab 2

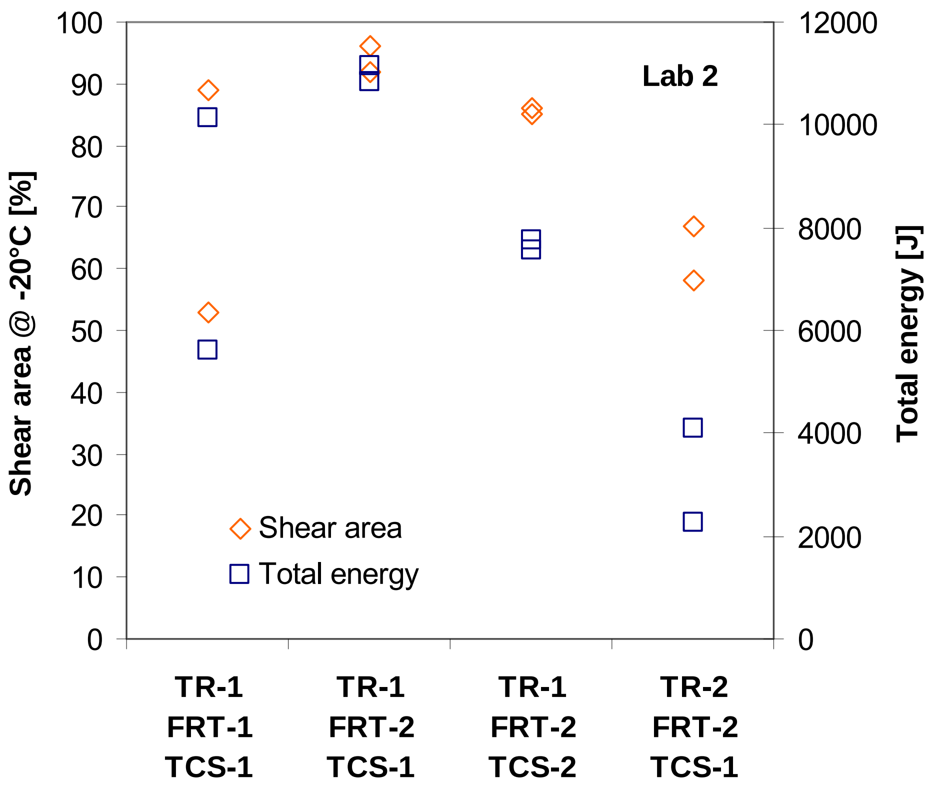

Figure 6: Effect of the variation of processing parameters on the shear area and total energy in the DWTT at -20°C of the laboratory heat Lab 2

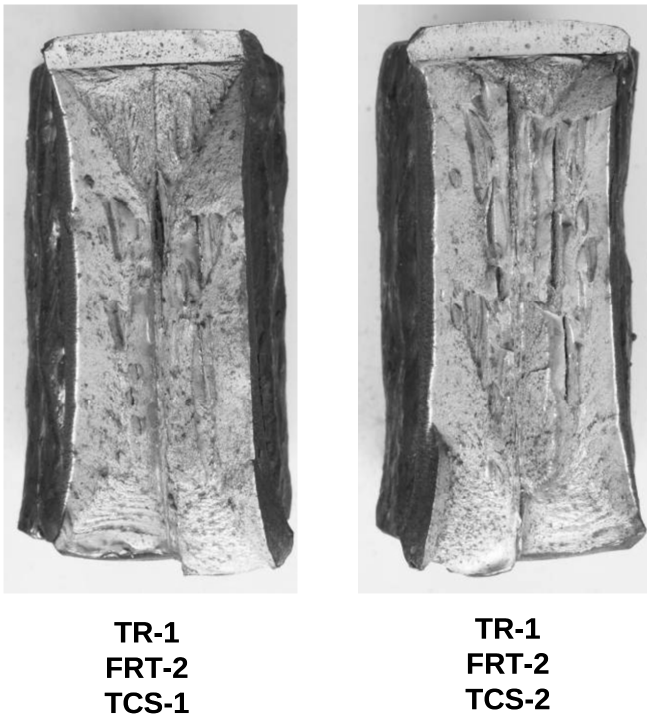

Figure 7: Two examples of fracture surfaces of DWTT-specimens of the laboratory heat Lab 2 illustrating the effect of the cooling stop temperature on the fracture appearance

In order to clarify the effect of the final rolling temperature on mechanical properties further, a second set of rolling trials on the heat Lab 3 was carried out. The reheating temperature was held constant but was increased compared to TR-1 by 50 K. The cooling stop temperature was maintained at the level of TCS-1. The final rolling temperature was decreased in steps of 20 K from FRT-2 to FRT-4.

