DCCT DIAGRAMS OF X70 PIPELINE GRADE INFLUENCED BY PREHEATING CONDITIONS

SEILLIER Yann[1], SCHINDLER Ivo[1], KAWULOK Rostislav[1], OPĚLA Petr[1],
TUROŇ Rostislav[2], JURČA Radek[2]

[1]*VSB - Technical University of Ostrava, Faculty of Metallurgy and Materials Engineering, Czech Republic, EU, seillier.yann@gmail.com, ivo.schindler@vsb.cz*
[2]*Třinecké železárny, a.s., Třinec - Staré Město, Czech Republic, EU, rostislav.turon@trz.cz*

Abstract

Heavy seamless tubes are produced by the Big Mannesmann Rolling Mill in Třinecké železárny plant. Their final microstructures and properties are not only given by steel chemistry, they are also largely influenced by cooling conditions after finish-rolling. Continuous cooling transformation (CCT) analysis of various steel grades has been increasingly an object of the researchers' attention. Nevertheless, the phase transformations are also affected by the initial microstructure (e.g. austenite grain size) and previous deformation (ratio of residual hardening and softening). For that reason the DCCT diagrams (including the influence of defined deformation e = 0.35) of the HSLA steel X70 were designed for two states of the initial microstructures. In rough outline, the tested steel has the following chemical composition: 0.16 C - 1.0 Mn - 0.2 Cr - 0.05 V - 0.03 Nb (in wt. %). Phase transformations in the course of cooling were studied by dilatometer tests (performed in the Gleeble 3800 hot deformation simulator, to wide cooling rates range, from 0.15 °C/s to 80 °C/s), metallographic analysis and hardness as well as micro-hardness measurement. Coarse gamma grains (G = 3) were produced by high-temperature preheating (1280 °C) of the dilatometer samples made from the continuously cast bloom with diameter of 400 mm. Fine initial microstructure (G = 11) was obtained by severe laboratory hot rolling of the cast material and normalizing. Lower temperature of austenitization (900 °C) has been set in the dilatometer to make such samples from the normalized material. Great differences were observed, some coarse-grain samples containing acicular ferrite, whereas mixture of martensite and austenite appeared in the fine-grain samples cooled with rates of at least 12 °C/s. Lower heating temperature substantially accelerated the gamma/alpha transformation at high cooling rates.

Keywords: Seamless tubes; HSLA steel; controlled rolling; continuous cooling transformation diagrams; microstructure

1. INTRODUCTION

Steels are largely used for the engineering purposes such as pipelines for few centuries [1]. Last decades, micro-alloyed steels have emerged and have been continuously improved [2, 3]. For this class of steel, more than qualities brought by chemical composition, a mastered making process gives great mechanical properties and weldability. Application of these steels on seamless tubes obtained by controlled rolling leads to specificities of input setting parameters [4, 6]. Among the input parameters some are critical, such as initial microstructure [7, 8] (e.g. austenite grain size). Because development of the optimum setting in the plant would slow down production and has unnecessary cost, this document offers analysis of the initial microstructure and previous deformation effect by laboratory work. All experiments in order to obtain DCCT diagrams and their correlation have been processed from a sample of a continuously cast bloom made by Třinecké železárny [9, 10]. The specific HSLA steel used for this seamless tubes production is the X70 grade which means that its yield strength is 70 ksi = 482 MPa. The chosen alloy is made from the following chemical composition: 0.16 C - 1.0 Mn - 0.2 Cr - 0.05 V - 0.03 Nb (wt. %).

2. EXPERIMENT DESCRIPTION

All experiments contained in this document have been processed from a sample of a continuously cast bloom of a diameter of 400 mm made by Třinecké železárny. **Figure 1** shows the path used to obtain DCCT diagrams and comparison from the given sample.

