MICROSTRUCTURE DEVELOPMENT IN THE PROCESS OF CONTROLLED ROLLING AND COOLING OF A Nb-MICROALLOYED PIPE STEEL

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Abstract

Examination of structure-forming processes of HSLA steel with 0.025 % Nb was performed in relation with rolling of the heavy seamless tubes in the Big Mannesmann mill. Based on the dilatometry data, a DCCT diagram after deformation 0.35 at temperature of 900 °C was designed. Hardness HV30 of value 157 was determined for low cooling rate of 0.2 °C·s^{-1} and the structure was consisting mostly of ferrite and pearlite. For faster cooling with rate of 60 °C·s^{-1} the hardness was equal to 404 with entirely martensitic structure. Non-recrystallization temperature was determined by the rolling-cooling-quenching tests and metallography just above 850 °C. Finally, steel samples were subjected to temperature controlled rolling and cooling with the rate of 0.25°C·s^{-1} in the laboratory reversing mill with the working rolls' diameter of 350 mm. Grain refinement as well as homogenization of the final microstructure was observed after lowering the finish rolling temperature in the interval from 990 to 850 °C. Greater effect of decreasing finish rolling temperatures was observed below 890 °C as a result of deceleration of the recrystallization kinetics due to the precipitation during the cooling phase and getting closer to non-recrystallization temperature. The smallest secondary grain size of 17 μm was achieved despite the initial coarse-grained structure (created by preheating at 1280 °C), low degree of material deformation and slow final cooling. Low-temperature finish rolling resulted in a significant increase in the roll forces - approximately by 50 % when comparing the results of experiments performed at temperatures of 990 °C and 850 °C.

Keywords: HSLA steel, DCCT diagram, non-recrystallization temperature, finish rolling temperature, microstructure.

1. INTRODUCTION

The objective of the work was to investigate the selected structure-forming processes during cooling of niobium-alloyed steel used for seamless tube rolling with the use of physical methods. Production of the thick-walled tubes in the Big Mannesmann mill was simulated. The investigated HSLA steel had the chemical composition 0.17 C - 1.1 Mn - 0.2 Si - Al 0.028 - 0.011 N - 0.025 Nb (in wt. %). The experiment was conducted in three main stages. First, a continuous cooling transformation diagram DCCT was compiled (with the influence of the previous deformation). This was followed by determination of the non-recrystallization temperature (NRT) of austenite. The experiment was completed by a simplified simulation of forming with various finish rolling temperatures and with the determination of its effect on the resultant microstructure after slow cooling.

The relation between finish rolling temperature and NRT is generally determined by the type of applied thermo-mechanical treatment, the purpose of which consists in the achievement of the finest possible secondary grain. Forming of austenite in the non-recrystallization region is more and more often studied and used not only for traditional HSLA steels [1, 2], but also for low-carbon bainitic steels [3], pipeline steels [4] and TRIP steels [5].

The importance and practical significance of DCCT diagrams at a selection of the optimum cooling technology excels in their comparison with traditional CCT diagrams for identical steels - from the recent works see e.g.

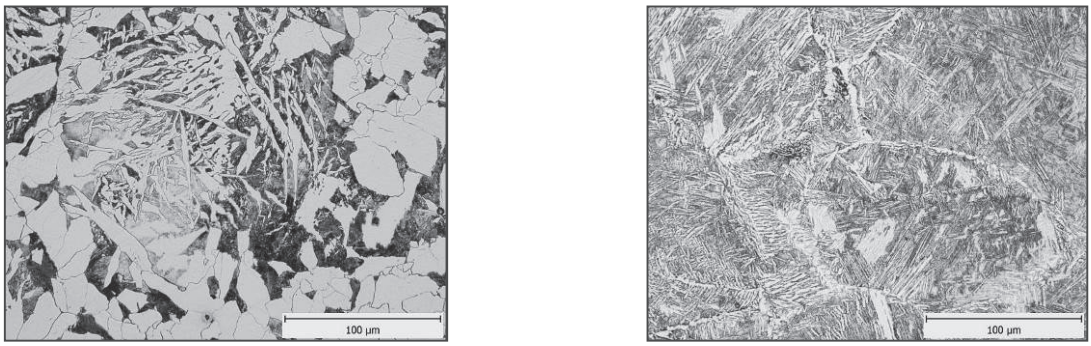
[6 - 8]. It is evident that the kinetics of individual phase transformation is significantly affected not only by the cooling rate, but also by the size of the initial austenite grain, magnitude of previously accumulated deformation and the rate of elimination of its influence by softening during cooling; quantification and generalization of these effects is, however, extremely difficult [9].