The results of the tensile tests, presented in **Figure 8**, show that higher levels of yield strength and tensile strength were achieved compared to Lab 1 at the lower reheating temperature even though the composition of Lab 3 varied only slightly. Both the yield strength and the tensile strength fulfilled the tensile test requirements independent of the final rolling temperature. The mean impact energy did not differ significantly either and reached values above 300 J at -40°C and above 275 J at -60°C (**Figure 9**).

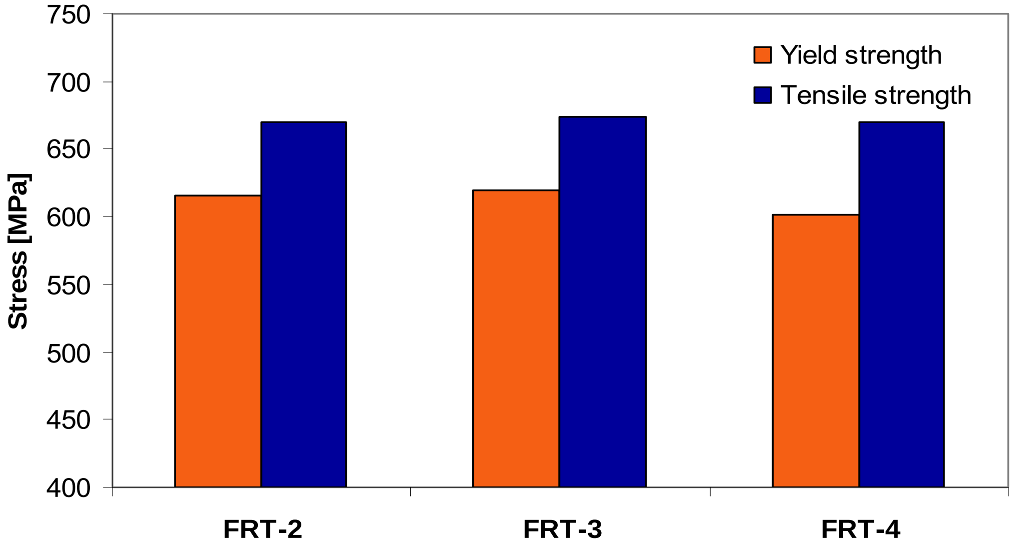

Figure 8: Effect of the variation of the final rolling temperature on the tensile test results of the laboratory heat Lab 3

In the drop-weight-tear tests at -20°C, the shear area and total energy was on a similar level compared to Lab 2 rolled under optimum conditions for DWT-properties. A further decrease of the final rolling temperature led to a higher scatter of the shear area fraction, but the total energy increased to above 15000 J (FRT-4).

The best compromise between strength and toughness properties was therefore realised with an intermediate reheating temperature and a low final rolling temperature.