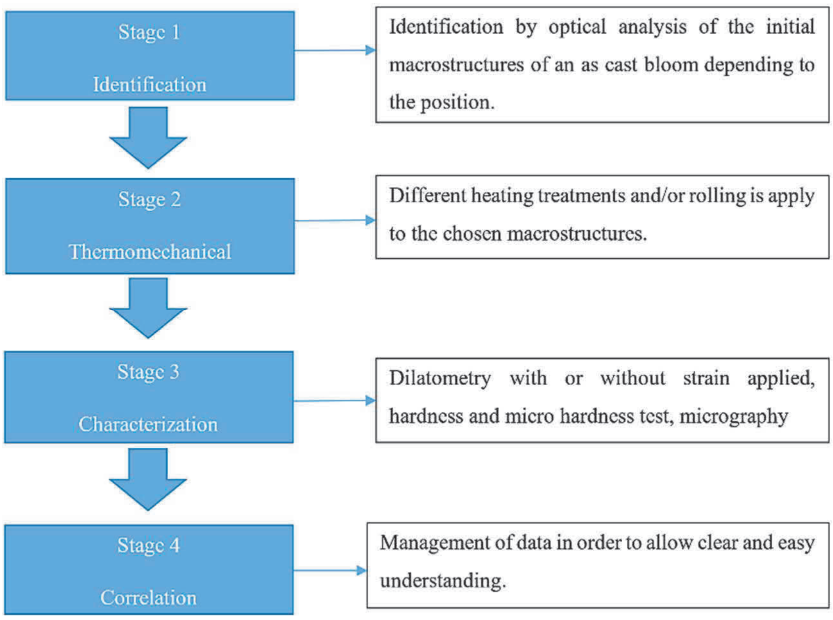

Figure 1 Flowchart of the practical analysis

During cooling process (which includes solidification) of the alloy in the mold, the temperature over the position is heterogeneous. In fact, the thermal insulation gradually decreases when approaching the wall of the mold. Local cooling rates lead to disparate level of precipitation, segregation, diffusion.

Said by simpler words, there are uneven physical and chemical properties of steel along the section of the casted bloom. Nevertheless, it is important to notify that the structures are not frozen once to ambient temperature, application of strain or/and high temperature can make them evolve.

From **Figure 2**, it is possible to give coarse estimation of grain sizes and share of the phases. In accordance with literatures [1, 2] the optical analysis of the bloom lighted up three kinds of macrostructure in the selected cross section which are:

1) Chill zone: Melted steel was in direct contact with much colder mold's wall. It provoked high cooling rate in the circumference of the bloom. This high cooling rate produced small and equiaxed grains.
2) Columnar zone: as the cooling decreases compared to the chill zone, the grains are bigger and preferentially oriented in the direction of the heat flow. The presence of dotted lines in the direction of the cooling rate gradient is observable in the unetched state.
3) Center equiaxed zone: in the center of the bloom, there is a super cooling phenomenon making relatively small equiaxed grains. Also because the volume of liquid is slightly lower than the volume of free space due to the reduction of temperature, it appears defects as cracks or porosity.

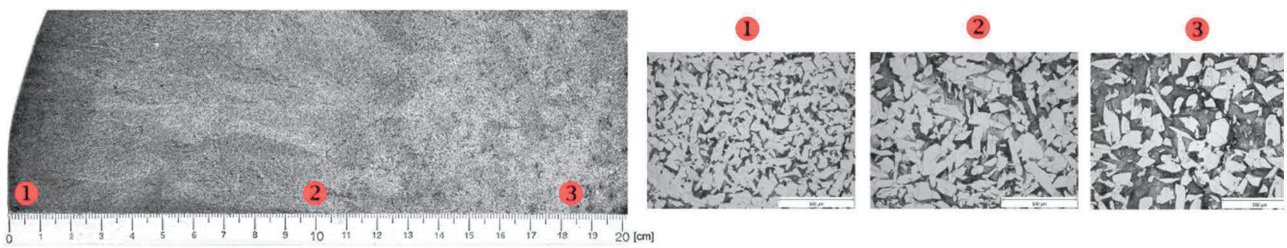

Figure 2 Overview of etched microstructures over the position in the continuously cast product

