IMPROVING MECHANICAL PROPERTIES OF API X60/X70 WELDED PIPELINE STEEL

Presented in QAI-ICF, 1-5 June 2008 Algiers

Résumé

L'objectif de cette étude est d'améliorer la microstructure et les propriétés mécaniques de l'acier pour gazoduc en utilisant un laminoir pilote. Pour atteindre la microstructure et les propriétés mécaniques requises des aciers à haute limite élastique et faiblement allié (HSLA) par un procédé thermo mécanique, il est nécessaire d'avoir une idée sur le rôle de la composition et les paramètres du procédé. Le grand nombre de paramètres obtenus au cours du processus de production dans l'usine a été systématiquement modifié afin d'optimiser la résistance et la ténacité. Les paramètres optimisés sont utilisés pour la production de l'acier API X60/X70. Cependant, le refroidissement contrôlé après le laminage devrait avoir comme conséquence la production de bandes qui présentent une excellente combinaison de la résistante et de la ténacité. Le bobinage à une température appropriée tire profit du renforcement de la précipitation, donnant à nouveau lieu au durcissement structural et également à une amélioration de la limite élastique et de la ténacité de l'acier. La température de bobinage est un paramètre décisif parce qu'elle détermine le début de la formation des précipitations fines. Par conséquent, quatre systèmes de refroidissement ont été employés, afin de simuler les conditions de laminage d'un procédé TMCP réel, et vérifier les possibilités d'améliorer les propriétés mécaniques de l'acier à tube.

Mots clés *: Micro allié ; TMCP ; Laminage contrôlé ; refroidissement contrôlé ; paramètres du procédé, propriétés mécaniques.*

Abstract

The aim of this study is to improving microstructure and mechanical properties of the weldable gas pipeline steel using laboratory mill. To achieve the required microstructure and mechanical properties of thermo mechanically processed HSLA steels, it is necessary to have an idea about the role of composition and process parameters. The large numbers of parameters obtained during the production process in the plant were systematically changed to optimize the strength and toughness properties. The optimized parameters were used for the production of the API X60/X70 steel. However, the controlled cooling after rolling should result in transformed products that provide excellent combination of strength and toughness. The coiling at an appropriate temperature have the advantage of the precipitation strengthening, giving further rise to the high yield strength and also improvement in toughness of the steel. The coiling temperature is a decisive parameter because it determines the beginning of the formation of fine precipitations. Therefore, four different laboratory cooling systems were used, in this study to simulate the rolling conditions of a real industrial Thermo mechanically controlled process (TMCP), as close as possible and to check the possibilities of improving the mechanical properties of the welded pipeline steel.

Keys words *: Micro alloying; TMCP; Controlled rolling; Controlled cooling; processing parameters; Mechanical properties.*

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ملخص

الهدف من هذه الدراسة هو تحسين البنية و الخواص الميكانيكية للفولاذ المخصص لصناعة أنابيب الغاز باستخدام جهاز درفلة مخبري ، و لتحقيق ذلك فإنه من الضروري معرفة دور التركيب الكيميائي و التحكم في ميكانيزمات و خصائص طريقة التشكيل.

العدد الكبير من المعطيات التي تم الحصول عليها أثناء عملية الإنتاج في المصنع تم تغييرها منهجيا لتحسين خاصيتي المتانة و الصلابة. عناصر التحكم المثلى استخدمت لإنتاج الصلب فصيلة APIX60/X70 .

إن التحكم في التبريد بعد عملية الدرفلة ، ينبغي أن يؤدي إلى إنتاج شرائط ممتازة توفر مزيجا من المقاومة و الصلابة ، و للتلفيف على درجة حرارة مناسبة فوائد تعزيز البنية الداخلية مما يؤدي إلى تصليب الهيكل من جديد و أيضا تحسين حد المرونة و صلابة المعدن ، درجة حرارة التلفيف عامل أساسي لأنها تحدد بداية التشكيل البنيوي ، لذلك أربعة أنظمة تبريد استخدمت لمحاكاة ظروف درفلة حقيقية(TMCP)، و للتحقق من إمكانية تحسين الخصائص الميكانيكية لأنابيب الصلب.

