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Effect of Hot-Rolled Heavy Section Bars Post-Deformation Cooling on the Microstructure Refinement and Mechanical Properties of Microalloyed Steels

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Abstract: In the industrial practice—especially in the reverse rolling mills—heavy section products with stable mechanical properties (YS, UTS) and ductility (A, Z) but with an impact toughness (KV) at too low levels are often observed. The results presented in the present work concern the relationship between the parameters of the cooling process of rolled products made of microalloyed steels, with different chemical compositions (such as Al-N, Al-N-V, Al-N-Ti) and their mechanical properties. Special focus was put on the relationship between chemical composition, grain size and impact toughness at subzero temperatures. It is shown, that by introducing the restrictions towards more strict control of the levels of Al, Ti, V, and N, it can be ensured that the final parameters are not that sensitive to process parameters variations which, hence, provides the required mechanical properties and especially impacts on the toughness requirements for a wide range of section products. It was also found that by slight modifications of microalloying elements and proper control of the process parameters, it is possible to replace commonly used normalizing annealing heat treatment after rolling with normalizing rolling.

Keywords: microalloyed steel; normalizing rolling; cooling; impact toughness

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1. Introduction

In the case of structural steels intended for the production of welded structures, in addition to the required strength, special attention is paid to toughness. Meeting the expected values is especially required for the temperature range from −20 °C to −60 °C. Currently, in the industrial practice, in place of traditional continuous rolling of bars, the so-called normalizing rolling [1–4] is often applied. The effect of such rolling is homogenization and refinement of the microstructure, which improves the mechanical and technological properties of the rolled product. The use of such technology also allows for a significant reduction in production costs as a result of lower energy consumption due to the elimination of normalizing annealing [5].

The normalized rolling of bars takes place in two stages, i.e., first the rolling process is carried out at a temperature higher than the recrystallization stop temperature, then the products are cooled in air. The first step should be performed in such a way that both the required dimensional tolerances and the expected microstructure development is ensured. The latter is achieved by applying an appropriately selected deformation pattern and the times between individual rolling passes. At the same time, attention should be paid to precise control of the rolling temperatures so as to maintain the deformation process in the austenite range. Thanks to this two-stage process, it is possible to produce a fine-grained microstructure under natural cooling conditions, which has a direct positive effect on the expected mechanical properties, toughness, dimensional tolerances, and surface quality. This way, a significant improvement in the stability of the rolling process can also be observed.

Advanced structural steel products are produced not only on the basis of an appropriately designed rolling process, but also by modifying the chemical composition, especially through the use of precipitation processes (Ti, V) N and V (C, N) [2]. It is known that the presence of microalloying elements in steel influences on the development of the microstructure in many ways. For example, in the paper [6], authors proved that inhomogeneous deformation in the material could be caused by the interaction of the precipitations (Nb, Ti) and dislocations. The main effect is the refinement of the microstructure through the use of thermomechanical processing, i.e., an appropriate deformation pattern, rolling temperatures and time between passes, as well as controlled cooling operations [7]. It should be stated that the influence of VCN precipitates would be significantly higher in the microalloyed steel characterized by a lower Al content [8]. It is also important that addition of the Ti has a crucial role in the grain refinement as well as matrix strengthening and could work even better in the presence of Nb and V [9]. Another important effect of the presence of microalloying elements is the initiation of additional strengthening mechanisms, i.e., precipitation and solution strengthening. Thanks to these effects, not only is a higher strength obtained, but also good toughness at low temperatures [10], mainly due to the refinement of the microstructure.

The results presented in the present work concern the relationship between the parameters of the cooling process of rolled products made of microalloyed steels with different chemical compositions (such as Al-N, Al-N-V, Al-N-Ti) and their mechanical properties. First, S355J2 standard steel grade was used for preliminary study for defining the chemical composition modifications (within the EN 10025-2 standard for S355J2 grade) to ensure a repeatable level of impact toughness energy. In the industrial practice—especially in the reverse rolling mills—heavy section products with stable mechanical properties (YS, UTS) and ductility (A, Z) but with too low impact toughness (KV) levels are often observed. This is the challenge due to the various transfer times between consecutive caliber rolling stands. In the presented work it is shown that by introducing the restrictions towards more strict control of the levels of Al, Ti, V, and N, it can be ensured that the final parameters are not that sensitive to process parameters' variations which, hence, provides the required mechanical properties and especially the impact toughness requirements for a wide range of section products.

Moreover, it has been shown that by more strict control of the chemical composition of the tested steel grade (within the standard chemical composition variations), it is possible to produce finished products with properties similar to those manufactured by controlled, especially normalizing, rolling for the existing production conditions of a continuous bar mill without the need for additional normalizing annealing.