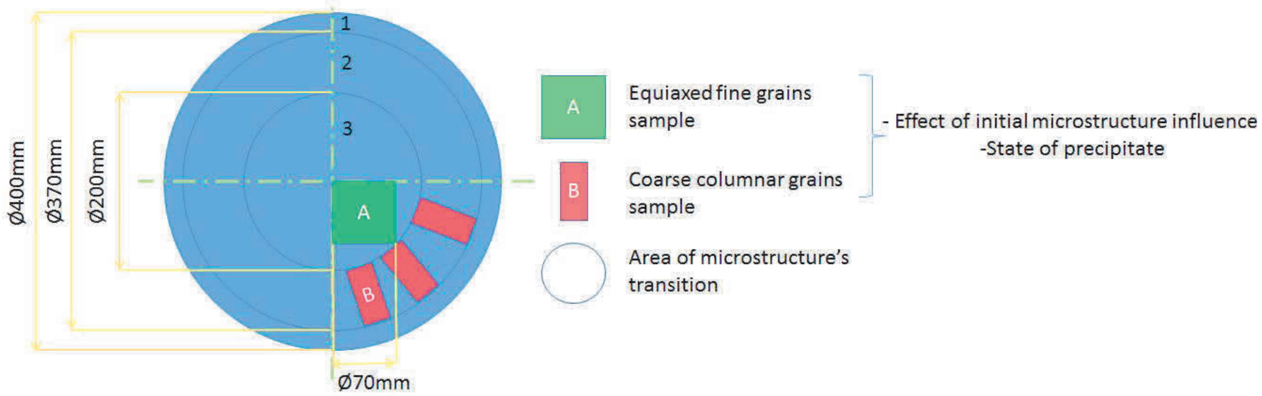

Figure 3 Positions of the samples on the bloom cross section

After determination of the given structures and their locations, samples has been taken off from the center of the equiaxed zone and from the columnar one (**Figure 3**). The purpose of this experiment is mainly to deduce influence of initial microstructure on the final product. In order to do it, it has been done a comparison between fine and coarse microstructures, and comparison of a cooling into the dilatometer while applying deformation. Deformation was applied at 900 °C by a plastometer Gleblee 3800 with amount of equivalent strain and strain rate respectively equal to e = 0.35 and $\dot{e}$ = 1 s^{-1}.

Samples A have been processes by rolling and normalizing to refine the already fine grains (compared with the samples B). The given samples were hot rolled by 16 passes between 1280 °C and 900 °C. More theoretically, during this phase, the austenite grains are stretched by deformation. These conditions promote recrystallization that means, the storage of the energy by the nucleation of brand new grains in the metal. The output part contains more grains than before, or in other words, finer grains size. In addition, the rolled products were normalized at 900°C and the final secondary grain size about 8 µm was obtained. Dilatometry samples machined from this material have been austenized at 900 °C / 180 s.