الكلمات المفتاحية: *ميكروسبيكي ، تبريد مراقب ، درفلة مراقبة ، عناصر التشكيل ، خصائص ميكانيكية*

The idea of the development of pipeline steels with high yield strength and toughness [1 to 3] was based on user demands towards improvement in weldability and reduction of welding costs, especially during the construction of the pipelines [4 to 15]. In modern pipeline technology, both high strength and toughness are of primary interest [16 to 19]. The appropriate selection of microstructure is an important factor to further improve the weldability, strength and toughness behaviors of the oil and gas pipeline steels [20 to 24].

Thermomechanical processing is known to improve the mechanical properties of a material. In the case of high grade steels, application of this process minimizes, and sometimes even eliminates the heat treatment and thus saves energy. So, in high-strength low-alloy (HSLA) steels Thermomechanically controlled processing (TMCP) is a widely used process. To achieve the required microstructure and mechanical properties of thermo mechanically processed HSLA steels, it is necessary to have a good knowledge about the role of composition and process parameters. The chemistry of the steel and the TMCP parameters, like reheating temperature, amount of deformation at different stages of rolling, the finish rolling temperature and the cooling rate are known to exert appreciable influence on the structure and property of the finished product.

It is known that the patterns relating inputs and outputs in TMCP steels are qualitatively recognized by the experts in the field of metallurgy [25 to 27].

The present work is a laboratory study of the effects of the processing parameters on the microstructure and properties of standard pipeline grade X60/X70 type API HSLA steel.

1. EXPERIMENTAL PROCEDURE

The chemical composition of the steel used in this investigation is given in Table 1. The steel was supplied by the Elhadjar Iron and Steel Factory (Elh-ISF), Algeria.

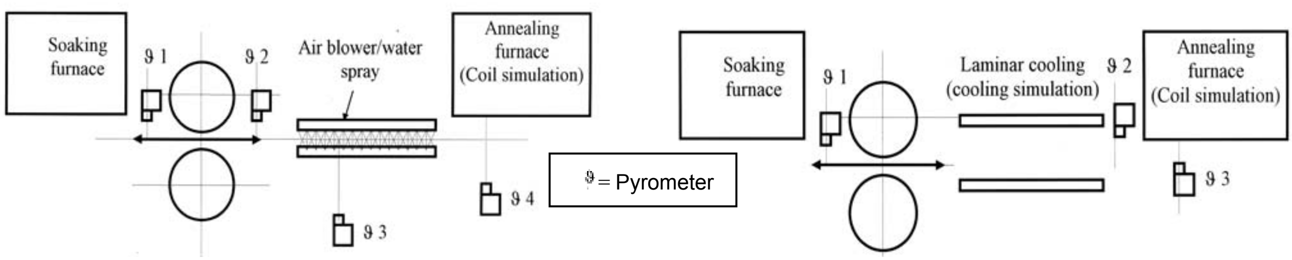
1.1. Rolling, cooling and annealing technology

After cooling down on air and reaching the start temperature, The rolling schedules (I) and (II) took place, at first the two passes of the simulated roughing process (passes 1 and 2 in table above). After reaching the temperature of finishing process, the other six passes took place (see passes 3 to 8), using two different pass sequences. The variation of reductions is pass per pass same reduction for the rolling schedule (I), and pass per pass reduced reduction for the rolling schedule (II). The roughing passes are done at temperature between 1150°C and 1120°C, and followed by cooling in ambient air while the finishing passes between 850°C, 800°C and 750°C. After the rolling process, compressed air is used for cooling.

Table 1 : Chemical composition (in % weight)

C	Si	Mn	P	S	Cr	Mo
0,139	0,130	1,51	0,011	0,004	0,01	0,01
Al	**N**	**Cu**	**Nb**	**Ti**	**V**	**Ni**
0,0243	0.0211	0,026	0,075	0,024	0,041	0,01

The schedule (III) consisted of six passes (finish rolling simulation only) with finish temperatures of 800°C, 750°C and 700°C. The material was heated in a furnace; afterwards it cooled to rolling temperature in ambient air. After rolling, the steel was cooled by water spraying, compressed air and in ambient air to temperatures between 650°C, 600°C and 550°C. This was followed, as in the former experiments, by a holding on finish temperature for 2 hours. New is, after the two hours of holding no cooling in ambient air to 20°C, but a furnace cooling to 300°C. An objective was to see possible effects of slower cooling rates on the properties after coiling. The program of this schedule can be seen below.



a) Equipment for cooling of rolled strips with compressed air or water spray.

b) Equipment for laminar cooling of rolled strips

Figure 1 : Schematic diagram of basic concept of the laboratory equipment used for the experiments

Plate-controlled rolling process followed by controlled cooling tests was carried out on laboratory rolling mill with 330 mm diameter rolls and rolling speed of 1m/s. In the present work, slabs (55x100x100) mm^3 were reheated at 1250°C for 30 min, and were rolled to (14.5 to 12) mm thick plates with six to ten phases as four (4) different rolling schedules (see figure 1).