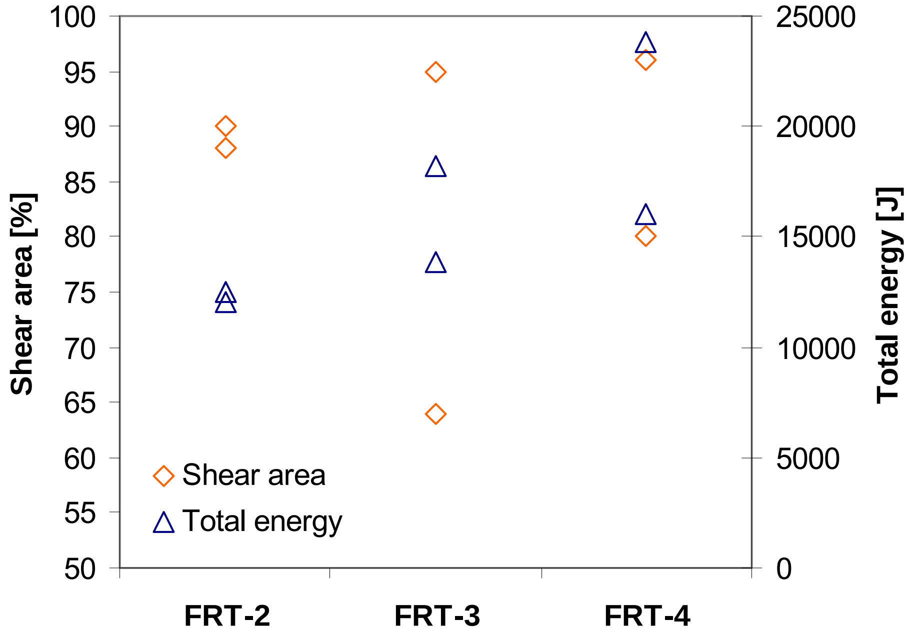

Figure 9: Effect of the variation of final rolling temperature on the shear area and total energy in the DWTT at -20°C of the laboratory heat Lab 3

MILL TRIALS AT THE HEAVY PLATE MILL

Production scale rolling trials were carried out at MGB using continuously cast slabs produced by HKM. The compositions of the three industrial heats used in these trials are shown in **Table 3**. The final rolling temperature was set above the A_{r3} temperature, followed by acclerated cooling down to temperatures in the range of 300-500°C. This resulted in a microstructure that consisted predominantly of bainite with minor volume fractions of ferrite. In a first set of industrial trials, all three heats were rolled under similar processing conditions using a higher reheating temperature. In a second set of trials on the heats Mill 2 and Mill 3, the reheating temperature was varied in order to investigate the effect on the mechanical properties. Compared to the first trial, the reheating temperature was lowered by 30 K. In the second set of mill trials, instrumented DWTT were carried out in order to better characterise the relationship between processing parameters and potential for crack arrest.

The results of the tensile tests in longitudinal and transverse direction of plates produced in the first set of rolling trials are shown in **Figure 10**. The error bars in the following diagrams reflect the scatter of the individual values. While all three heats achieved similar levels of yield strength in longitudinal direction, the highest values in transverse direction were obtained with the heat Mill 1 which has the highest niobium content of the three heats. This material also showed the highest tensile strength in both directions. The reduction of the carbon content in Mill 2 and in the niobium content in Mill 3 resulted in lower strength levels which were nevertheless above the required values. The CVN impact tests showed that all three materials were above the required levels, but the heats Mill 2 and Mill 3 achieved higher levels of impact toughness.

	Composition [wt.%]						
	C	Si	Mn	others	Nb	Ti	CE_{IIW}
Mill 1	≥0.05	≥0.20	≥1.8	Cu, Ni, Mo	≥0.050	≤0.02	0.44
Mill 2	≤0.05	≥0.20	≥1.8	Cu, Ni, Mo	≥0.050	≤0.02	0.44
Mill 3	≥0.05	≥0.20	≥1.8	Cu, Ni, Mo	≤0.050	≤0.02	0.45

Table 2: Compositions of the investigated industrial heats

This result was mirrored in the results of DWTT presented in **Figure 11**. All three materials achieved a mean shear area above 85%. However, the scatter of values was reduced significantly in the case of the heats Mill 2 and Mill 3. For this reason, the heat Mill 1 was not used for the second set of mill trials in which the effect of the reheating temperature was investigated. Since both the heats Mill 2 and Mill 3 achieved comparable results in the first trial, the results presented in the following are combined results of these two compositions and are compared with those obtained using the higher reheating temperature.