2. Materials and Methods

2.1. Materials

In the current work, in order to study the effect of post deformation cooling, four modifications of S355 steel with chemical compositions summarized in Table 1 were produced at COGNOR SA HSJ Branch (Stalowa Wola, Poland) using a continuous casting process. It should be noted that Mo content is not intended as a microalloying element here as it is residual from the steelmaking process.

Table 1. Chemical composition (in wt.%) of the studied steel grades.

Steel Group	C	Mn	Si	Cr	Ni	Mo	Ti	Alc	V	N_2
A	0.20	1.22	0.28	0.16	0.09	0.02	0.002	0.030	0.002	0.0070
B	0.19	1.29	0.22	0.08	0.11	0.03	0.001	0.029	0.002	0.0068
C	0.19	1.26	0.22	0.09	0.17	0.02	0.002	0.032	0.025	0.006
D	0.19	1.3	0.2	0.12	0.11	0.03	0.016	0.023	0.004	0.0071

2.2. D-700 Rolling Mill at COGNOR HSJ

The rolling process was realized at COGNOR SA HSJ Branch. The rolling process (Figure 1) begins with heating the billets that come from the continuous casting process. The billets are heated for a minimum of 5–6 h. After leaving the furnace, the heated billet goes to the descaler and then, by the means of a roller table, it is transferred to the zone of four reverse rolling stands. The deformation process in rolling stands takes place with the use of grooved rolls. Depending on the currently rolled profile, some rolling stands may be omitted. Moreover, the rolling sequence may vary depending on the dimensions and shape of the final product. After the rolled bars leave the last rolling stand, they are transferred to the area of the cooling bed, where the air cooling process takes place.

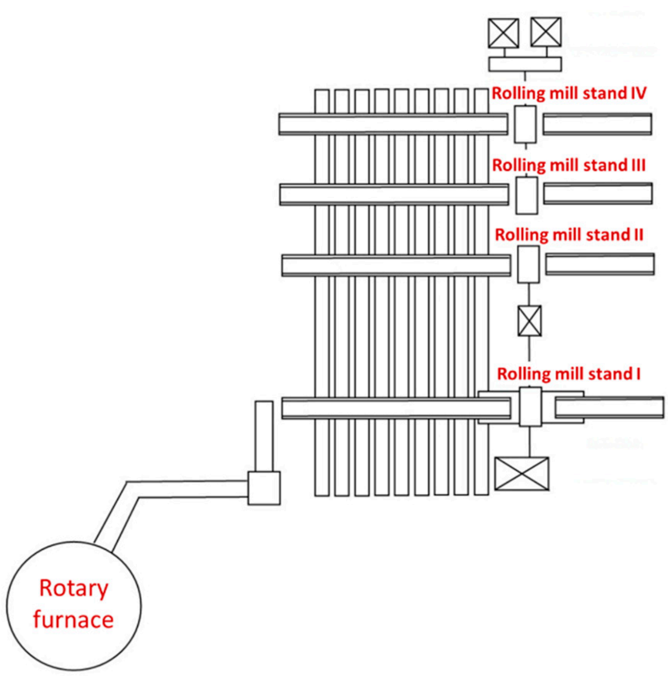
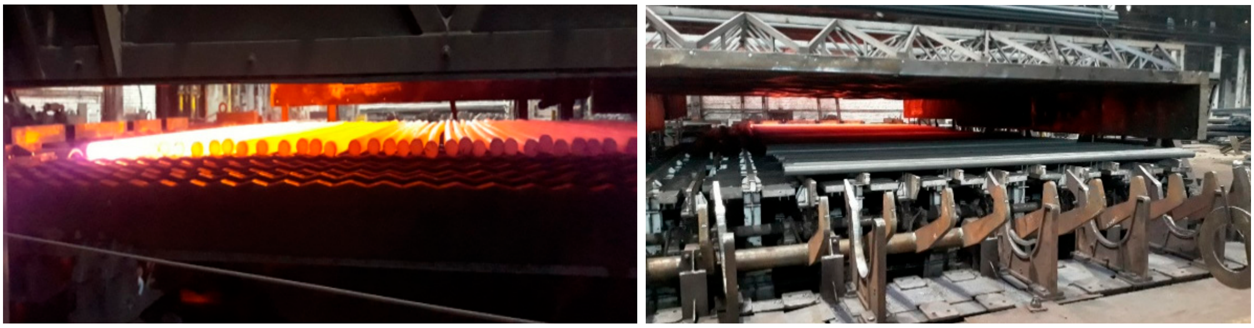


Figure 1. Scheme of the D-700 rolling mill used in the current work.

