

Article

Method of Verification of Carbon Segregation Ratio Determined with Experimental Methods

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Abstract: The problem of macrosegregation of alloying elements occurring during cast strand solidification in the continuous casting process is still valid; it is the subject of numerous experiments and theoretical considerations. A large percentage of this research is dedicated to carbon segregation, which, for understandable reasons, is vital for the production of high-carbon steels. The background knowledge on the mechanism of segregation occurrence indicates that it is a very complex effect, and a broad range of factors influencing the continuous casting process need to be considered. Therefore, it is difficult to translate information (provided by complex models of metal flow through a diphasic area at the solidification interface of a cast strand) into practical engineering recommendations to reduce the macrosegregation effect. The presented study shows the latest research related to the carbon macrosegregation effect for selected high-carbon steel grades cast with a continuous caster. Problems related to the recording of the effect concerned have been pointed out. The second part of the paper presents the influence of selected casting parameters on carbon macrosegregation intensity when casting 160 × 160 billets with a six-strand caster. In this case, the main subject of the research was the influence of the casting speed on macrosegregation intensity. In the following step, an attempt was made to find the relationship between the cast strand structure and the distribution of carbon content on its cross-section. The ultimate objective of the presented study was to find an answer to the question on the technological capabilities of restricting the segregation effect.

Keywords: continuous casting process; carbon segregation; high-carbon steel

1. Introduction

The first descriptions of the macrosegregation effect concentrated on the analysis of effects occurring during the metal solidification process at the interface. Assuming that the solubility of the segregating element was known, both in the liquid phase and the solid phase, the metal bath liquidus temperature variability profile was determined by the intensity of the liquid phase stirring near the interface. The determination of the variability of the actual melt temperature and the corresponding liquidus temperature at a specific point allowed us to determine the undercooling caused by the segregating element's concentration gradient. The intensity of the constitutional undercooling occurrence was associated directly with the structure of the forming cast strand [1], presented in Figure 1. Every single case of the liquidus temperature profile and the actual bath temperature, near the crystallization front, was correlated with the observed structure. The temperature profiles were introduced for both the beginning and the end of a given crystallization step. It can be seen that the type of crystalline structure strongly depends on the initial value of the constitutional undercooling.

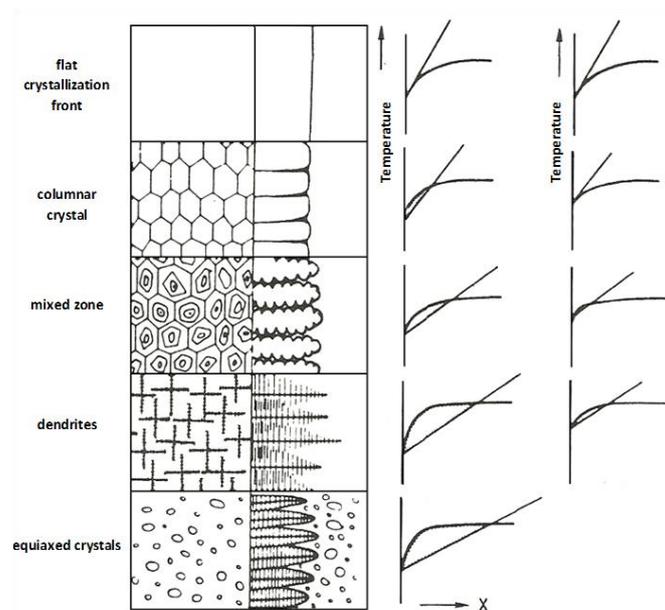


Figure 1. The type of crystals formed, based on the constitutional undercooling, versus distance from the crystallization front (coordinate axis X).

Applying the presented concept to a quantitative description of the macrosegregation effect would not allow for the many complex effects occurring within the solidification zone. Fleming [2,3] presented one of the more interesting analyses of the problem. He aimed to challenge the possibility of macrosegregation occurrence as the result of simply pushing the segregated addition out into the liquid phase, with high concentration gradients of the addition being pushed out before the crystallisation front. Fleming recognised the interdendritic flow varying in time as the basic mechanism of macrosegregation occurrence. Referring to a conducted experiment [3], he presented the macrosegregation as a result of the solidification process with a decreasing cross-section area of the flow (Figure 2). The mathematical model developed on the basis of this theory, taking into account the shrinkage effect and the deformation of the liquid phase space is based on the following relationship [3]:

$$-\frac{\partial g_L}{\partial C_L} = \left(\frac{1-\beta}{1-k} \right) \left(1 - \frac{v_x}{R} \right) \frac{g_L}{C_L} \quad (1)$$

where g_L is the liquid fraction volume, C_L is the interdendritic liquid composition, β is the solidification shrinkage, v_x is the component of flow in the x direction, R is the velocity of the planar isotherm perpendicular to, and moving in, the x direction, k is the partition ratio.