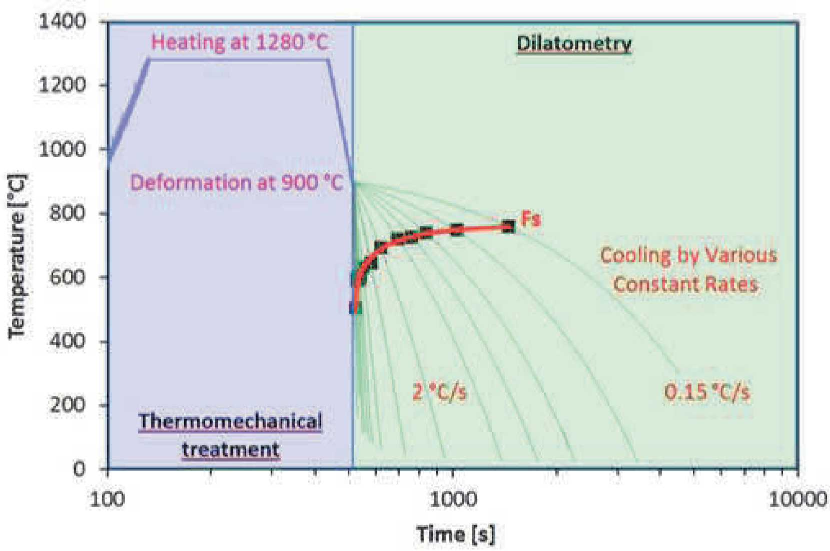

Figure 4 Thermic cycle for grains growth in the sample B

Dilatometry samples B have been heated at a rate of 10 °C/s to 1280 °C / 300 s to increase the grain size of the columnar zone. Such a temperature encourages the grains growth. The sample is then cooled down to 900°C at a rate of 5 °C/s (**Figure 4**). Temperature is then stabilized during a dwell time of 5 s and the defined compression deformation is applied. In real conditions of the seamless tubes production a deformation is also applied - it corresponds to finish of pilgrim rolling. Deformation becomes therefore an interesting parameter to take into account. After this preheating cycle dilatometry measurements can start.

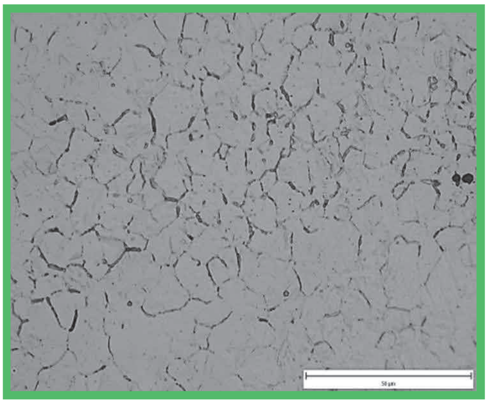

Figure 5 Initial fine microstructure

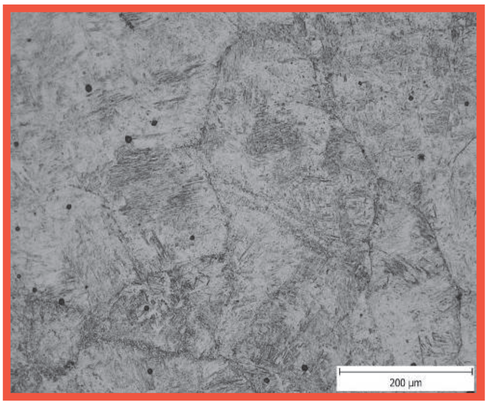

Figure 6 Initial coarse microstructure

Combining the location in continuously cast bloom with the described material's thermomechanical treatment, initial austenite grain size of approx. 10 µm was obtained at low-temperature heating and more than 130 µm at high-temperature heating of the dilatometry samples (see **Figures 5** and **6**). All pictures in this document framed by green line relate to fine initial microstructure and red line frame for the coarse microstructure.

3. DISCUSSION OF RESULTS

With the aim to come to a conclusion, samples have been through several tests such as dilatometry, metallography, micro-hardness and hardness [11, 13] (**Figure 7**). All information have been correlated together to obtain further information as phase share, mechanical properties and transformation temperatures.

By metallography of the dilatometry samples after cooling,the following phases has been found [14] : ferrite, (with equiaxed and acicular morphology), pearlite, M/A (which is mixture of martensite and austenite), martensite and bainite. HSLA steels show their highest qualities when their final microstructure is fine and even ferritic/pearlitic; cooling rate to obtain such microstructures is relatively low [15]. Indeed, it produces material with high thoughness, yield strength and weldability. Martensite and bainite microstructures make materials with high hardness and low thoughness.