The cooling bed (Figure 2) consists of two transporters covered with thermal insulation covers. The cooler is adapted to cooling bars with a diameter range from 55 to 150 mm and square billets from 50 to 130 mm and a product length up to 8000 mm. The basic technical characteristics of the walking frame cooling bed are as follows: Transporter no. 1 is characterized by the following parameters: stroke 200 mm conveyor length 7000 mm, time of one cycle 4 s linear speed 3.0 m/min. Transporter no 2 is characterized by the following parameters: stroke 100 mm maximum load 6000 kg/rm spacing of bars/billets on the conveyor grate: bars/billets up to 90 mm transport with 100 mm stroke, bars/billets over 90 mm transport with 200 mm stroke length of the transporter 9800 mm, time of one cycle 5 s linear speed 1.1 m/min. The design of the cooling bed allows for partial regulation of the cooling speed of products by the possibility of removing thermal insulation covers as well as adjusting the distance between the covers and the cooled products.

In order to assess the effect of post rolling cooling on the grain refinement and mechanical properties under industrial conditions, the temperature at the entrance to the cooling bed was measured as well as the cooling time and the temperature at the exit from the cooling bed (the temperature of the products before dropping them into the receiving bin).

After dropping into the receiving basket, the rolled bars were formed into production packages (maximum 5 tons) and further cooled down in stacks to the ambient temperature. After cooling down to the ambient temperature, hardness, tensile tests, and toughness tests were performed on the final products.



(**a**) (**b**)

Figure 2. Arrangement of rolled bars on the walking beam of the cooling bed: (**a**) entry section; (**b**) exit section.

Quasistatic tensile tests were carried out in accordance with EN ISO 6892-1B: 2016 [11] on five-fold round samples with a diameter of 10 mm. Three samples per rolled bar were cut in a direction parallel to the rolling direction. The impact toughness tests were carried out in accordance with the EN ISO 148-1: 2016 standard [12] V-notch specimens were cut in parallel direction to the rolling direction. Additionally, microstructural studies were performed using optical microscopy. Grain size was measured according to EN ISO 643 standard [13]. Specimens were cut from transverse cross-sections of the bars. They were mounted in Bakelite and mechanically grinded using SiC paper with 600–1200 grit first, then they were polished with diamond pastes (9, 6, 3, 1 micrometer particles sizes) and finally etched with 2% Nital solution. In order to provide statistically representative results, at least 100 grains were taken into account per sample.

3. Results and Discussion

3.1. Preliminary Results—Steel A

An example microstructure of the rolled round bar (85 mm in diameter) made of steel A is presented in Figure 3. It consists of a homogeneous ferrite-pearlite structure with a grain size ranging from 6 to 8 (ASTM). Results of mechanical properties are summarized in Table 2. Based on the analysis of the mechanical properties of steel A, it was found that the current production technology is highly unstable. The data show that with a very high stability of Yield Strength (YS) and Tensile Strength (TS), there is a high instability of the impact toughness (KV) at temperatures ranging from −20 to −40 °C.

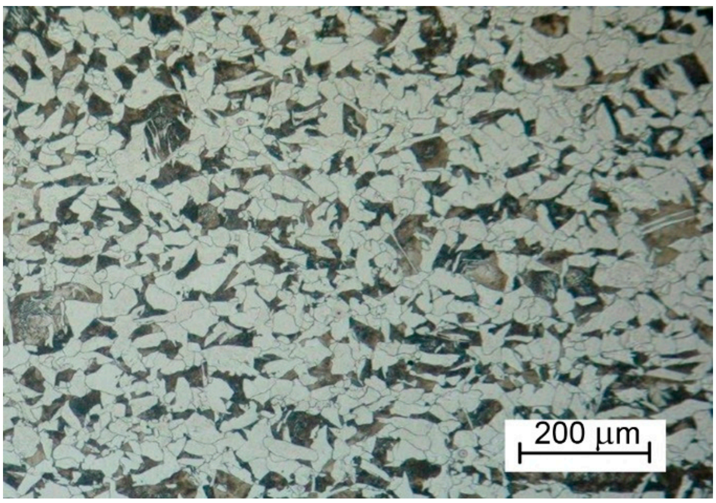


Figure 3. Example of microstructure (optical microscopy) of the cross-section (at mid radius) of 85 mm round bar after rolling and cooling to room temperature.

Table 2. Mechanical properties and impact toughness measured on 83 mm diameter round bar. Steel A.

YS, MPa	UTS, MPa	A, %	Z, %	Avg. KV-20, J	Avg. KV-40, J
363	535	30.6	67.5	62	21.6

An alternative to the traditional continuous rolling of bars is normalizing rolling, as a result of which the microstructure is homogenized and refined, which improves the mechanical and technological properties of the product. The use of such a technology also allows for a significant reduction in manufacturing costs as a result of lower energy consumption due to the elimination of normalizing annealing operations.