It is worth noting that the simplification of Equation (1), by assuming no shrinkage and zero velocity in the x direction, leads to a relationship (known from the first studies) based on the following assumptions: full diffusion in the interdendritic liquid, no diffusion in the solid phase, no macroscopic liquid movement, and no undercooling before nucleation [3]:

$$-\frac{\partial g_L}{\partial C_L} = \left(\frac{1}{1-k} \right) \frac{g_L}{C_L} \quad (2)$$

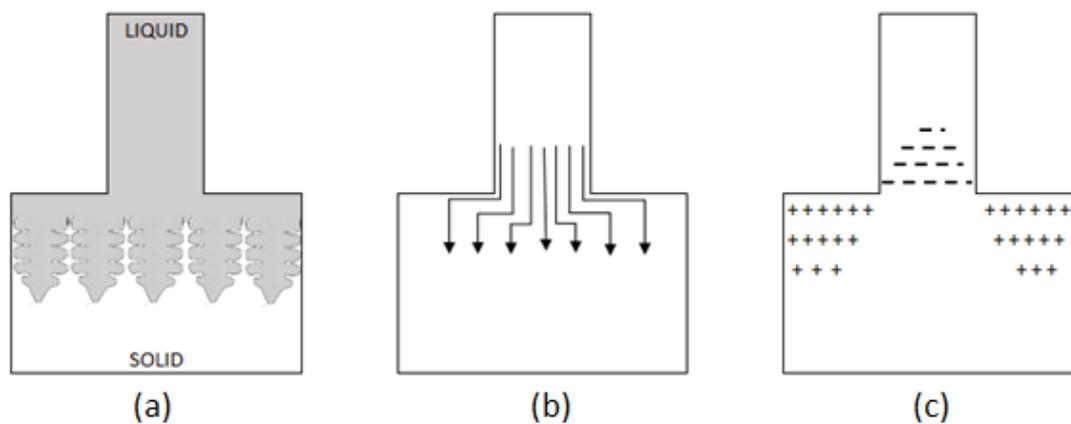


Figure 2. Macrosegregation in a unidirectionally solidified casting of reduced cross section. Schematic diagrams of (a) solidification when location of the reduced cross section is just below the liquidus temperature, (b) flow lines for feeding solidification shrinkage at this time of solidification, (c) resulting macrosegregation.

An analysis of very complex mathematical models of the macrosegregation effect leads to the conclusion that credible verification of these models is difficult, given that we cannot be certain of the validity of any assumptions. This analysis attempts to ascertain whether convection in a liquid significantly affects the segregation in the semi-solid zone. Today, it is difficult to expect an answer on the basis of a theoretical model only, and, therefore, experimental tests are still very relevant, both on a laboratory scale and an industrial scale. The published information [4–9] concerning the positive influence of electromagnetic stirrers on the reduction of carbon segregation in the continuous steel casting process shows that the mentioned direction of actions is very promising.

Difficulties relating to the verification of the mathematical model of the segregation effect do not exhaust the list of occurring problems. Moreover, the issue of the segregation effect recording is extremely important. It may seem trivial, but, after practical examination of the existing hazards, one should propose a formal criterion for the assessment of the experimental tests conducted. This issue becomes even more important if the segregation is recorded in industrial conditions as one of the essential components of the quality inspection system.

The problem of measuring the carbon content at the selected points of the continuous cast strand is considered negligible and is erroneously ignored in the discussion concerning macrosegregation. The authors analyzed a large number of samples, which helped them to introduce a new balance model. The authors' model allows defective samples to be rejected. Furthermore, precise calculation of the carbon segregation ratio, along with the ingots proper quality control, facilitates the best casting parameters selection, such as casting speed versus a water cooling intensity in each spray zone.

2. Measurement Problems when Recording the Segregation Effect in the Process of Continuous Steel Casting

A typical cross-section of a long cast strand always has a zonal structure. Depending on the steel chemical composition and casting conditions, the share of individual crystal zones varies. In extreme cases, a defective structure can be formed [9]. One of the most frequent defects that may occur when casting long strands is an axial porosity. It does not need to disqualify a product, but it is a serious obstacle in determining the chemical composition of the axial area of a billet. It is a well-known fact that during the segregation of carbon in high-carbon steels, the axial carbon concentration is usually the highest [10–13].

In experimental tests of the element segregation effect, two methods can be applied to determine the chemical composition: conducting a measurement with a spark spectrometer, or using samples obtained by drilling with a LECO Analyzer CS125 carbon and sulphur analyser (LECO Polska Ltd.,

St. Joseph, MI, USA). In the former method, the occurrence of a porosity in the measurement field leads to a substantial error. Note that this error is very likely to occur in the area that is the most interesting when investigating the segregation effect. Because of the observed inconvenience, many researchers prefer to take so-called “drill samples” and determine the chemical composition with a LECO analyser [9]. However, this method also has some deficiencies, as the sampling method itself causes averaging of the obtained result, and understating the value of the local carbon content may lead to incorrect conclusions (e.g., the assessment of cementite formation probability).