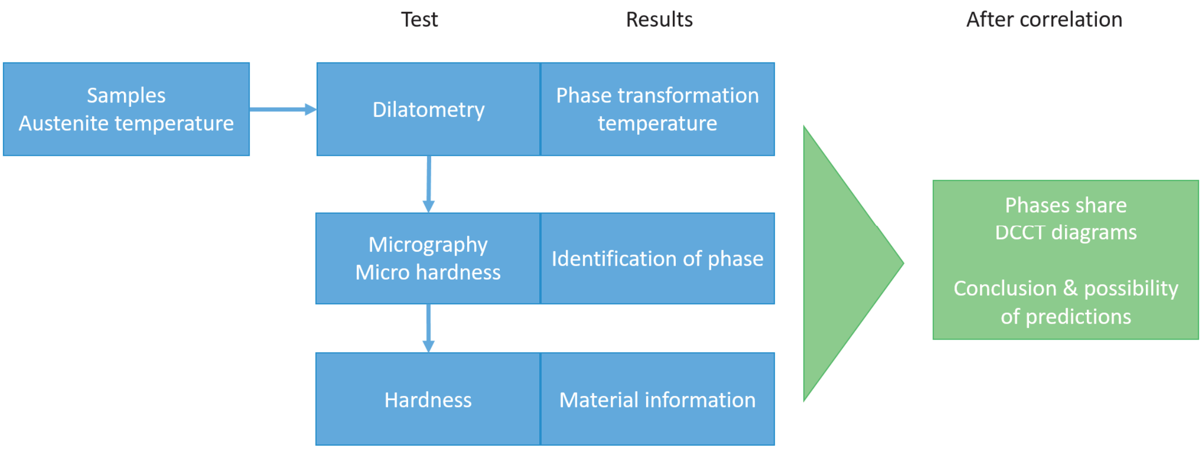

Figure 7 Different assessments and data they offer

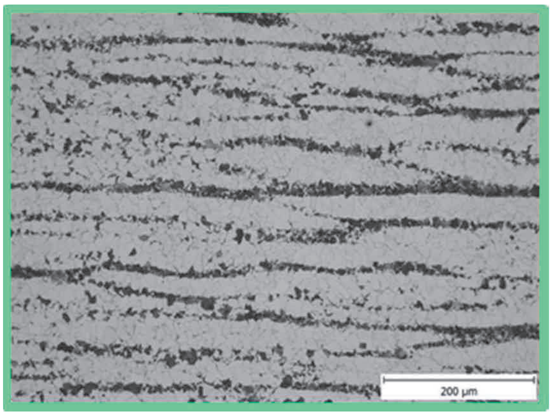

Figure 8 Low cooling rates results, from fine-grain initial microstructure

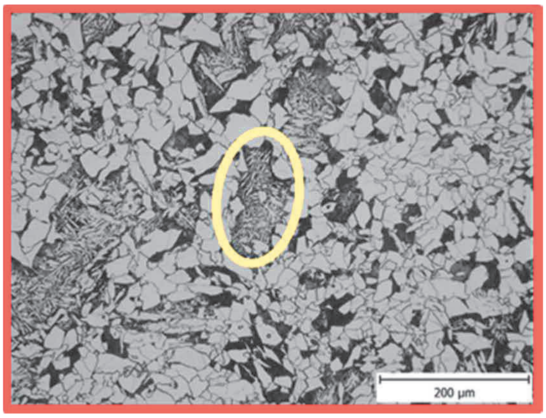

Figure 9 Low cooling rates results, from coarse-grain initial microstructure

The micrograph in **Figure 8** comes from a sample with fine initial microstructure cooled at a cooling rate of 0.15 °C/s. It is observable that two phases forms the assessed sample. Long horizontal dark strips are known to be pearlite, which used to be lamellar. In other hand lighter grains are known to be equiaxed ferrite. This structure is typical and quite easy to identify with little bit of experience, it is usual on iron cooled on relatively slow cooling rate. This kind of microstructure used to be known as ductile with low hardness. Mechanical performances of this microstructure mainly depends on grain size and alloying element content.