3.2. Results for Steels B, C, D

In order to determine the temperature changes on the surface of the bars cooled in the cooling bed, the method using the FLIR THERMA CAM S60 thermal imaging camera was used. The measurements were made directly during the real process of cooling bars in the cooling bed. Temperature measurements were made for one selected element moving in the cooling bed. To determine the values of the emissivity coefficient the method proposed in the literature was used. The method consists of comparing the indications of the thermocouple measuring the surface temperature with the indications of the infrared camera and determining the value of the emissivity coefficient, so that these two values of temperature are equal to each other. Surface temperature was measured by using K-type thermocouple, 0.5 mm in diameter. After the temperature measured by thermocouple stabilized, a thermogram was made. On the basis of these measurements, the emissivity coefficients were obtained, which changed from 0.87 to 0.94.

The reflected temperature was determined on the basis of the measurements of the temperature of the vault and the walls side surfaces of the cooling bed. The ambient temperature in the production hall was measured with an air temperature sensor.

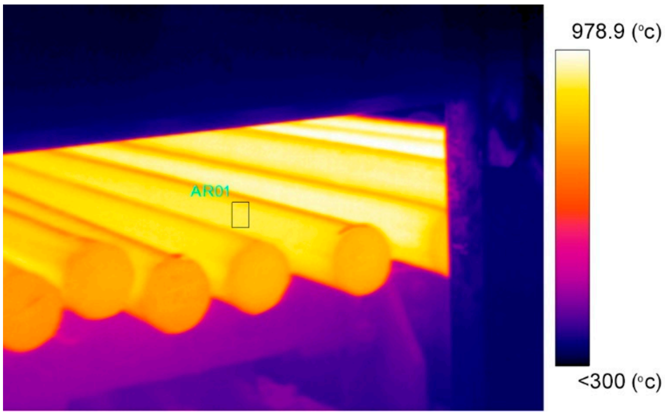
Thermograms were analyzed by using ThermaCAM Resarcher Professional (FLIR Systems Inc., Wilsonville, OR, USA). In Figure 4a, an exemplary thermogram obtained during measurements with the program tool "area" option marked, used for analysis, is presented. The "area" tool allows to determine the maximum, minimum and average temperature of the bar surface for a large field of view. Such action allows one to eliminate the errors resulting from local overcooling of the bar surface caused, for example, by contact with cold rails or air movement in one of the elements of the cooling bed.

During the registration of the thermograms, time was measured. This allowed for the development of a relationship between the time of the bar passing through the following stages of the cooling bed and the thermograms made, which in turn made it possible to determine the temperature changes as a function of time. In Figure 4b, temperature changes for a round cross-section bar during cooling are presented. The graph shows the maximum, minimum, and average temperatures.

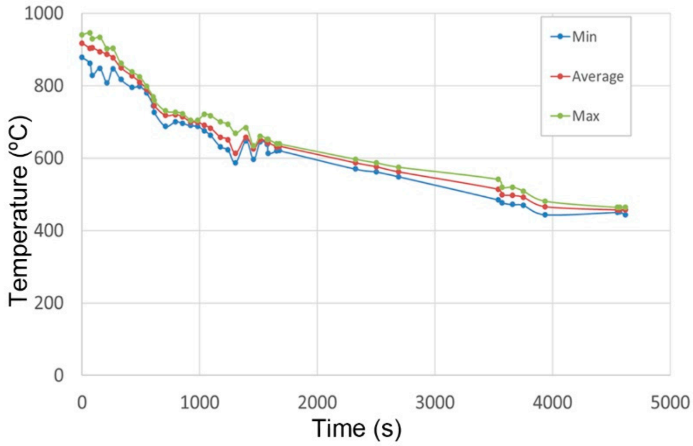
Assessment of the impact of material and process parameters on the microstructure and properties obtained after the cooling process was performed on the three steel melts with different combinations of grain refining elements (Table 1), i.e., Al-N (group B), Al-N-V (group C), and Al-N-Ti (group D).

Table 3 summarizes the results of mechanical properties assessment of 130 diameter bars after normalizing rolling and cooling in the cooling bed.

Calculations of the influence of the cooling start temperature and cooling rate on the microstructure of the products made of steel C were performed using the JmatPro (Version 10.2, Sente Software Ltd., UK) and are presented in Figures 5–7.



(**a**)

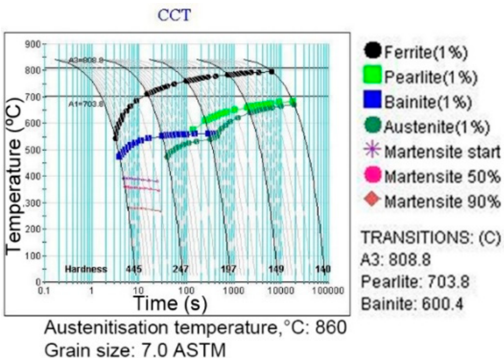


(**b**)

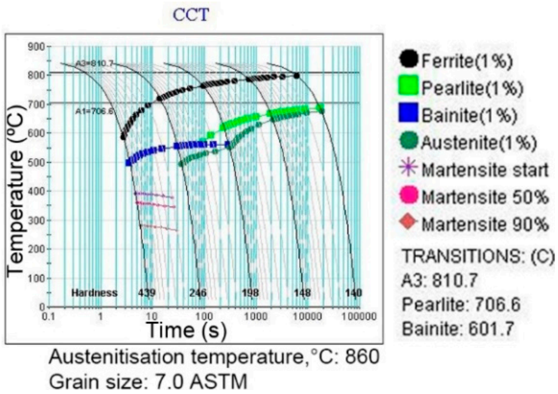
Figure 4. (**a**): Thermogram of the bars loaded on the walking beam of the cooling bed with the "area" option marked; (**b**) Example graph of temperature changes during cooling for a bar with a round cross-section.