The test results obtained in ArcelorMittal Poland (the Dąbrowa Górnicza Branch) for billets made in a six-strand caster, with strand dimensions 160 mm × 160 mm, confirm that the considerations, presented above, are right. During comprehensive tests of the carbon segregation effect, samples were taken from billets number 5, 10, and 15 (beginning, middle, and the end of the 300 t heat) from strands number 1 and 3. The sample number directly indicates a strand and the billet number in relation to the heat; for example, sample no. 1/5 was taken from the first strand and the fifth billet. The sampling method is presented in Figure 3. 30 mm thick “slices” were cut out from all cast strand samples taken, and the carbon content was measured at points presented in Figure 4.

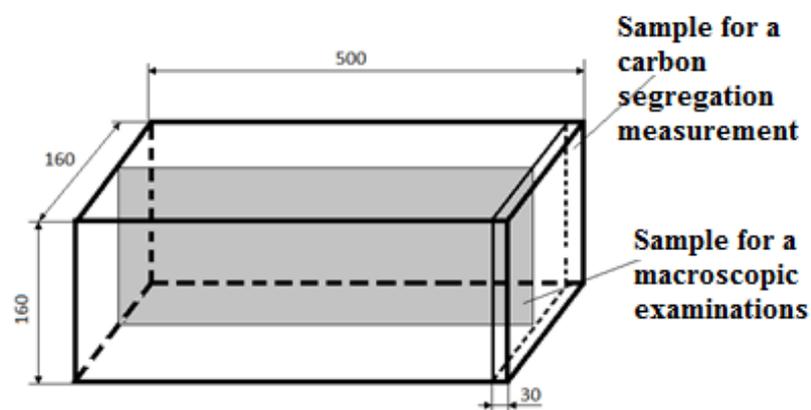


Figure 3. The shape of samples and the sampling method for macroscopic examinations and the assessment of the carbon segregation intensity.

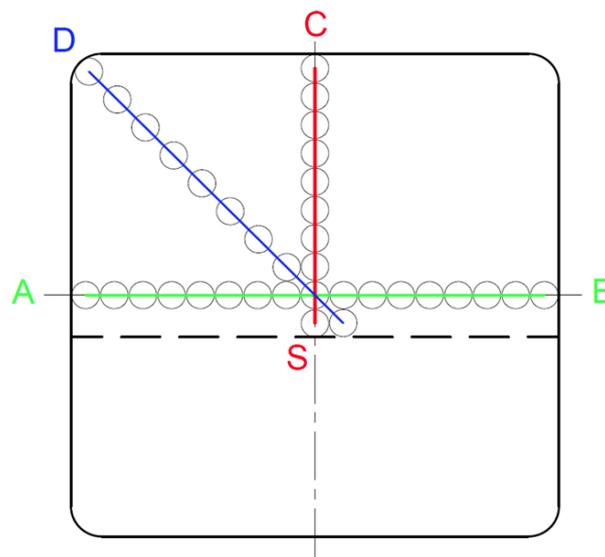


Figure 4. Billet cross-section with marked points for carbon content measurement.

Additionally, for random samples at points on the DS line (a diagonal of the square 160 mm × 160 mm—marked in the Figure 4), first spark spectrometry was performed, followed by drilling at

these points to take material for the second analysis with a LECO analyser. The picture shown in Figure 5 presents the condition of a sample after drilling. The overall results for all measurement points obtained with both methods are summarised in Tables 1 and 2. Heat number 1 was cast with the lower casting speed (1.8 m/min), and heat number 2 was cast with the higher casting speed (2.5 m/min).

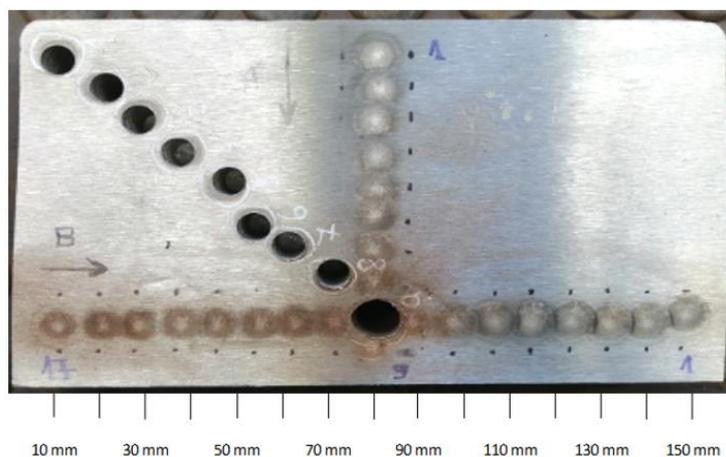


Figure 5. Example of sample taken from a billet cross-section with marked points for carbon content measurement.