The schedule (IV) was, more adapted to the real rolling conditions in (Elh-ISF). So it consisted of 5 roughing passes and of 5 finishing passes, all in all 10 passes. More passes were not possible because of the delivered raw material thickness of 55 mm. The roughing passes are done at temperature between 1200°C and 1050°C, followed by cooling in ambient air and the finishing passes between 950°C and 800°C, res.750°C.After the rolling process two cooling systems were used : Laminar water cooling and cooling with compressed air.

After Rolling, annealing and cooling the samples were mechanically worked to get specimen for tensile tests, notch impact tests (at room temperature as at lower temperatures of 0°C and -30°C with longitudinal specimen) and microstructure investigations.

1.2. Cooling tests

Four different cooling systems were used, in order to simulate the rolling conditions in (Elh-ISF), as close as possible and to check the possibilities of improving the mechanical properties of steel X60/X70. In so doing, very different cooling rates were possible. At the rolling schedules (I) to (IV), all experimental methods were in operation, so that a comparison of the effects of the different cooling methods and especially of different cooling rates on the properties of the finished strip is possible. The real temperature in the centre of a finish rolled strip was always measured with thermocouples. Table2 gives an overview of the used cooling methods and cooling rates. As expected, laminar cooling shows the highest cooling rates (50K/s), followed by water spraying (21K/s). Compressed air brought not so high cooling rates (3, 6 K/s); the lowest rates are achieved with cooling in ambient air (1,1K/s).

Table 2: Used cooling methods and cooling rates of all experiments

Cooling method	Rolling Schedule				Cooling rate between 800°C and 600°C
	I	II	III	IV	
Ambient air	X				1,1 K/s
Compressed air	X	X	X	X	3,6 K/s
Water spray	X				21 K/s
Water Laminar cooling		X			50 K/s

The problem with the used configuration for water spraying was especially a strong reheating effect after the end of cooling. So a reheating of about 35 to 40 took place, which could have affected the properties of the final strips. At the other cooling systems, especially at laminar cooling, such a reheating effect was also seen, but of much smaller magnitude. Apparently when cooling with water spraying the strip was not cooled completely to the centre, though the pyrometer indicated the desired surface temperature, so that such a reheating could appear.

1.2.1. Laboratory equipment for cooling of rolled strips

1.2.1.1. Equipment for water spraying and compressed air

For an improved cooling of the rolled strips after the rolling process laboratory equipment should be used, with which it would be possible to adjust defined cooling conditions. In the former rolling schedules (I) and (II) the finished strips were cooled by pressured air and / or in ambient air. To adapt the cooling conditions in the laboratory mill more to the real industrial conditions in (Elh-ISF) first a water spraying system was developed and tested. The cooled strip was not reversed between the spraying tubes. To find the precise data before the rolling experiments the complete system was calibrated by thermocouples which were drilled to the centre of a rolled strip. In these preliminary tests the temperatures measured by thermocouples were compared with the data of the pyrometer. Actually in the rolling experiments the temperature of the cooling system is followed making use of the calibrated pyrometer.

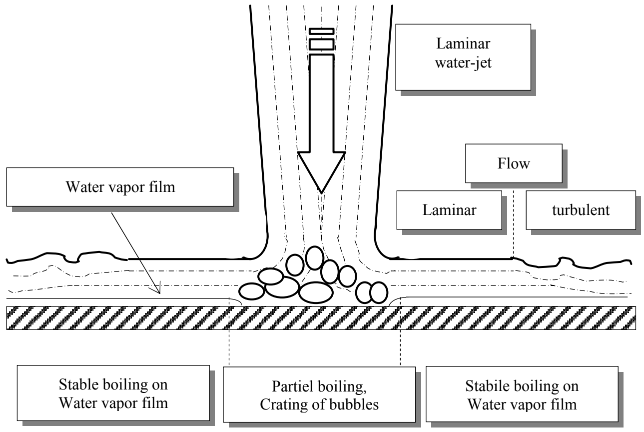


Figure 2 : Schematic illustration of regions of local heat transition on the hot strip at laminar cooling