Table 3. Mechanical properties and impact toughness of the rolled 130 mm diameter bars.

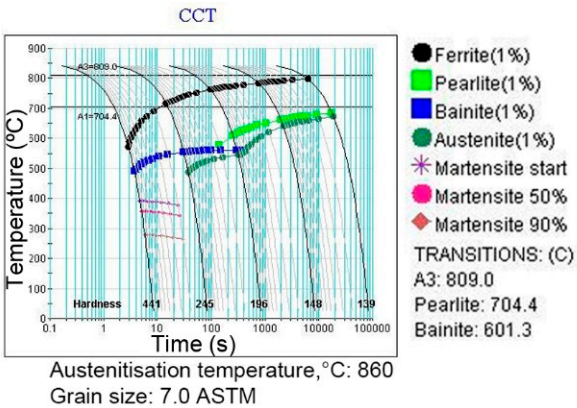
Steel	YS, MPa	TS, MPa	A, %	Z, %	Avg. KV-20, J
B	350	554	31.0	69.7	74.3
C	363	548	30.2	67.5	60.6
D	331	522	30.8	66.3	84.3



(**a**) B

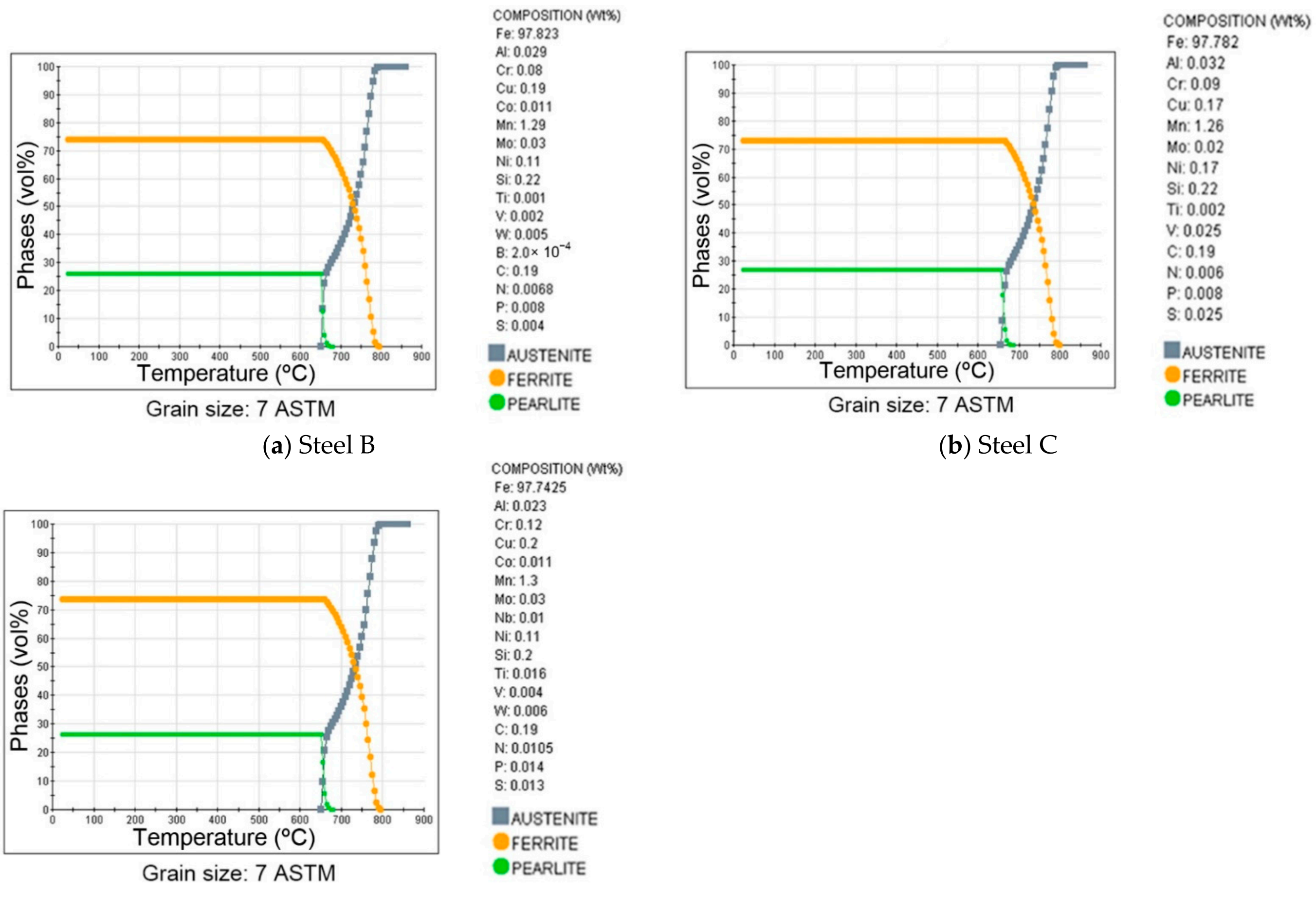


(**b**) C



(**c**) D

Figure 5. Continuous Cooling Transformation (CCT) diagrams obtained from JMatPro software for (**a**) B (**b**) C and (**c**) D steel.



(**a**) Steel B (**b**) Steel C (**c**) Steel D

Figure 6. Changes of the volume fraction of microstructure components during cooling process. Steel (**a**) B, (**b**) C and (**c**) D.

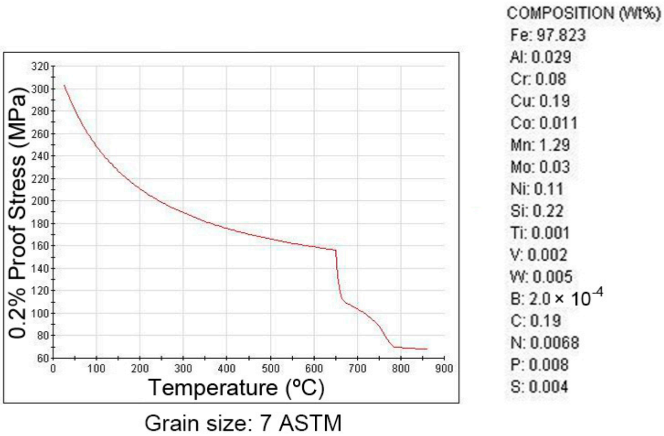
It can be seen that the phases obtained in all steel grades are typical for S355J2 grade (ferritic-pearlitic) and the differences in the critical phase transformation temperatures are negligible. Therefore it can be assumed that the differences in the level of impact toughness come from a different grain size.