Table 1. Summary of analyses of carbon content in random samples, performed with a LECO analyser and with a spark spectrometer (Spectrom.) for a casting speed of 1.8 m/min (Heat no.1).

Sample Number	Type of the Measurement Method	Carbon Content [%]-DS Line-9 Measurement Points								
		1	2	3	4	5	6	7	8	9
1/5	LECO	0.82	0.82	0.83	0.83	0.83	0.82	0.82	0.83	0.92
	Spectrom.	0.77	0.78	0.81	0.86	0.88	1.02	1.20	1.33	1.75
1/10	LECO	0.83	0.83	0.85	0.84	0.87	0.86	0.84	0.84	0.95
	Spectrom.	0.75	0.78	0.79	0.83	0.84	1.33	1.62	1.14	1.77

Table 2. Summary of analyses of carbon content in random samples, performed with a LECO analyser and with a spark spectrometer (Spectrom.) for a casting speed of 2.5 m/min (Heat no.2).

Sample Number	Type of the Measurement Method	Carbon Content [%]-DS Line-9 Measurement Points								
		1	2	3	4	5	6	7	8	9
1/10	LECO	0.83	0.83	0.83	0.83	0.84	0.83	0.84	0.83	0.95
	Spectrom.	0.75	0.78	0.79	0.80	0.79	0.82	0.85	0.68	1.28
1/15	LECO	0.82	0.82	0.81	0.82	0.82	0.82	0.82	0.73	1.16
	Spectrom.	0.77	0.79	0.80	0.85	1.00	0.96	1.24	1.83	2.05
3/15	LECO	0.82	0.82	0.83	0.82	0.82	0.82	0.83	0.83	0.98
	Spectrom.	0.76	0.78	0.80	0.80	0.81	0.85	1.01	2.47	1.88

The average carbon content, measured as average from tundish samples, was 0.82% for heat number 1 and 0.83% for heat number 2. The comparison of results of analyses performed with both methods raises some concerns, obliging us to ask fundamental questions concerning the methodological correctness of the measurements performed. The fact that measurement errors seem the biggest at the crucial measurement point (the strand axis) is an additional problem. Therefore, it was proposed to make a simple balance method, allowing incorrect measurements to be eliminated for big data sets. It was also proposed that the carbon content in the strand axis could be adjusted.

3. The Balance Model Based on the Values of Local Segregation Ratios

The method of the segregation ratio calculation is usually based on the relationship given by the formula:

$$k_i = \frac{[C]_i}{[C]_{ladle}} \quad (3)$$

where $[C]$ is the carbon percentage at point “ i ” of the sample and $[C]_{ladle}$ is the carbon percentage in a ladle sample. Both the numerator and the denominator of this fraction influence the accuracy of the estimation of the value of the segregation ratio. It is observable that the accuracy of the ladle sample analysis is particularly relevant as its potential error is transferred onto all values of the k -ratio. In addition, a principal approach to the segregation effect requires that the stability of the melt chemical composition be considered. In the continuous steel casting process, at each replacement of the main ladle, there is a transition period, during which the chemical composition changes. Additionally, small composition gradients within the heat cannot be excluded, because of the imperfect homogenization of the melt, in particular at the beginning of casting the subsequent heat in a sequence. Determining the denominator in Equation (4) can be another method used to solve the problem.

$$[C]_0 = \frac{\sum_{i=1}^n [C]_i}{n} \quad (4)$$

where $[C]_0$ is the carbon percentage in the tested fragment of the strand, at the ideal lack of concentration gradient, and n is the number of measurement points from the edge to the centre of the strand. However, this method is incorrect as it leads to overstating the $[C]_0$ value. Therefore, the authors propose to apply the weighted average method, in the form:

$$[C]_0 = \sum_{i=1}^n [C]_i w_i \quad (5)$$

where w_i denotes weights to calculate the weighted average.

The table of results for tests—Table 3—shown in Figure 5, using weights for a 160 mm × 160 mm square billet, is as follows:

Table 3. Values of weights for calculation of the average carbon content in a sample for the strand 160 mm × 160 mm, at 9 measurement points.

Weight Number	Values of Weights, [–]
w_1	0.221453
w_2	0.193772
w_3	0.166090
w_4	0.138408
w_5	0.110727
w_6	0.083045
w_7	0.055363
w_8	0.027682
w_9	0.003460

Derived weight values and the justification of the selection will be presented in the next step with the definition of Equation (7). The starting point for the balance analysis conducted is the assumption that the balance of any segregating element does not change. It means that its content in the sample after segregation is exactly the same as in the hypothetical condition without any segregation. Let us consider, for instance, a test sample, which is a rectangular prism with dimensions 160 mm × 160 mm × h mm, where h is its height. Assuming that no concentration gradient occurs, the mass of carbon $m_{[C]}$ contained in this sample should be determined from the equation:

$$m_{[C]} = F_0 h \rho [C]_0 / 100 \quad (6)$$

where F_0 is the billet cross-section area, h is the sample height, ρ is the steel density, $[C]_0$ is the carbon percentage in the sample if there were no segregation. In theory, the last value is the carbon content in the ladle sample, but due to the already described fluctuations in the melt composition, this value is a source of additional errors when determining the values of the segregation ratio.