1.2.1.2. Equipment for laminar cooling

The principle of laminar cooling is a water curtain, which allows a high cooling capacity as a uniform cooling over the strip in lateral direction. Figure 2 gives a schematic illustration of the conditions at laminar cooling of strips. For laminar cooling of rolled strips a new experimental device was introduced. The complete system for simulation of water laminar cooling consists of a cooling system, a sleigh on railways, a propulsion unit, as of a unit for measuring and controlling. The advantage of this apparatus is that by regulation of water flow, jet dimensions and transport speed of the strip it is possible, to adjust defined cooling rates.

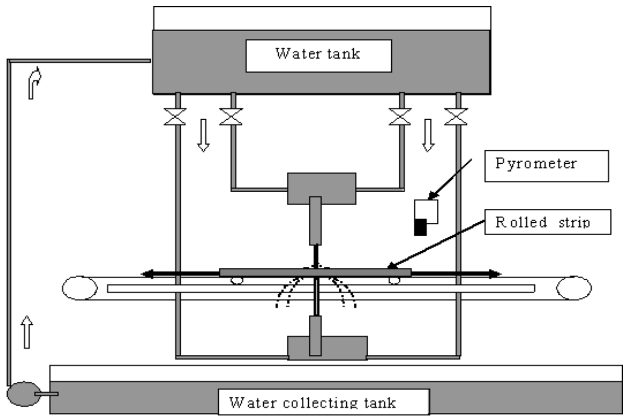


Figure 3 : Schematic diagram basic concept of the laboratory equipment for laminar cooling of rolled strips

With the used equipment, which is schematically shown in Figure 3. The cooling takes place from both sides of the strip that means from above, as from beneath. But it is also possible to cool from one side only. For a better heat transfer in these experiments a cooling from both sides was selected.

For a sprinkling with water, there are different nozzle cross sections. The flow volume was adjusted by vents. The distance between nozzles and strip surface is also precisely adjustable, but was not changed during the experiments. For a controlled laminar cooling the rolled specimen is placed between the nozzles on a sleigh and transported through the water curtain. The desired cooling rate is finely adjusted through the selection of the quantity of water and of the transport sleigh speed. But to find the precise data, it was necessary before start of the real experiments to calibrate the complete system that means to check the quantity of water, and the sleigh speed by specimens with thermocouples. So the temperature in the centre of the specimen was compared with the data from a pyrometer.

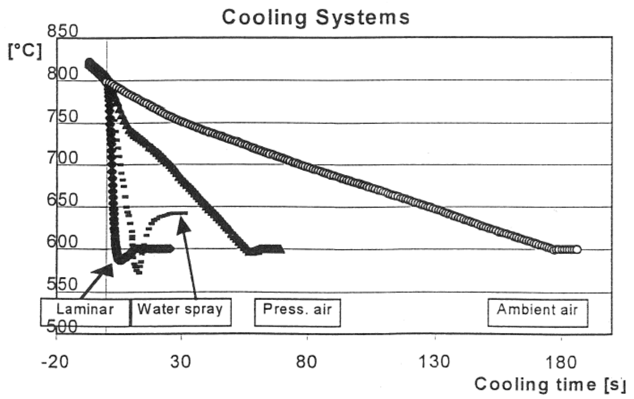


Figure 4 : Cooling times and reheating after rolling dependent on cooling methods

This was the basis for a controlled cooling of the later rolled strips. A short sketch of the described configuration for cooling of hot rolled strips is seen in Figure 4. The technical data of the used device are summarised in the following Table 3.

Table 3 : Technical data of the used laminar cooling device

Cooling field	1000°C-20°C
Cooling rate at strip a thickness of 20mm	max. 35°/s
Strip cooling at the top	
Nozzle cross section	140 x 5mm, 140 x 10mm
Water exit speed	max. 3,2m/s
Distance nozzle strip	50-300mm
Strip cooling from below	
Nozzle cross section	120 x 5mm, 100 x 10mm
Water exit speed	max. 4m/s
Distance nozzle strip	50-250mm
Water tank	400 l

1.3. Rolling experiments

1.3.1. Rolling Schedule (I)and (II)

After withdrawal of the specimens from the soaking furnace, these were transported to the reversing Duo mill.