The mechanical properties of hot-rolled bars were the point of reference for assessment of the nature and size of changes in the factors determining the strength and ductility of the investigated steel.

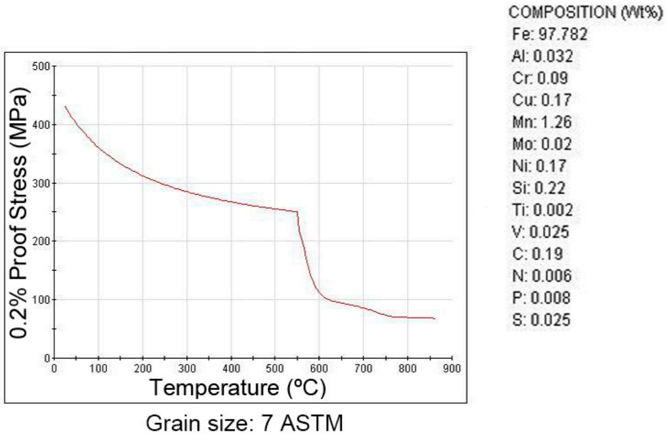
For the steel C, group Al-N-V, the tests of mechanical properties were carried out on qualification samples, a rolled dimension of 80 mm, specified in the standardization of normalizing annealing (normalizing), i.e., heat treatment consisting of heating the material to a temperature of 920 °C, heating for 2 h and cooling it in the air. As a result of this heat treatment, the following properties were obtained: YS = 369 MPa, TS = 541 MPa Elongation, A5 = 30.6% and impact toughness at −20 °C of 169, 141, 165 J (in three tested Charpy specimens).

The result for the impact toughness of ~160J is related to the normalizing heat treatment of bars after rolling. For the normalizing rolling, there is a logical decrease in the impact toughness value related to the rolled dimension and the conditions of the heat dissipation rate upon cooling.