In fact, the distribution of carbon in the sample is non-uniform, and, therefore, a calculation of its total content must take into account this diversity. In the conducted experiment, we have nine analyses of chemical composition, starting from the edge to the centre of the strand (Figure 6).

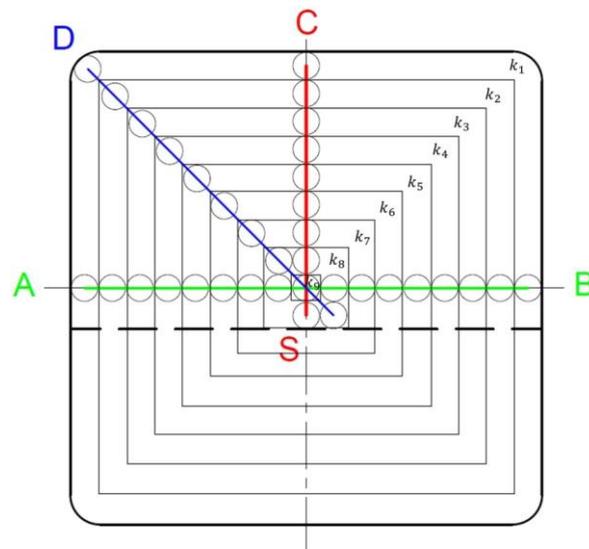


Figure 6. Layout of measurement points on the billet cross-section and division of the cross-section area into 9 zones.

Assuming that in each of nine measurement zones the average carbon content is the same as its measured value, the carbon balance equation can be presented as follows:

$$m_{[C]} = \sum_{i=1}^9 F_i h \rho k_i [C]_0 / 100 \quad (7)$$

where F_i is the area of the “ i -th” zone, h is the sample height, ρ is the steel density, $[C]_0$ is the carbon percentage in the sample if there were no segregation, k_i is the carbon segregation ratio in the “ i -th” zone (equal to $[C_i]/[C_0]$, [-]), $[C]_i$ is the carbon percentage measured in the “ i -th” zone. As the calculated carbon contents in Equations (6) and (7) must be identical, it results in this relationship:

$$\sum_{i=1}^9 F_i k_i = F_0 \quad (8)$$

Equation (8) also allows us to determine the weights referred to in Equation (5). After substituting k_i in Equation (8) with the relationship defined by Equation (3), we obtain:

$$[C]_0 = \sum_{i=1}^9 \frac{F_i}{F_0} [C]_i \quad (9)$$

Hence:

$$w_i = \frac{F_i}{F_0} \quad (10)$$

Equation (9) was used to determine the basic carbon content in each sample, which later was used for determining adjusted values of segregation ratios. These values take into account potential fluctuations of the melt chemical composition during casting. It follows directly from Equation (8) that fluctuations of segregation ratios do not have to yield an average near 1. For instance, Equation (8) is satisfied for the following set of segregation ratios, presented in Table 4:

Table 4. Values of segregation ratios calculated for individual zones 1–9.

Zone Number	$k_{i,r}$ [–]
1	0.8889
2	0.8955
3	0.9324
4	0.9975
5	1.0191
6	1.1742
7	1.3898
8	1.5322
9	2.0212
Average	1.205644

The arithmetic mean of these ratios is 1.2. This effect arises directly from the fact that the metal mass in zone 1 for the case concerned is more than 64 times higher than in zone 9. When attempting to determine the average value of segregation ratios with the arithmetic mean method, it is tacitly assumed that these zones have the same mass.

Criterion for Evaluation of the Segregation Measurement Quality

Comprehensive research of the element segregation effect in the continuous steel casting process is usually carried out with a big measurement database, created on the basis of many tests. In the tests described, the number of chemical spectral analyses exceeded 1300. With such big data sets, the balance method described above allows measurements burdened with too high an error to be quickly and credibly eliminated. Rearranging Equation (8), while estimating the number of measurement zones, we can write:

$$\frac{\sum_{i=1}^n F_i k_i}{F_0} = 1 \quad (11)$$

It is understood that Equation (11) assumes the idealisation of the problem by assuming that, in each i -th zone, no concentration gradient occurs. This is obviously not true. Therefore, the criterion defined in Equation (11) should be considered satisfied when the obtained value fluctuates within a previously imposed and consciously selected range. In practice, it can be the range 0.9–1.1 (or even wider). Filtering the database results of the segregation assessment does not exhaust the problem of the assessment of measurement quality in the billet axis. Very often, as already mentioned, both the measurement carried out with the sample drilling method and the analysis with the LECO apparatus show a result lower than the spectral analysis. In such cases, when the spectral analysis result is doubtful, Equation (11) allows the theoretical carbon content in the axis to be checked. In this case, the estimation of carbon content in the axis can be calculated as follows:

$$[C]_n = \left(1 - \frac{\sum_{i=1}^{n-1} F_i k_i}{F_0} \right) \frac{F_0 [C]_0}{F_n} \quad (12)$$