2. DCCT DIAGRAM

Dilatometric tests used cylindrical samples with a diameter of 6 mm and a length of 86 mm. Due to the high heating temperature necessary for dissolution of niobium carbo-nitrides, it was necessary to use contact dilatometric system of the plastometerGleeble 3800 and platinum thermocouples. The samples were heated at the rate of 10 °C/s to a temperature of 1280 °C and after a dwell of 300 s they were cooled down a rate of 5 °C·s^{-1} to a temperature of 900 °C. At this temperature a compressive deformation of the magnitude of 0.35 was applied at the strain rate of 1 s^{-1}, followed by cooling at a constant cooling rate ranging from 0.2 to 60 °C·s^{-1}, during which the sample dilatation was measured. Analysis of the recorded curves in the CCT software determined the temperatures of phase transformations with consideration of the results of metallographic analysis and hardness measurement of the selected samples (see **Table 1)**.

Table 1 Effect of cooling rate on the occurrence of phase components and hardness

Cooling rate (°C·s^{-1})	Hardness HV30 (-)	Ferrite (%)	Pearlite (%)	Martensite (%)
0.2	157	69	31	0
0.7	181	65	35	0
3	208	57	43	0
6	225	49	51	0
10	243	43	57	0
20	320	20	0	80
40	381	5	0	95
60	404	0	0	100



a) 0.7 °C/s b) 20 °C/s

Figure 1 Influence of cooling rate on the microstructure of the samples after dilatometry

Figure 1 presents microstructures of selected samples. Their evaluation is complicated by the occurrence of two morphologies of ferrite - equiaxed and acicular, already at the lowest cooling rates (**Figure 1a**). Acicular ferrite is present inside the grains and chains of equiaxed ferrite are found at the grain boundaries at cooling rate around 6 °C·s^{-1}. With an increase of the cooling rate the acicular ferrite inside the grains is gradually replaced by martensite (**Figure 1b**), however, only after cooling at the cooling rate of 60 °C·s^{-1} the structure is composed solely of martensite.

As it is apparent from **Figure 2**, the hardness of the samples increases in direct proportion to the decrease in the share of ferrite and with the increase in the share of pearlite and martensite in the structure.Illustration of cooling curves in traditional semi-logarithmic scale time - temperature was complicated by the different austenitizing temperature and deformation of the samples. In order to obtain the final form of the DCCT diagram in **Figure 3**, it was necessary to subtract the time period corresponding to the phase of preheating and deformation. It is obvious that after usual rates of the free cooling it is possible to expect the final ferritic-pearlitic structure. Martensite appears in the structure only after cooling rates exceeding approx. 20 °C·s^{-1}. Legend in **Figure 3**: Fs - start of ferrite transformation; Ps - start of pearlitic transformation; Pf - end of pearlitic transformation; Ms - start of martensitic transformation; Mf - end of martensitic transformation.

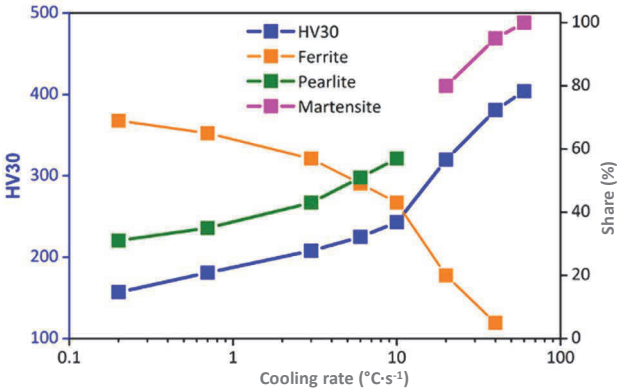


Figure 2 Influence of cooling rate on the HV30 hardness and shares of structural components