The micrograph in **Figure 9** comes from the sample with coarse initial microstructure cooled at a cooling rate of 0.7 °C/s. Here there are observable 3 types of morphologies, the equiaxed ferrite, the acicular ferrite (needle shape, see yellow circle on the **Figure 9**), and a darker mixture. The darker mixture has been identified later by hardness test as pearlite and acicular ferrite.

Phase share assesment **Tables 1, 2** and **Figures 10, 11** reveal by comparison that samples produced from fine austenite:

- Have simplier microstructure, they are not composed of ganular bainite and acicular ferrite, which have potentially negative effects.
- At high cooling rates, martensite share stay lower.
- Ferrite share is always higher.

Table 1 Hardness and structure parameters after cooling of the finer structure

Cooling rate [°C/s]	Hardness HV 30	Ferrite [%]	Ferrite Grain ø [µm]	Pearlite+M/A [%]		Martensite+Bainite+M/A [%]
0.15	147	79	11.9	21		
0.5	151	77	11.6	23		
4	174	68	9.3	32		
12	221	54		46	(M/A)	
35	238	45			(M/A)	55
80	266	32			(M/A)	68

Table 2 Hardness and structure parameters after cooling of the coarser structure

Cooling rate [°C/s]	Hardness HV 30	Ferrite+ Acicular Ferrite [%]		Ferrite Grain ø [µm]	Pearlite+ Granular Bainite[%]		Martensite+ Bainite [%]
0.3	166	68		17.1	32		
0.5	187	65	(AF)	16.3	35		
0.7	193	62	(AF)	15.9	38		
1	198	60	(AF)		40		
4	229	52	(AF)		48	(GB)	
12	265	40	(AF)		60	(GB)	
20	333	20	(AF)				80
35	389	5					95
60	413	1					99

Hardness is one of the basic values of material engineering, therefore it is perfectly mastered experiment [1, 2]. Additionally, it has been found that there exists a relation between the hardness and the ultimate tensile strength. Unfortunately, hardness is generally opposed to ductility and toughness. Rupture of hard materials have also often the disadvantages to be brutal and sudden. Regarding the **Figure 10** and basic material engineering knowledge, fine initial microstructure should procure much higher toughness and ductility. Indeed, hardness is higher for every cooling rates (especially the highest) of the sample produced with a coarse initial austenite. Effects of fine final microstructure are well known by metallurgist and beneficial for tubes' engineering purpose see **Figure 11**.