4. Impact of Casting Speed on the Segregation

To evaluate the influence of casting speed on the segregation intensity, the DS measurement line (Figure 4) was assumed, where, in most cases, fairly stable changes of the k -ratio were observed. In this case, the difference between the maximum values of segregation ratios for samples taken from strand

number 1 and strand number 3 is relatively low. When determining the average for the maximum values of the obtained segregation ratios, we found that for a heat cast with a speed of 2.5 m/min, the value of the k -ratio increased by about 13%, compared to the casting speed of 1.8 min. The graphs in Figures 7–10 allow the stability of segregation effect to be estimated for the selected measurement DS line, in comparison to relevant values of the k -ratio for billets number 5, 10, and 15.

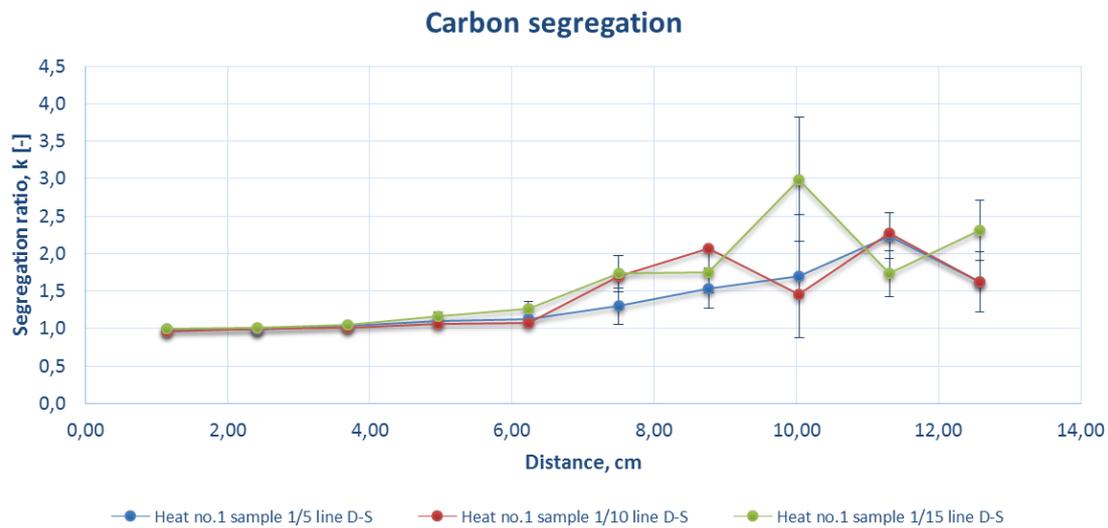


Figure 7. Comparison of the carbon segregation ratio for the sample DS line of strand 1 at a casting speed of 1.8 m/min (heat number 1).

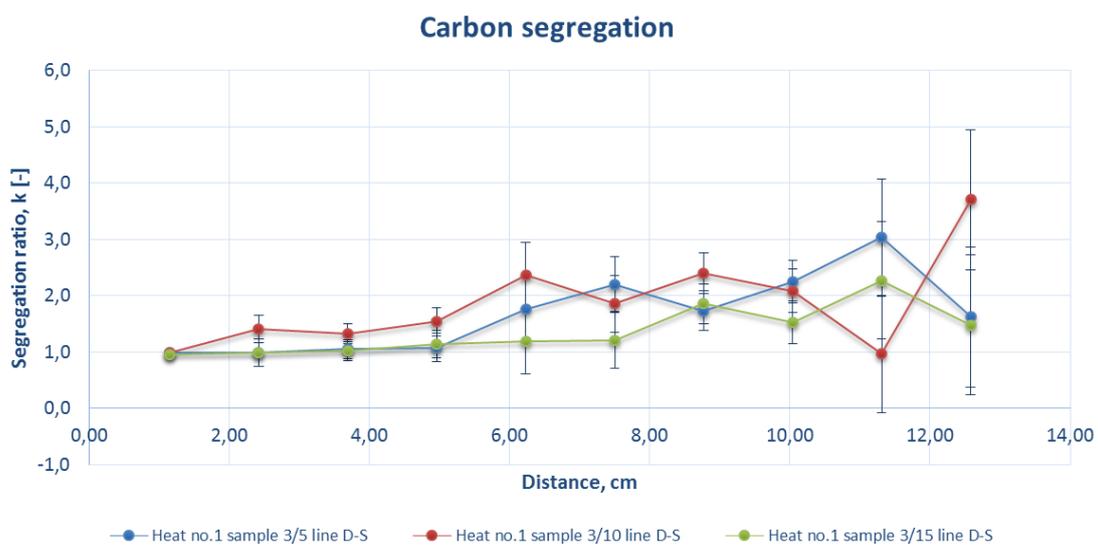


Figure 8. Comparison of the carbon segregation ratio for the sample DS line of strand 3 at a casting speed of 1.8 m/min (heat number 1).