The start temperature, like the temperature of each pass, was measured by pyrometers. Alter cooling down on air and reaching this temperature, rolling took place, at first the two passes of the simulated roughing process. When reaching the temperature of finishing process, the other six passes took place. In each pass the measured temperature (pyrometer) gave the signal for the beginning of rolling.

After pass 8 the specimens were laid under an air cooling devise, which used compressed air, so a very fast cooling was possible. Here a low temperature pyrometer was used to control the cooling process. After reaching the desired temperature the specimens were laid in other furnace, to simulate the cooling process of a rolled strip (coil simulation). Her they laid for another two hours. Afterwards the samples were withdrawn from the annealing furnace and cooled in stable air to ambient temperature.

Figure 5 shows the temperature-time regime at rolling in three different temperature settings (variation of rolling temperature).At selected specimens the dimensions (height and width) were measured between passes. The results of these first experiments are a prerequisite for later investigations with a revised experimental schedule.

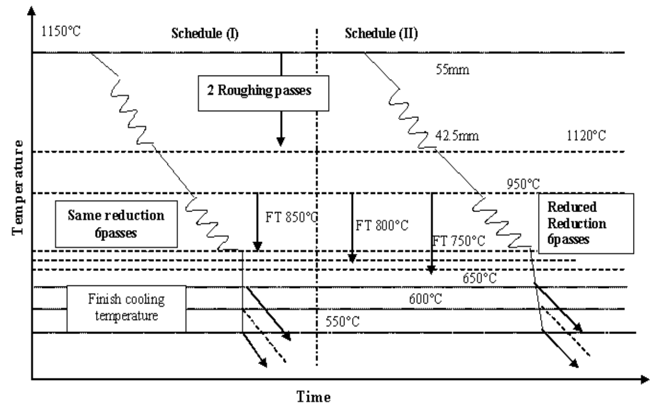


Figure 5 : Temperature-time regime of the complete technological process at rolling schedule (I) et (II)

1.3.2. Rolling schedule (III)

After rolling according to schedule (III) the strips were cooled by water spray or compressed air res. ambient air. Afterwards the specimens were kept two hours at coil temperature in a furnace. Then, the furnace was switched off and the rolled strips cooled in a longer time period within the furnace to 300°C.

Later they cooled down to room temperature in ambient air. Figure 6 shows graphically the temperature-time regime of rolling schedule (III) until the finish of cooling to coil temperature by spray water or air, and the complete technological process can be seen. It starts with the withdrawal of the specimens after soaking and finishes with the extraction of the strips from the furnace after cooling to 300°C and following cooling in ambient air to 20°C. All in all it required 17h 52mn 20s to cool down to 300°C and 18h to reach 20°C. This is in a very good with the cooling times of a complete coil in (Elh-ISF).

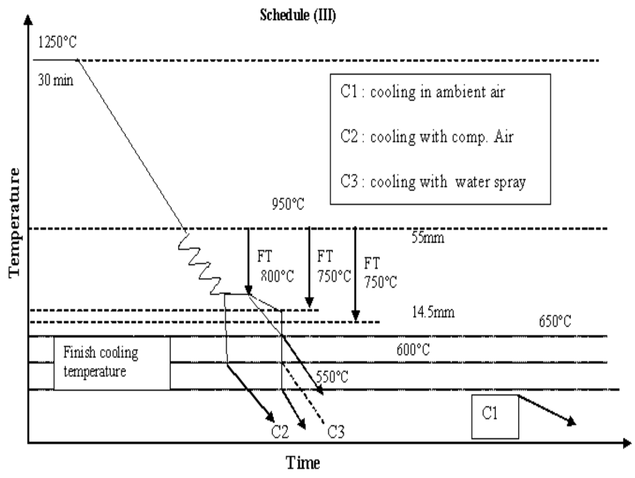


Figure 6 : Temperature-time regime of the complete technological process including furnace cooling at rolling schedule (III).