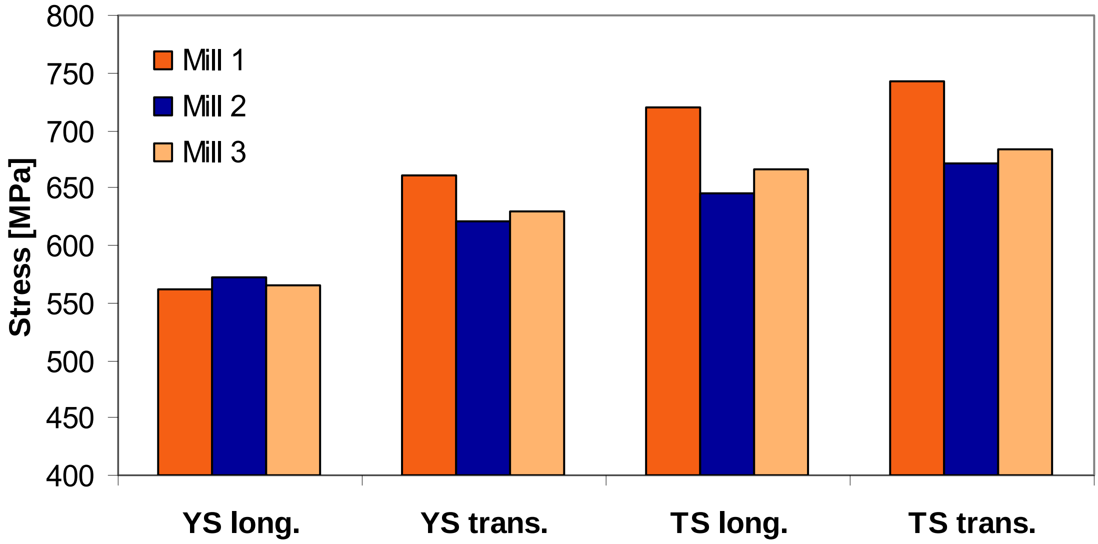

Figure 10: Results of tensile tests of plates produced in the first set of mill trials

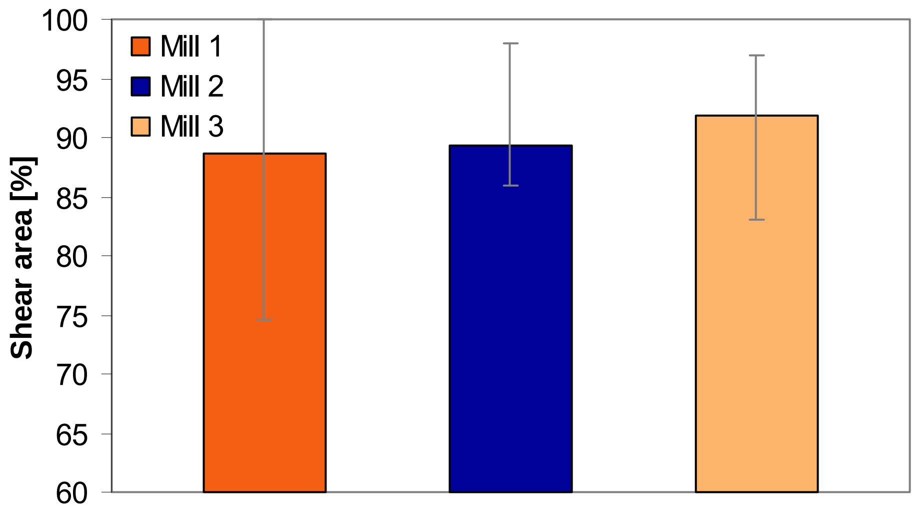

Figure 11: Shear area at -30°C obtained in DWTT of plates produced in the first set of mill trials

The results of the tensile tests of the second mill trial are presented in **Figure 12**. Based on the results of the laboratory trials, a drop in the strength values was expected as a result of the lower reheating temperature. This was indeed observed in case of the transverse yield strength and the tensile strength. However, the results were nevertheless above the required values, as the expected drop in tensile values was sufficiently compensated by the addition of molybdenum.