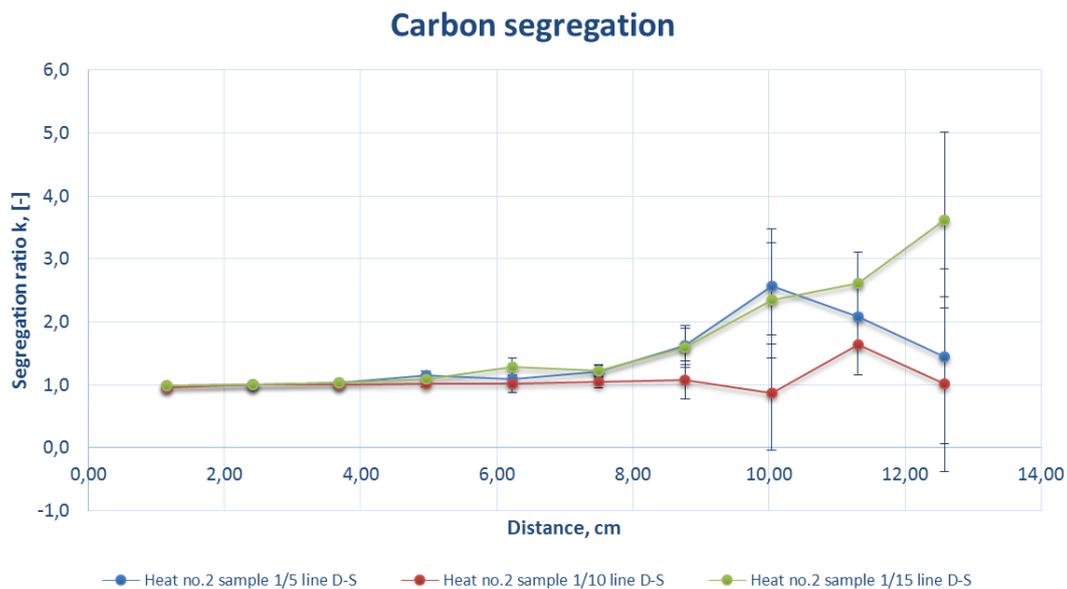


Figure 9. Comparison of the carbon segregation ratio for the sample DS line of strand 1 at a casting speed of 2.5 m/min (heat number 2).

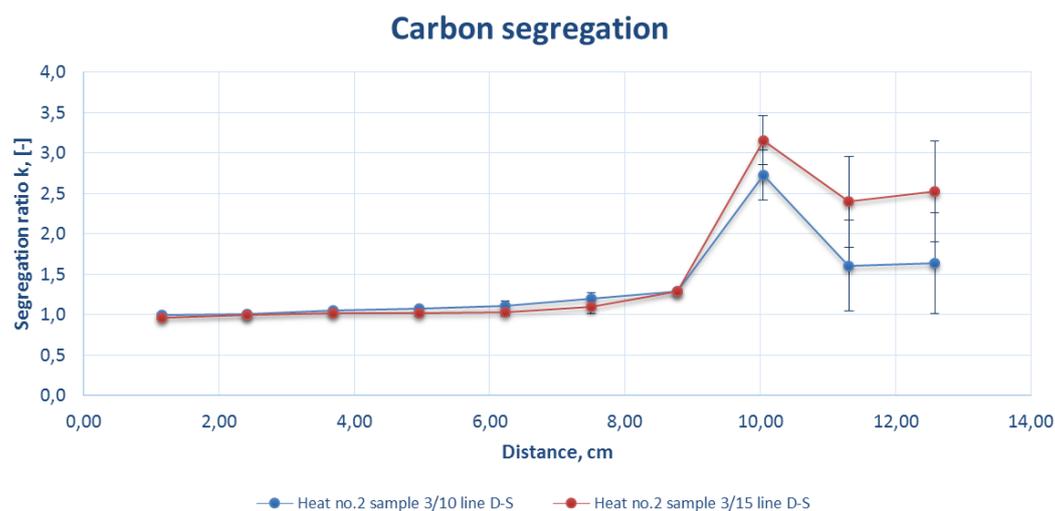


Figure 10. Comparison of the carbon segregation ratio for the sample DS line of strand 3 at a casting speed of 2.5 m/min (heat number 2).

It should be emphasised that, in order to assess the carbon segregation effects properly, one should take into account both the maximum values of the k -ratio and the percentage of cases where this ratio reaches values, from the top range, that are considered unacceptable.