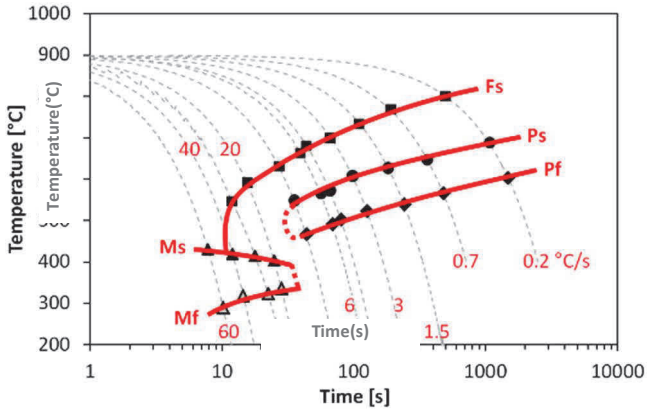


Figure 3 DCCT diagram (deformation at 900 °C)

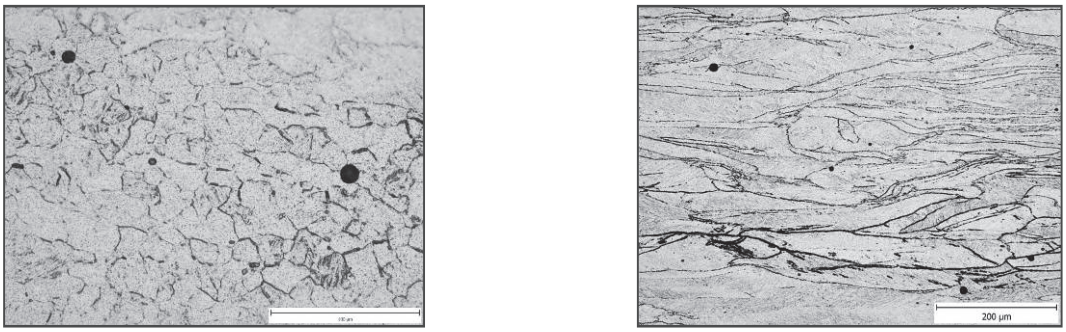
3. NON-RECRYSTALLIZATION TEMPERATURE OF AUSTENITE

After a uniform preheating at 1280 °C/30 minutes in an electric furnace the flat samples with a thickness of 12.5 mm and a width of 50 mm were freely air cooled to the temperature of the first pass (i.e. 900-1100 °C). After a ten-minute stabilizing dwell in the second or third resistance furnace (heated to the appropriate temperature for the first pass), the sample was flat-rolled by two reductions on the semi-continuous reverse stand of the laboratory rolling mill. The used smooth part of the rolls had a diameter of 350 mm and speed rolls of 18 rpm were chosen. The first reduction of 40 % was supposed to achieve a thickness of 7.5 mm and possibly to invoke a stress-induced precipitation. Immediately following second reduction (also of 40 %) resulted in the rolling of the final thickness of 4.5 mm. Two temperature scanners situated right before and after the stand measured the surface temperature of the sample during the rolling and also during the free cooling, immediately following after the second pass. After approx. 10 seconds from the last reduction the sample was quenched in water. Metallographic development of austenitic grain in longitudinal vertical section of the quenched and tempered rolled product (350 °C / 30 minutes / cooling in the furnace) then enabled obtaining of information about the evolution of static recrystallization during the period of cooling before quenching. **Table 2** presents the temperatures characterizing the evolution of the rolling of individual samples.

Micrographs in **Figure 4** document the selected results of demanding metallographic analyses at various magnifications. Finish rolling at a temperature of 1010 °C lead to a complete recrystallization of the deformed structure (**Figure 4a**), as evidenced by equiaxed grains. After reduction performed at the temperatures from 960 to 880 °C, the structure was in various degrees and in relatively heterogeneous manner composed of deformed and equiaxed grains. Only in the sample that was finish rolled at a temperature of 850 °C no signs of recrystallization were found (**Figure 4b**), and the resultant structure consisting of elongated grains corresponded to an intense cold forming. The temperature of non-recrystallization of the investigated steel is therefore under these conditions just above the value of 850 °C.