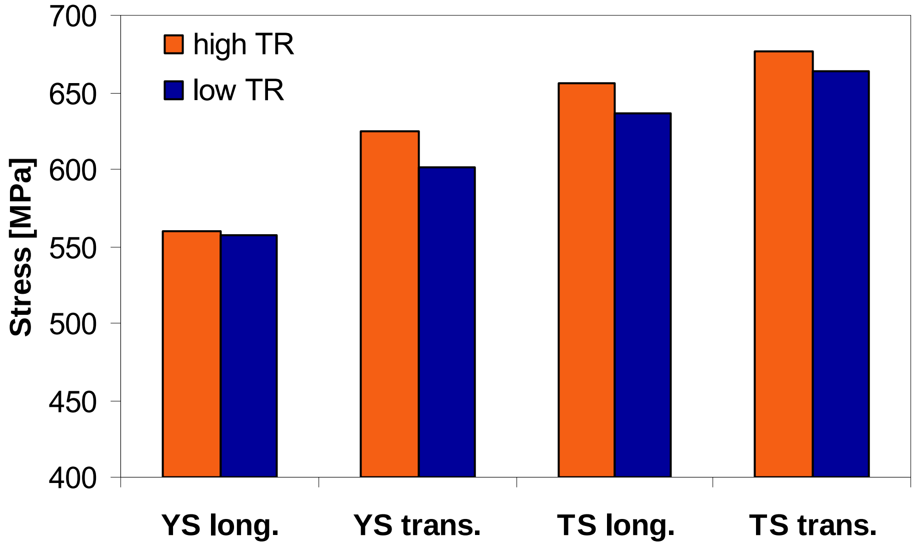

Figure 12: Effect of the reheating temperature on tensile properties

In the Charpy test, around 300 J mean impact energy were achieved at both reheating temperatures, i.e. the Charpy test did not give a conclusive indication of the effect of lowering the reheating temperature on the potential for crack arrest. Similarly, the mean shear area that was measured in the DWTT, was practically identical for the two reheating temperatures, see **Figure 13**. However, the instrumented DWT tests showed that the mean total energy was indeed affected significantly by the reheating temperature, as was already observed in the laboratory investigation.

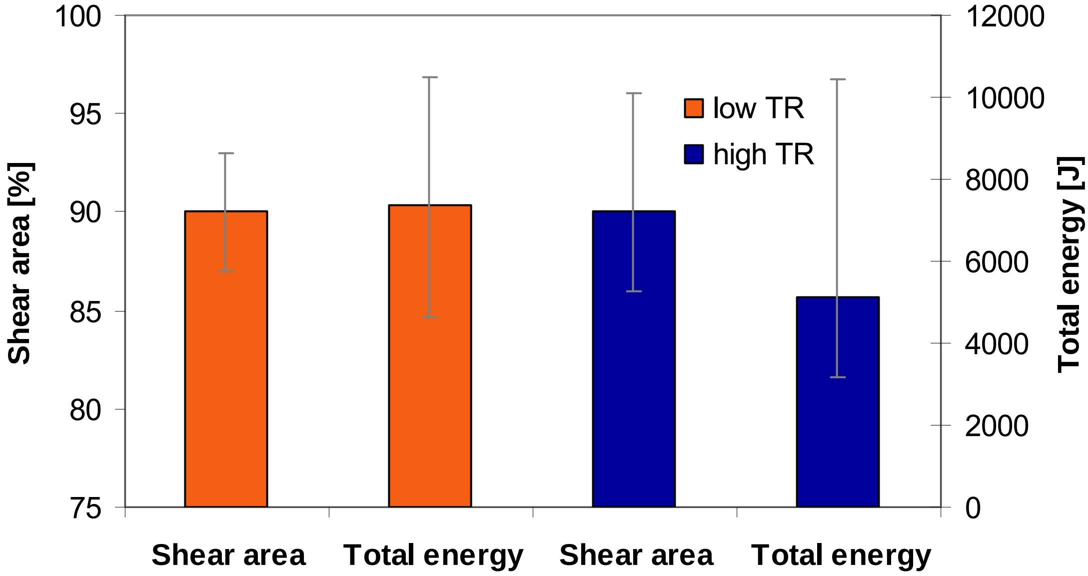

Figure 13: Results of DWTT of plates produced in the mill trials

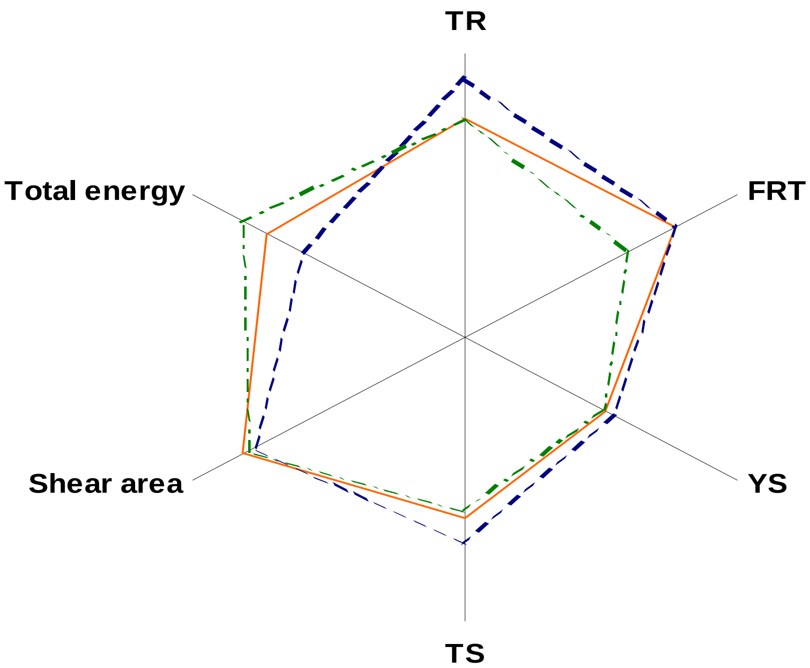

Figure 14: Effect of reheating and final rolling temperature on tensile and DWTT properties