1.3.3. Rolling schedule (IV)

At rolling schedule (IV), the roughing process needed 90 seconds (cooling of the strip after each pass in ambient air), finishing took place within 150 seconds. The complete rolling process according to rolling schedule (IV) took 237 seconds.

Especially here was a longer inter pass time of 50s at cooling of the strip in ambient air between roughing and finishing. This should simulate the transport of the strip from roughing mill to finishing mill. The simulation of coil cooling was finished after 7400s (2 hours holding at cooling temperature after finish rolling) Afterwards the strips were extracted from the furnace and cooled in ambient air. So the complete process ended after 11000s at a temperature of 20°C.

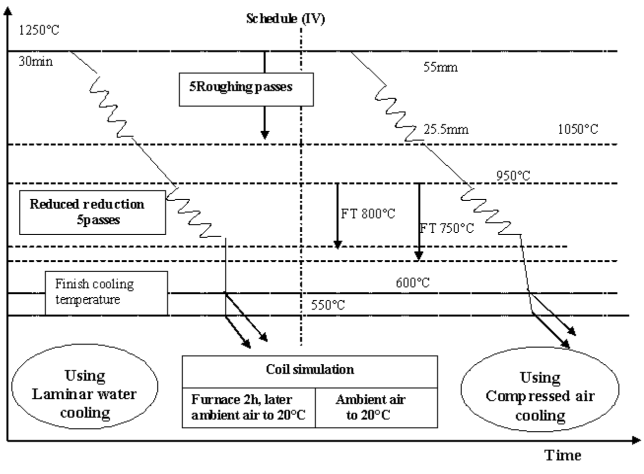


Figure 7 : Temperature-time regime of the complete technological process in rolling schedule (IV)

Figure 7 shows the temperature-time regime of the complete technological process according to rolling schedule (IV) including simulation coil cooling in the furnace and afterwards cooling in ambient to 20°C.

2. EFFECTS OF DIFFERENT COOLING SYSTEMS ON MECHANICAL PROPERTIES

Four different cooling systems were tested, to see the effects of varied cooling strategies on the mechanical properties of steel X60/X70 and to simulate the practical cooling and coil conditions in (Elh-ISF) as close as possible. Right from the start of the investigations besides cooling in ambient air also a compressed air cooling on both sides of the rolled strip was used. With the cooling rate of about 3,6K/s could be realized, compared with 1,1K/s at cooling in ambient air.

So a water spraying system was developed. This worked like the used system for cooling with compressed air and brought cooling rates of about 31K/s. But the experiments with water spray cooling showed also, that after finish of cooling a reheating affect appeared. The cooled strip heated again, using the residual heat. To avoid this and to simulate the practical conditions in (Elh-ISF) as close as possible with cooling rates of about 50K/s. For these purposes the mechanical properties depending on finish and cooling temperature and especially on the cooling rate were assembled. This is illustrated in Figures 8, 9 and 10. As we can see at the mechanical properties of steel X60/X70, the cooling rate is of lower weight, but water laminar cooling brought the best results.