Table 2 Surface temperatures at rolling and cooling of individual samples

Sample	Temperature at the 1st reduction (°C)	Temperature at the 2nd reduction (°C)	Temperature prior to quenching(°C)
X1	1100	1010	860
X2	1040	960	835
X3	990	925	805
X4	960	900	795
X5	930	880	785
X6	900	850	760



a) 1010 °C b) 850 °C

Figure 4 Initial austenitic grain in quenched samples in dependence on the 2nd pass temperature

4. INFLUENCE OF FINISH ROLLING TEMPERATURE ON THE MICROSTRUCTURE

A strategy of a simplified physical simulation involving the three pass rolling of flat samples with an initial thickness of 12.5 mm to the final thickness of 7.0 mm was chosen. Rolling was carried out on the smooth part of the rolls of the reverse stand using the working rolls with a diameter of 350 mm. After a uniform preheating at 1280 °C / 30 minutes in an electric furnace, the samples were immediately subjected to the first height reduction of 22 %, at the speed of rolls of 12 rpm. This was followed by reduction of temperature of the rolled product freely on air until it reached the temperature set for the second (and analogously for the third) reduction of approx. 15 %, or 16 %, imitating low-temperature finish rolling; the speed of rolls was 13 rpm. The surface temperature was measured by temperature scanners. The required temperatures T2 and T3 for both finish rolling passes are shown in **Table 3**.The diagram in **Figure 5** documents the progress of in time of the experiment with the lowest finish rolling temperatures. The rolled products were slowly cooled in the electric resistance furnaces programmed at the programmed cooling rate of 0.25 °C/s, which simulated in a simplified manner the cooling of the rolled stock with a thickness of 40 mm on air. Cooled down rolled products were in the middle of their width cut in a manner enabling an analysis of microstructure in longitudinal vertical section (in the direction of rolling) on metallographic polished sections.

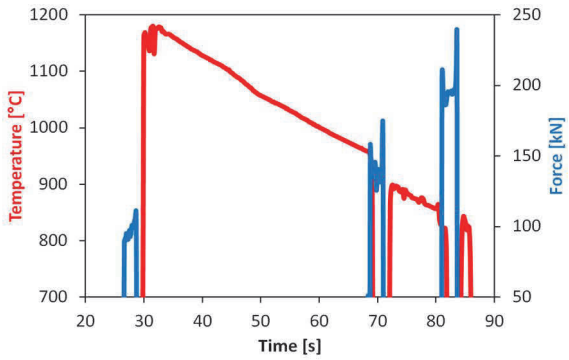
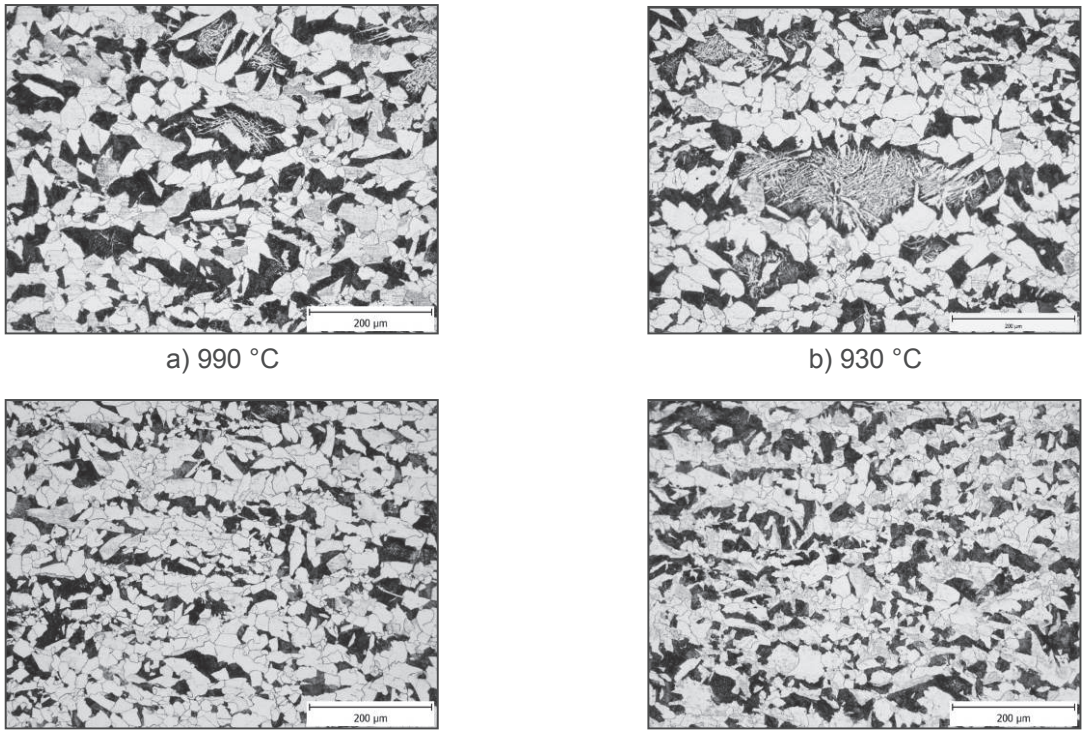