The effect of the processing parameters is summarised schematically in **Figure 14**. The rolling trials showed that a strength increase could be achieved by increasing the reheating temperature. However, this led to a significantly lower total energy in the DWTT. A reduction of the final rolling and the cooling stop temperature was found to have a positive effect on the total energy while the tensile properties were not affected significantly. Based on the results of these trials, the processing window for the production was defined and plates for pipe production at EUROPIPE were rolled at MGB.

PIPE PRODUCTION AT EUROPIPE

In order to qualify MGB and EUROPIPE for the supply of pipe for the arctic project Bovanenkovo-Uchta (Yamal peninsula), a trial pipe production was conducted. The pipe dimension was (56”) 1420 mm x 27.7 mm. The steel grade specified was K65 (SMYS=555 MPa). Pipes were produced in 12 m lengths at the Mülheim 18m-line of EUROPIPE. After forming the pipes were double-submerged arc welded with 4 wires inside and 5 wires outside using an optimised TiB wire.

For the qualification a wide range of tests had to be carried out. **Table 3** shows an extract of the requirements and the relevant results. All strengths properties of the base material were comfortably met. A special focus was put on the toughness properties. Due to the fact that hydraulic burst tests with notches (test temperature -20°C) and a full scale burst test (test temperature -10°C) had to be performed, high toughness in base material and weld material were substantial.

Tensile Test	Requirements		Test Results	
	min.	max	min.	max.
YS transverse [MPa]	555	665	566	609
TS transverse [MPa]	640	760	654	733
YS longitudinal [MPa]	500	665	555	561
TS longitudinal [MPa]	610	760	634	647
CVN [J] @ -40°C	Requirements		Test Results	
	min. single value	min. average value	min. / max. single value	average value
BM	150	200	284/ 338	318
WM	42	56	91/ 207	144
HAZ	42	56	76/ 264	212
DWTT-SA @-20°C [%]	Requirements		Test Results	
	min. single value	min. average value	min. / max. single value	average value
	75	85	85/100	97

Table 3: Mechanical properties of produced pipes

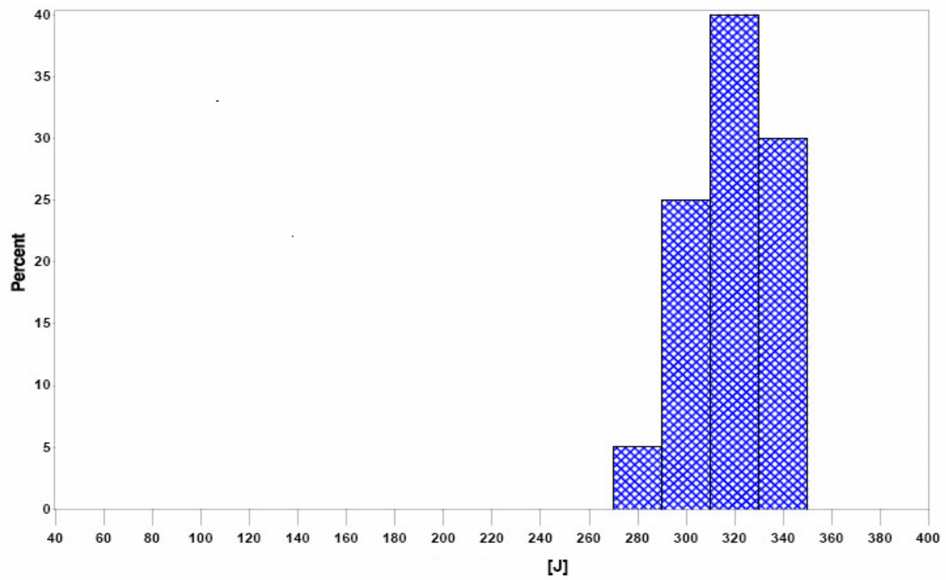

Figure 15: Charpy-V notched bar impact test, base metal, test temperature -40°C

The Charpy test temperature was set to -40 °C and base **(Figure 15)** and weld material easily fulfilled the required 200 J respectively 56 J. Also good results were achieved in the HAZ but single low values can never be avoided.