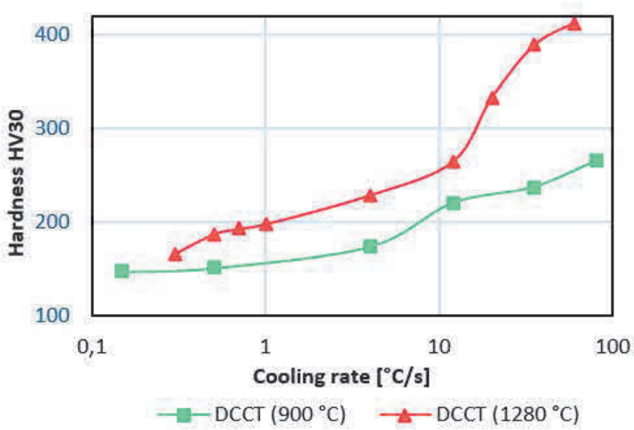

Figure 10 Hardness comparison of the samples

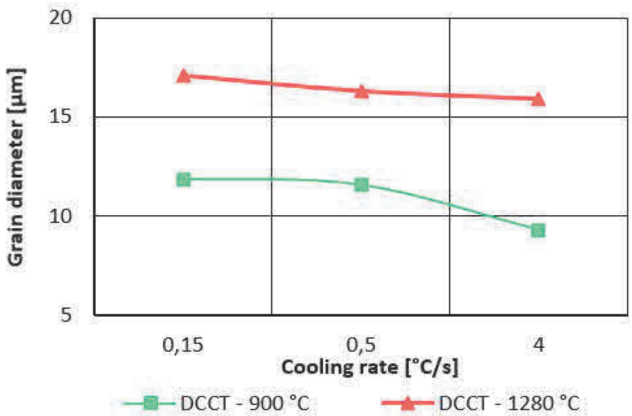

Figure 11 Ferrite grain size comparison

Based on the combination of dilatometry tests, metallography and hardness measurement, two DCCT diagrams could be assembled. Temperatures of phase transformations were variously influenced by the initial microstructure - see **Figure 12**. DCCT diagrams correlation revealed that the finer initial microstructure:

- Increases the ferrite start temperatures, mainly at higher cooling rates.
- Broadens the range of pearlite forming temperatures
- Supports the formation of bainite, pearlite and ferrite.

In the other hand at the lowest cooling rates results are similar for both initial grain sizes and martensite-start temperature is not influenced at cooling rate higher than 25 °C/s.