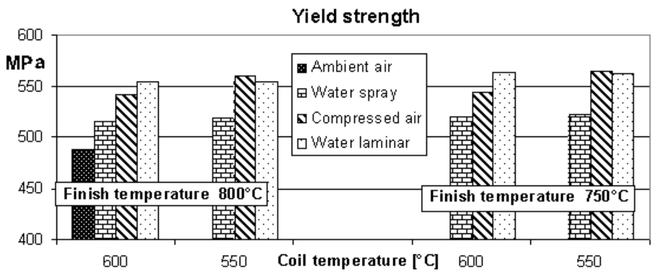


Figure 8 : Effects of different cooling systems on yield strength

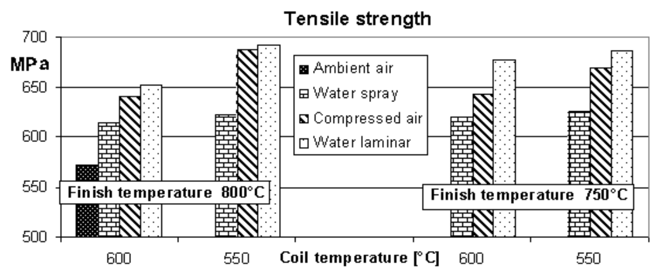


Figure 9 : Effects of different cooling systems on tensile strength

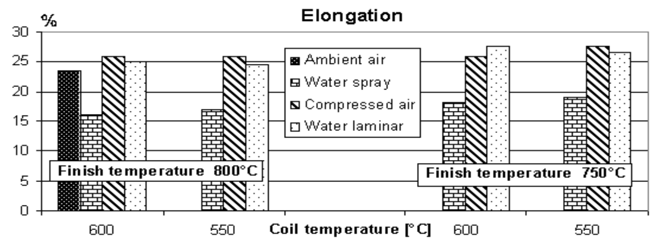


Figure 10 : Effects of different cooling systems on elongation

So the yield strength of the finish rolled and cooled strip improves going from cooling in ambient air to spry water, then compressed air, ending by laminar cooling. If we observe the cooling rate only, water spraying (31K/s) brought better values than compressed air (3.6K/s) and lies beside laminar cooling (50K/s). An explanation for this phenomenon should be the already mentioned reheating phenomenon of the cooled strip after water spray cooling. On the other hand does this mean that the achievement of a precise cooling temperature is of more importance than the cooling rate. The cooling rate affects the properties slightly, if we compare the results of tensile tests in Figs.8 to 10. So, the yield strengths after cooling in ambient air and laminar cooling do not differ substantially (13.5% only). Concerning the tensile strength we see the same relations (14%). Amazing are the high values of elongation after cooling in ambient air. Here water spraying brought the lowest values, which differ to a maximum of 36%compared to either compressed air or laminar cooling. Concerning the toughness, (in Figures 11, 12 to 13), cooling in ambient air brought at all test temperatures good results. Here also a cooling with spray water showed the lowest data. Observation does not also change at low test temperatures of 0°C or -30°C. If we see all tested mechanical properties (strength and toughness) as an entity, so the best results for steel X60/X70 were found after laminar cooling, especially at finish cooling temperatures below 600°C.

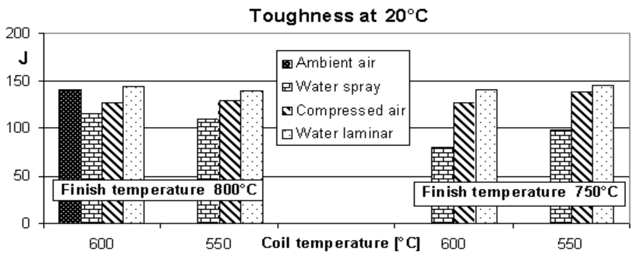


Figure 11 : Effects of different cooling systems on toughness at 20°C

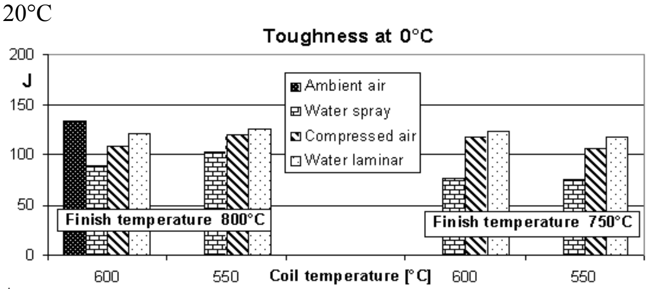


Figure 12 : Effects of different cooling technologies on toughness at 0°C

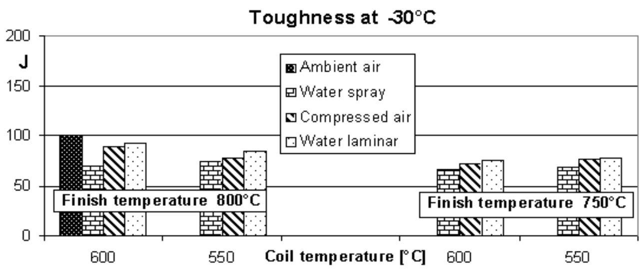


Figure 13 : Effects of different cooling technologies on toughness at -30°C

3. MICROSTRUCTURE

In order to estimate the existing grain size very precisely, for the microstructure investigations of each rolled strip one specimen was extracted and prepared.

In an area of 10mm x l0mm, 6 to 8 micrographs were taken and the grain size measured. The grain sizes of all rolled strips according to rolling schedule (IV) do not differ very much, see Table 7 below.