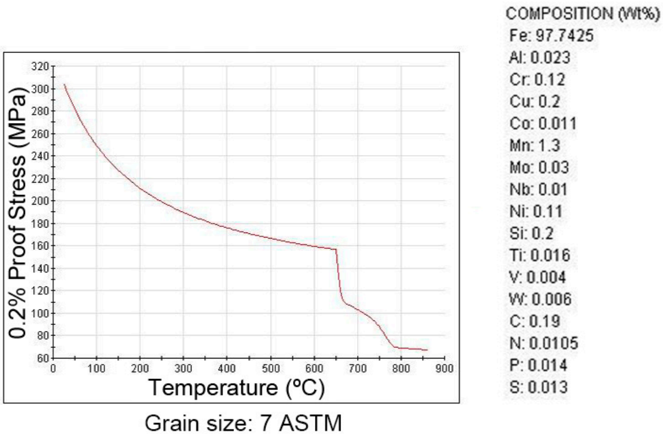
The Al-N-V steel (grade C) was selected as the first group to verify the assumptions of the production processes, based on the highest yield strength results obtained.



(**a**) Steel B



(**b**) Steel C



(**c**) Steel D

Figure 7. Yield strength (YS) of the bars during cooling process calculated in JMatPro—(**a**) B, (**b**) C and (**c**) D steel.

The mechanical properties of various dimensions of rolled bars for the Al-N-V steel group under industrial conditions are summarized in Table 4 and Figure 8.

Table 4. Mechanical properties and impact toughness of the Al-N-V steel rolled bars after cooling.

Bar Dimension, mm	YS, MPa	TS, MPa	A, %	Z, %	Avg. KV-20, J
150	369	561	25.4	64.0	36.33
140	369	561	25.4	61.5	46.33
150	350	535	30.0	64.0	35.33
150	338	529	30.0	66.3	46.00
110	357	548	26.4	66.3	40.67
120	331	548	32.4	70.8	59.33
140	363	554	29.4	64.0	46.67
150	370	552	28.6	63.3	52.33
120	376	535	29.4	64.0	43.00
120	344	535	26.8	64.0	58.33
100	376	548	28.6	64.0	63.33
80	401	548	27.2	68.6	80.67
90	376	561	27.0	65.2	86.33
95	401	573	27.8	65.2	65.67
95	401	567	26.8	69.7	71.00
135	369	561	28.4	62.8	51.00
85	389	554	30.4	70.8	59.00
130	363	548	30.2	67.5	60.67

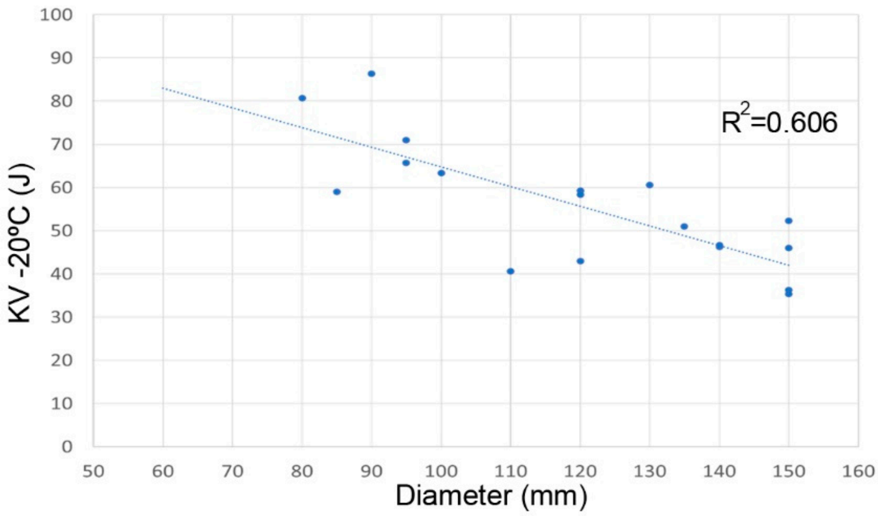


Figure 8. Impact toughness versus bar diameter. Steel C.

The reasons for the difference in impact strength may result from the different microstructure of the tested bars and the changes in the cooling rate under the production conditions of round bars. A parameter that has a particularly strong influence on the value of the impact toughness, as well as on the nature of the transition of steel into the brittle state, is the grain size of the material, which depends, among other things, on the grain refining components and their homogeneity both at the cross-section and along the length of the bar.

It should be stated that the characteristics of the cooling process after the completion of the rolling process is a key element in the thermomechanical treatment process. Due to the different dimensions of the cross-sections of the finished product, it is appropriate to formulate general rules for the cooling process for each bar dimensional regime separately.

The differentiation of austenite grain growth kinetics in different families of microalloy steels seems to be mainly due to the scale of the concentration of microalloying components.

On the basis of the obtained test results, it can be concluded that additional grain refinement was obtained by applying continuous cooling after the rolling process. However, accelerated heat removal from the volume of the bars after rolling is not always advantageous from the point of view of the performance of the bars. It is necessary to determine the optimal range of cooling rates that would guarantee the grain refinement, but not cause the appearance of undesirable phases, such as bainite (please see Figures 5 and 6).