5. Cast Strand Structure versus Segregation

In accordance with the current knowledge on the mechanisms influencing the macrosegregation intensity, one should pay special attention to the structure of a solidifying cast strand. The term ‘steel cast strand structure’ should be understood as: the way of distribution, dimensions, and shapes of crystals within the cast strand volume. As a result of steel solidification, three basic zones form in a cast strand: the chilled grain zone, the columnar zone, and the centre-equiaxed zone. The cast strand structure is a derivative of the casting speed and the applied cooling schedule. In the conducted tests, both the mentioned factors influenced the obtained cast strand structure. The quantification of the problem discussed is obviously not a simple task, but even ordinary comparative analysis allows us to capture tendencies, which should be taken into account when selecting technological casting

parameters. Figure 11a,b presents typical structures, obtained at two different casting speeds: 1.8 and 2.5 m/min.

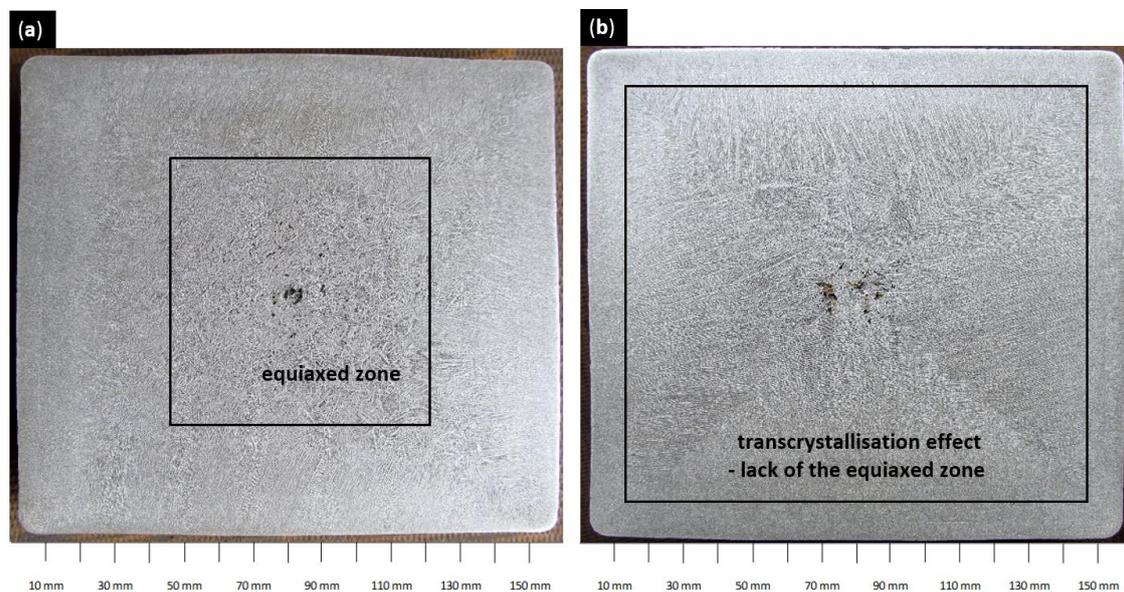


Figure 11. Cast strand structure (a) strand number 1, sample number 10, and casting speed 1.8 m/min; (b) strand number 1, sample number 10, and casting speed 2.5 m/min.

The structure of the billet presented in Figure 11a is a model structure with a clearly formed equiaxed zone. However, in Figure 11b, one can observe the so-called transcrystallisation effect, or a strong growth of the columnar zone, with a trace presence of the equiaxed zone. The conducted macrosegregation examinations in billets (examination with a spark spectrometer and a LECO analyser) directly confirmed the correlation between the billet structure quality and the carbon segregation level. For strands cast with a lower speed of 1.8 m/min, a lower segregation degree was measured, compared to the higher casting speed of 2.5 m/min (with the same amount of water fed per mass unit of cast steel). In the conducted examinations, the selected samples were etched to reveal their macrostructures. The mentioned examinations involved the following stages:

1. Cutting transverse samples from billets;
2. Grinding transverse samples;
3. Etching samples in a 50% solution with 38% HCl at a temperature of about 70 °C;
4. Rinsing samples under running water after etching;
5. Coating samples with transparent varnish.

6. Conclusions

The analysis of the carbon segregation problem in high-carbon steels indicates that it continues to be a valid problem, requiring both further development of theoretical description in the form of better mathematical models and improved experimental methods for recording this effect. The mathematical model of the segregation effect already includes its key components. However, it would be risky to say that, nowadays, it is possible to control the process on the sole basis of a mathematical model. The conducted series of tests in industrial conditions at the ArcelorMittal Poland Unit in Dąbrowa Górnicza indicates the trend in potential changes. These changes should include measures to restrict the growth of the dendritic zone by determining the maximum acceptable casting speeds and the correct selection of cooling parameters. The quality inspection should be based upon the full spectrum of tests, conducted in line with adopted procedures, and the quality of these tests should be verified on the basis of the presented balance model.

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Conflicts of Interest: The authors declare no conflict of interest.

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