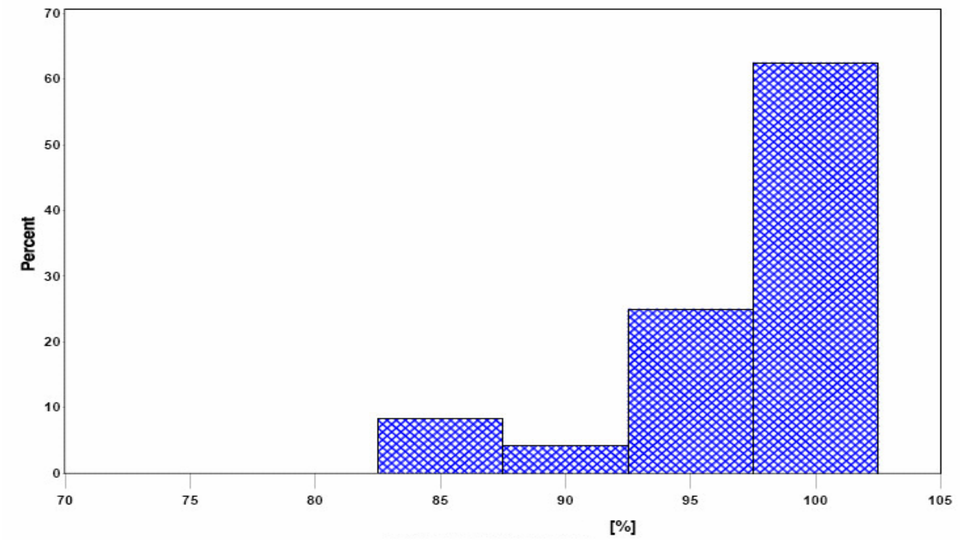

Figure 16: DWTT, test temperature -20°C

Also the DWT tests at -20 °C showed good results. **Figure 16** shows the distribution where most specimens reached 100% shear area. Not only the shear area specified as min average of 85% is important but also a high DWT energy influences significantly the arrest of long running cracks.

Selected pipes of this trial production were shipped to Kopeisk in Russia for the performance of the full scale burst test.

FULL SCALE BURST TEST

The result of a full scale burst test should be a verification of the design of the pipeline with the given parameters steel grade, diameter and wall thickness, max. pressure and lowest possible temperature. Several full scale burst tests were performed in the last years with X80 material, but the qualification tests in Russia were the first with reduced temperatures.

The selected pipes were jointed to a string of about 60 m with a short starter pipe in the center and pipes with increasing toughness to both directions. Reservoir pipes completed the test loop to a max. length of 200m. The pipes were buried by about 2/3 of the circumference with the longitudinal weld in the 5 to 7 o'clock position. To reach the final test pressure and test temperature, special compressors and cooling devices were installed.

After reaching a pressure of 150 bar and a temperature of -10 °C an explosive charge was ignited in the starter pipe. **Figure 17** shows the moment of the explosion. As it was forecasted due to the design of the material, a crack arrest occurred in the pipe placed directly after the starter pipe. **Figure 18** is a photo of the open ditch directly after the test. The crack turned from longitudinal to circumferential direction and stopped in the tough base material. The ductile fracture surface close to the area of crack arrest is shown in **Figure 19**.

Figure 17: Full Scale Test in Kopeisk, Russia

Figure 18: Crack Arrest after full scale test

Figure 19: Fracture surface of arrest pipe

CONCLUSIONS

25 years ago, it was a challenge to develop high strength X80 materials. The latest reports were given 4 years ago with the first use of X80 in Italy [9] and with a summary last year in China [10]. Today the new challenge was the development of a high strength X80 for arctic application. In this qualification for K65 required also a full scale burst test at low temperatures.

To develop such a steel and such a pipe, the whole production chain had to cooperate with the support of Salzgitter Mannesmann Forschung. Starting with lab trials continued by plate and pipe trial production and steady improvements along the entire process chain finally the qualification to supply pipes for the Bovanenkovo-Uchta line was successfully achieved.

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