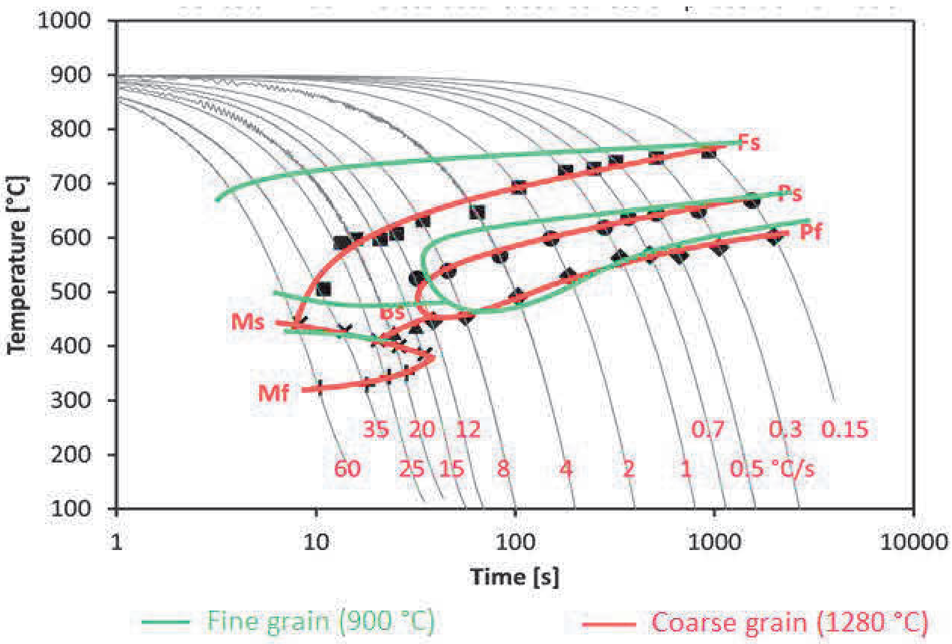

Figure 12 Correlation of DCCT diagrams influenced by the austenite grain size

4. CONCLUSIONS

DCCT diagrams of the pipeline HSLA steel X70 were designed for two states of the initial microstructures with a grain size of more than 130 µm or 10 µm. Coarse initial microstructure use to significantly increase hardness, and more likely decrease substantially ductility and toughness. By looking deeper in microstructures, it appears that at low cooling rates (< 0.7°C/s) the initial grain size does not influence the phase transformation kinetics. Indeed, coarse austenite increases the secondary grain size but above all, it brings high heterogeneity of the ferrite, which reduces considerably mechanical properties of the rolled part. At higher cooling rates results are totally different, transformation temperatures of ferrite and pearlite is kept up and remain steady from the fine initial microstructure. At the cooling rates > 12 °C/s, fine initial microstructure supports formation of ferrite and bainite, whereas coarser grains support formation of martensite. Great differences were observed, some coarse-grain samples containing acicular ferrite, whereas mixture of martensite and austenite appeared in the fine-grain samples cooled with high rates. More generally speaking, fine initial microstructure strongly supports ferrite formation at the expenses of other phases. Subsequently it gives more homogenous secondary microstructure and arguably better mechanical characteristics (even though it lowers hardness). Lower heating temperature substantially accelerated the gamma/alpha transformation at high cooling rates. Management of the initial grain size is a key parameter of a stable and resilient production using this steel. In the case of coarse-grain initial microstructure, it is necessary to refine it by using the proper thermomechanical treatment (e.g. controlled rolling [16]) at the heavy seamless tubes production by the Big Mannesmann Rolling Mill in Třinecké železárny plant.

ACKNOWLEDGEMENTS

The research was supported by the grant projects FV10253 (Ministry of Industry of the Czech Republic), LO1203, SP2017/62 and SP2016/103 (Ministry of Education of the Czech Republic).

REFERENCES

[1] BAÏLON, J. P., DORLOT, J. M. *Des matériaux*. 2003.

[2] CAMPBELL, F.C. *Elements of metallurgy and engineering alloys*. 1. Materials Park, Ohio: ASM International. 2008.

[3] VERLINDEN, B., et al. *Thermo-Mechanical Processing of Metallic Materials*, Volume 11 (Pergamon Materials Series). Oxford: Elsevier, 2007.

[4] MATROSOV, M. Y., ÉFRON, L. I., KICHKINA, A. A., LYASOTSKII, I. V. A study of the microstructure of niobium-microalloyed pipe steel after different modes of controlled rolling with accelerated cooling. *Metal Science and Heat Treatment*. 2008, vol. 50, no. 3-4, pp. 136-141.

[5] MONTMITONNET, P., AUBIN, J. L. Formage des tubes métalliques - Procédés - Analyse détaillée du laminage à pas de pèlerin; PDF; *Techniques de l'ingénieur; Matériaux* | *Mise en forme des métaux et fonderie*; 10 march 2009; Réf.: M3068.

[6] PUSSEGODA, L. N., HODGSON, P. D., JONAS, J. J. Design of dynamic recrystallisation controlled rolling schedules for seamless tube rolling. *Materials Science and Technology*. 1992. vol. 8, no. 1, pp. 63-71.

[7] PUSSEGODA, L. N., YUE, S., JONAS, J. J. Laboratory simulation of seamless tube piercing and rolling using dynamic recrystallization schedules. *Metallurgical Transactions A*, 1990, vol. 21, no. 1, pp. 153-164.

[8] BRENSING, K.H., SOMMER, B. *Steel Tube and Pipe Manufacturing Processes*. http://www.smrw.de/files/steel_tube_and_pipe.pdf

[9] Třinecké železárny-Moravia Steel, http://www.trz.cz

[10] Třinecké železárny-The tube Mill, http://www.tube.cz/

[11] Standard Test Methods for Determining Average Grain Size, 2004, Designation: E 112 - 96e3, ASTM International.

[12] MURAT, M. *Dilatométrie*. 1. Techniques de l'ingénieur. 1984.

[13] POKORNY, J., POKORNY, A. *Métallographie*. 2. Techniques de l'Ingénieur. 1994.

[14] HAUSEROVA, D., JIRKOVA, H., MASEK, B. Investigation of phase transformations in high-strength low-alloyed steel. *DAAAM International Vienna*. 2009.

[15] TAMURA, I., et al. *Thermomechanical Processing of HSLA Steels*. London: Butterworths, 1988.

[16] ŠEVČÁK, V. et al., Structure Forming Processes at Controlled Rolling and Cooling of Seamless Tubes Made of HSLA Steel (in Czech), Hutnické listy, 2016, vol. 69, no. 6, pp. 39-46.