Based on the data presented in Table 5, it can be seen that problems with reduced impact toughness at −40 °C of the tested steels result from too high a content of 3 elements: P, Mo and Cu. Phosphorus increases strength and hardness, but at the expense of ductility and toughness, especially in the case of steels with increased carbon content, which are quenched and tempered. Therefore, its content in most steels is limited to a maximum of 0.05%.

Table 5. Example of mechanical properties (YS) and impact toughness (KV-40C) variations with respect to chemical composition changes. Industrial data.

Batch No.	Grade	KV-40C, J	YS, MPa	C	P	Mn	Si	Cr	Ni	Cu	Mo	V	N_2
1	A	63	459	0.19	0.01	1.38	0.37	0.08	0.27	0.14	0.02	0.099	0.0082
2	C	75	471	0.18	0.009	1.35	0.37	0.13	0.22	0.17	0.03	0.095	0.0095
3	C	73	452	0.2	0.005	1.39	0.39	0.11	0.21	0.17	0.03	0.095	0.0077
4	B	31	497	0.19	0.011	1.54	0.40	0.15	0.44	0.16	0.06	0.104	0.0159
5	B	17	427	0.17	0.014	1.54	0.41	0.12	0.32	0.17	0.06	0.102	0.0080

In steels with increased strength properties, it can be seen that increasing the Mo content accelerates the transformation of austenite under continuous cooling, which is not as expected, because Mo is an additive intended to increase hardenability. The assessment of the influence of the chemical composition on the hardenability of steel is often presented by comparing the differences between the temperatures of the beginning of the austenite transformation during cooling and the temperatures resulting from the phase equilibrium system (Ae3–Ar3 and Ae1–Ar1). The differences take into account the influence of the cooling rate and the chemical composition. Therefore, when referring to the transformation start temperature (Ae3–Ar3), it turns out that, for example, the addition of Mo has no effect on the kinetics of the transformation, because the increase in the transformation start temperature is the same as for steel without its addition. This confirms the statement that the influence of Mo is more a consequence of the influence on the equilibrium than on the kinetics of the transformation process. Given the fact that Mo has a large positive effect on hardenability, it can be expected that this effect must increase with the cooling rate. Previous studies have confirmed that in the case of low-carbon steel with Mo addition, the transformation of austenite into ferrite was delayed at high cooling rates [1]. On the other hand, at low cooling rates, the austenite phase transformation was accelerated. Such an influence of Mo, with its increased content, will generally improve the strength properties of the steel, but the toughness will be lowered if the thermomechanical processing conditions that improve the refinement of ferrite are not used.

Also of interest is the often underestimated effect of Cu, delaying the transformation of austenite. This effect is more pronounced under continuous cooling than the thermodynamic calculations show. Cu does not split between the phases during the transformation of austenite into ferrite, which means that, in the first place, sufficient subcooling is required to effect this transformation. In fact, given the low solubility of Cu in ferrite, the non-equilibrium concentration of Cu in ferrite will greatly increase its free energy, thus lowering the eutectoid temperature and reducing the overall momentum of the transformation. This is an indirect explanation of the influence of Cu on hardenability. The effect of the Cu micro-additive, which is typically found in steels produced with large amounts of scrap, can be of great importance in the production of bars and billets. This is precisely due to its influence on the hardenability, favoring the process of formation of polygonal ferrite, which has greater strength properties than conventional granular ferrite, but adversely affects the impact toughness.

One way to quickly assess the heating conditions for steel is to use the empirical formula proposed by Cuddy et al. [14]. The differentiation of the austenite grain growth kinetics in different families of microalloyed steels seems to be mainly due to the scale of the concentration of microalloyed components. For example, the austenite grain growth temperature can be increased by about 200 °C when the niobium content is increased from 0.01 to 0.11% by weight. In industrial practice, it is assumed that steels tested according to [9] should exhibit a grain size ASTM no of 5 and larger. Aluminum is used as the grain refining element (e.g., a minimum of 0.020% aluminum is sufficient to bind the nitrogen).

4. Conclusions

In the presented work, the effect of chemical composition on grain refinement and mechanical properties and impact toughness of heavy-section bars was studied under industrial conditions. It has been shown that by careful control of chemical composition and cooling rate, the stability of properties can be achieved. It has been also proved that by proper control of normalizing rolling parameters, additional heat treatment operations may be avoided. Based on the performed research, the following conclusions can be drawn:

(1) During normalizing rolling of heavy section bars made of S355J2 steel, the most crucial elements with respect to ensuring the sufficient level of impact toughness are Al, V, Ti and N. It has been found that steel with increased level of Al-N-V (within the limits of EN-10025-2 Standard) provides the most optimal ratio of strength and impact toughness.

(2) Additional grain refinement can be obtained by applying continuous cooling after the rolling process. However, accelerated heat removal from the volume of the bars after rolling is not always advantageous from the point of view of the performance of the bars. It is necessary to determine the optimal range of cooling rates that would guarantee the grain refinement, but not cause the appearance of undesirable phases, such as bainite.

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