Table 7 : Average grain size of strips rolled according to rolling schedule (IV)

Strip N°	Temp.[°C]		Cooling after finish rolling	Annealing temp. [°C]	Average Grain size [µm]
	Fin	Coil			
IV-1	800	611	Water laminar	600	6.1
IV-7	800	570	Water laminar	550	6.8
IV-11	750	606	Water laminar	600	6.6
IV-16	750	564	Water laminar	550	6.5
IV-2	800	606	Water laminar	-	5.5
IV-20	800	540	Water laminar	-	5.0
IV-13	750	620	Water laminar	-	6.8
IV-21	750	545	Water laminar	-	5.7
IV-3	800	600	Compressed air	600	7.2
IV-6	800	550	Compressed air	550	6.3
IV-12	750	600	Compressed air	600	6.6
IV-18	750	550	Compressed air	550	5.9
IV-4	800	600	Compressed air	-	6.9
IV-9	800	550	Compressed air	-	6.5
IV-14	750	600	Compressed air	-	6.1
IV-19	750	550	Compressed air	-	6.6

It is observed that the ferrite grain refinement is mostly the result of the deformed austenite below the recrystallization temperature and accelerated cooling after deformation; both processes increase the nucleation of ferrite phase [28]. Several increase mechanisms of nucleation rate of ferrite by deformation have been put forward.

These mechanisms include an interrelation between the increased nucleation rate of ferrite with the:

(a) bulges formed by local austenite grain boundary migration [29],

(b) formation of subgrains near the deformed austenite grain boundaries, and strain energy of the dislocations stored in the deformed austenite [30].The grain refinement is obtained by control of the rolling conditions sush as time, temperature and deformations during the whole production process. Grain refinement in steels is enhanced through a combination of controlled rolling and microalloying.

The primary grain refinement mechanism in controlled rolling is the recrystallization of austenite during hot deformation. Small additions of alloying elements like Nb, V and Ti result in the formation of carbonitrides in the microstructure. These very fine precipitates are effective in preventing grain growth. By the use of controlled rolling, recrystallization is retarded during the last passes. The average grain size after rolling according to the rolling schedule (IV) was found between 5.0 and 7.2µm. The different finish rolling temperatures as cooling strategies didn't affect much the grain size as the shares of recrystallized phases. Here the phase proportions were not investigated in detail.

CONCLUSIONS

In experimental investigations the deformation conditions at hot strip rolling should be simulated. The Object was a corporate optimisation of the rolling technique, to create advantageous microstructures and to improve the mechanical properties of the finished strip. The investigated material is used for weldable pipelines.

The finish temperature of the rolling experiments was verified between 850°C and 700°C. The results obtained in the laboratory with different techniques of rolled strips confirmed the conclusions, which were also found at the investigations of strips rolled in (Elh-ISF), that a reduced finish temperature improves mechanical properties of the final strip sush as yield strength and tensile strength. But the finish rolling temperature affects the mechanical properties only slightly.

Of far greater importance on the quality of the hot strips is the coil temperature. Different coil temperatures were simulated in the experiments by varying heat treatment temperature in a furnace (between 500°C and 630°C) and annealing times (between 30 minutes and 24 hrs) after finish rolling. All rolling experiments showed that a reduced coil temperature improves yield strength and tensile strength as well as reduction of area.

The relation between yield strength and tensile strength was not affected essentially. While the elongation marginal deteriorated at low coil temperatures. Maximum yield strengths of about 560 MPa and maximum tensile strengths of about 675 MPa, where reached consequently. Few strips with a low coil temperature of 550°C showed no yield strength, but tensile strength of more than760MPa. These strips had also a deterioration of elongation and toughness. So, coil temperature at 550°C or below are not recommendable. As a result of all investigations a possible finish rolling temperature for steel X70 between 850°C and 830°C is proposed. These would result in a coil temperature of about 560°C. An optimum balance between strength and toughness should be found. The restrictions of roughing, finishing and coil temperatures would also reduce the wide large range of properties of different strips. The average grain sizes after rolling was found between 5 and 7.2µm. The different finish rolling temperatures as well as the cooling strategies didn't affect much the grain size when recrystallisation is taking place.

Acknowledgements

The authors would like to express their gratitude for the financial support by CERCIM/SIDER Center. Experiments on the Thermomechanical simulator were performed at Freiberg University, and this is gratefully acknowledged.

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