Figure 5 Time dependence of the surface temperature and rolling force registered during rolling of the sample R5

Table 3 Values of nominal surface temperatures for the 2nd and 3rd passes

Sample	T_2 (°C)	T_3 (°C)
R1	1100	990
R2	1030	930
R3	990	890
R4	970	870
R5	950	850

Selected micrographs from the area of half the height of the rolled stock are shown in **Figure 6**. The structure is in agreement with the DCCT diagram in **Figure 3** formed in all cases by equiaxed ferrite and pearlite, however, after two highest finish rolling temperatures, also an acicular ferrite was discovered (with particularly distinct formations in the case of the sample R2 - see **Figure 6b**). The expected band structure in the direction of rolling was not manifested. Reduction of the finish rolling temperature led to a gradual grain refinement, but an influence of the finish rolling below NRT was not significantly reflected - compare **Figures 6c** and **6d**.



a) 990 °C b) 930 °C

c) 870 °C d) 850 °C

Figure 6 Influence of the finish rolling temperature T_3 on the resulting microstructure

All the samples contained approximately 65 % of ferrite and 35 % of pearlite. The grain size was measured by the linear method, and the results were compiled into a diagram presented in **Figure 7**.

A more intensive drop of the grain size at a finish rolling temperatures below approx. 890 °C is probably due to deceleration of recrystallization during the phase of cooling down of the rolled products, caused by precipitation of niobium carbo-nitrides. This is in good agreement with the published results, see e.g. [10] - strain-induced precipitation of NbC runs the most rapidly at approx. 900 °C.

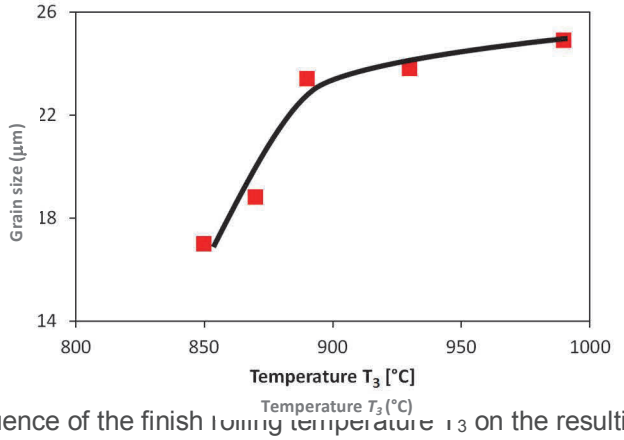


Figure 7 Influence of the finish rolling temperature T_3 on the resulting grain size

5. CONCLUSIONS

- For HSLA steel with niobium, used for thick-walled seamless tube Mannesmann rolling, a DCCT diagram was assembled after austenitization at a temperature of 1280 °C and deformation at 900 °C. In dependence on the cooling rate, the structure consists of two different morphologies of ferrite (equiaxed and acicular), of pearlite, and at a cooling rate exceeding 20 °C·s^{-1}also of martensite.
- Non-recrystallization temperature (NRT) of the tested steel was determined to be just above 850 °C.
- Laboratory rolled products with a thickness of 7 mm, slowly cooled in the furnace at a cooling rate of 0.25 °C·s^{-1}, had a structure composed only of ferrite and pearlite. The finish rolling temperatures of 930 °C and 990 °C resulted in a relatively coarse-grained and heterogeneous structure with large formations containing acicular ferrite.
- From the perspective of the resulting microstructure (i.e. homogeneity and grain size) the optimal results were achieved after finish rolling in the vicinity of NRT. Finish rolling at a temperature of 870 °C resulted in a grain size of 18.8 µm and in slightly more homogeneous structure than in the case of the finish rolling temperature of 850 °C (with a grain size of 17.0 µm). Such grain sizes are the result of significant coarsening of the structure during the high-temperature heating, low magnitude of material deformation and final slow cooling.
- Low-temperature finish rolling resulted in a significant increase in the roll forces (approx. by 50 % when comparing the results of experiments performed at finish rolling temperatures of 990 °C and 850 °C).

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