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Mechanism of Coking Pressure Generation in the Light of the Results of Laboratory Tests

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Abstract: The phenomenon of coking pressure is crucial for cokemaking both in respect of a safe coke oven battery operation and a proper quality of the produced coke. In spite of that, the mechanism of this phenomenon has not been clearly explained yet. The aim of the presented research was to clarify which of the phenomena most commonly mentioned in the literature on the subject, i.e., the reduced gas permeability of the plastic layer or the swelling of plasticized coal grains, is responsible for generating internal coking pressure. To that end, laboratory equipment was developed which enabled examining the pressure generated by the bed of plasticized coal grains under conditions of a varying possibility of its expansion. The results of the examinations suggest that the swelling phenomenon of plasticized coal grains is the direct cause of coking pressure, and the coking pressure strongly depends on the possibility of plastic layer expansion. The results confirm also the migration phenomenon of plasticized coal matter, especially towards the cool part of the charge as well as the possibility of compression of this part of coal charge. As a result of both these phenomena, it becomes possible for the plastic layer to expand, which results in a reduction of generated coking pressure.

Keywords: coal; coking; plastic layer; coal swelling; coking pressure

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1. Introduction

The common application of the PCI technology, as well as the large volume of currently used blast furnaces, requires the application of a high quality coke, especially of high mechanical properties. The production of such a coke necessitates an increased share of coals of the highest coking parameters (coals of the hard-premium type). Due to the deficiency of such coals, other solutions are applied, e.g., coal blend predrying [1,2], briquetting of some components of a coal blend [3,4], hydrothermal treatment of sub-bituminous components of a coal blend [5,6] as well as introducing additives to a coal blend such as: petroleum coke [7], HPC [8–10], or coal tar pitch [11]. All these operations contribute to the growth of the coking pressure.

On the one hand, the internal coking pressure favors good agglomeration of plasticized coal grains and, consequently, improves mechanical properties of the resulting coke [12–16]. On the other hand, this pressure is transferred through the semicoke and coke layers to the walls of the chamber which is the source of the so-called "wall pressure" [3,17]. Its excessive volume can cause difficulties while pushing coke out of the chamber. Even worse, it can also cause damage and, in extreme cases, even complete destruction of the ceramic block of the coke oven battery [18,19]. For this reason, the wall pressure limits should not exceed 9.5–14 kPa, depending on the battery design [18,20–28]. Despite such an important role of coking pressure, the mechanism of its formation has not been fully explained yet. Furthermore, there is even a disagreement about the place where this pressure is generated. This is evidenced by the fact that there exist several hypotheses on this subject. Chronologically, the hypothesis of the so-called "plastic envelope" is the first. According to this hypothesis, the internal coking pressure is generated within the "cool"

part of the coal charge, where the temperature does not exceed the temperature of coal softening. The plastic layer surrounding this area creates a kind of an "envelope", inside of which, similar to in a trap, some of the volatile products of pyrolysis accumulate [29]. As the coking process progresses, the quantity of volatile products accumulated inside this envelope increases and, consequently, the coking pressure grows. At the final stage of the coking process, i.e., after the contact of both plastic layers in the middle of the coke chamber, the volatile products accumulated within the "envelope" are released as a result of the cracking of resolidified plastic layers. This phenomenon is accompanied by a rapid coking pressure drop. This hypothesis, however, is criticized because in the coke chamber plastic layers form a "plastic tube" rather than a "plastic envelope" and, therefore, the majority of volatile pyrolysis products (80–90%) leaves the coke chamber through the layers of hot semicoke and coke [30–33]. In addition, the results of pressure distribution measurements within the coke chamber do not confirm the fact that the highest pressure can be attributed to this very part of the carbonized coal charge [34–38].

In the light of the next hypothesis, the coking pressure is generated within the layers of semicoke and coke as a result of their low gas permeability, additionally limited by the cracking, polymerization, and condensation processes of volatile products of coal pyrolysis during the contact with hot coke [39–48]. The intensity of these processes depends on the number of volatile products released at temperatures exceeding the temperature of plasticized coal mass resolidification as well as on their chemical composition. The increase in coking pressure, observed with the progress of the coking process, is caused by a successive increase in the thickness of semicoke and coke layers [37,45,46], while the sharp increase of wall pressure in coke chambers, recorded at the moment when the two plastic layers meet in the middle of the carbonized charge, is attributed to the disappearance of the "cool" part of the charge which constituted the "container" for volatile products [45].

The last hypothesis assumes that the place of pressure generation is the plastic layer in the carbonized coal charge. According to some of the supporters of the hypothesis, the "entrapment" of volatile products of coal pyrolysis inside this layer is the direct source of coking pressure [41,43,49–54]. This layer is adjacent to the impermeable layer of semicoke from one side, and to the "cool" charge, full of condensed tar products and equally impermeable, from the other side (the so-called "sandwich effect"). Other authors suggest that pressure generation is a result of the obstructed flow of volatile products of pyrolysis through this layer [34,55–58]. It cannot be ruled out that the volume increase of coal grains within the temperature range of coal plasticity, caused by an increase in pressure inside these grains, is a direct source of pressure exerted by the plastic layer on the adjacent layers of semicoke and the "cool" charge [59,60]. The results of model calculations confirm that a high pressure is indeed created inside plasticized coal grains [61]. If the volume growth of these grains is smaller than the volume of voids between coal grains, the plastic layer will not be able to increase its volume and, consequently, will generate a high coking pressure. The occurrence of this phenomenon was confirmed i.a. by [62]. The shrinkage of semicoke and coke layers as well as the compression of the "cool" part of coal charge accompany this phenomenon. As a result, the plastic layer has the opportunity to expand [63–66]. However, if the effects of coke shrinkage and cool charge compression are not sufficient to compensate for the increase in plasticized coal grains volume, the plastic layer will generate a high coking pressure and, consequently, also a high wall pressure. In the case of coke oven batteries with vertical coke chambers, the possibility of plastic layer expansion depends on the following:

- Extent of lateral shrinkage of semicoke and coke layers;
- Possibility of migration of the plasticized coal matter outside the plastic layer, i.e., both into the voids between grains of the "cool" part of the coal charge as well as into microcracks and microgaps existing in semicoke [36,37,66–68];
- Compression ability of the "cool" part of the coal charge.

Figure 1 synthetically presents the last two hypotheses which are currently considered to be most reliable. The only difference between these two hypotheses concerns the role of

the pressure generated inside the plasticized coal grains. In the first case, this pressure is the direct cause of coking pressure and, consequently, of the wall pressure. In the second case, the inner pressure of the plastic layer is only a side effect of the growth in the volume of the plasticized coal grains; as it was already stated, the growth in the volume of the plasticized coal grains is the direct source of coking pressure.

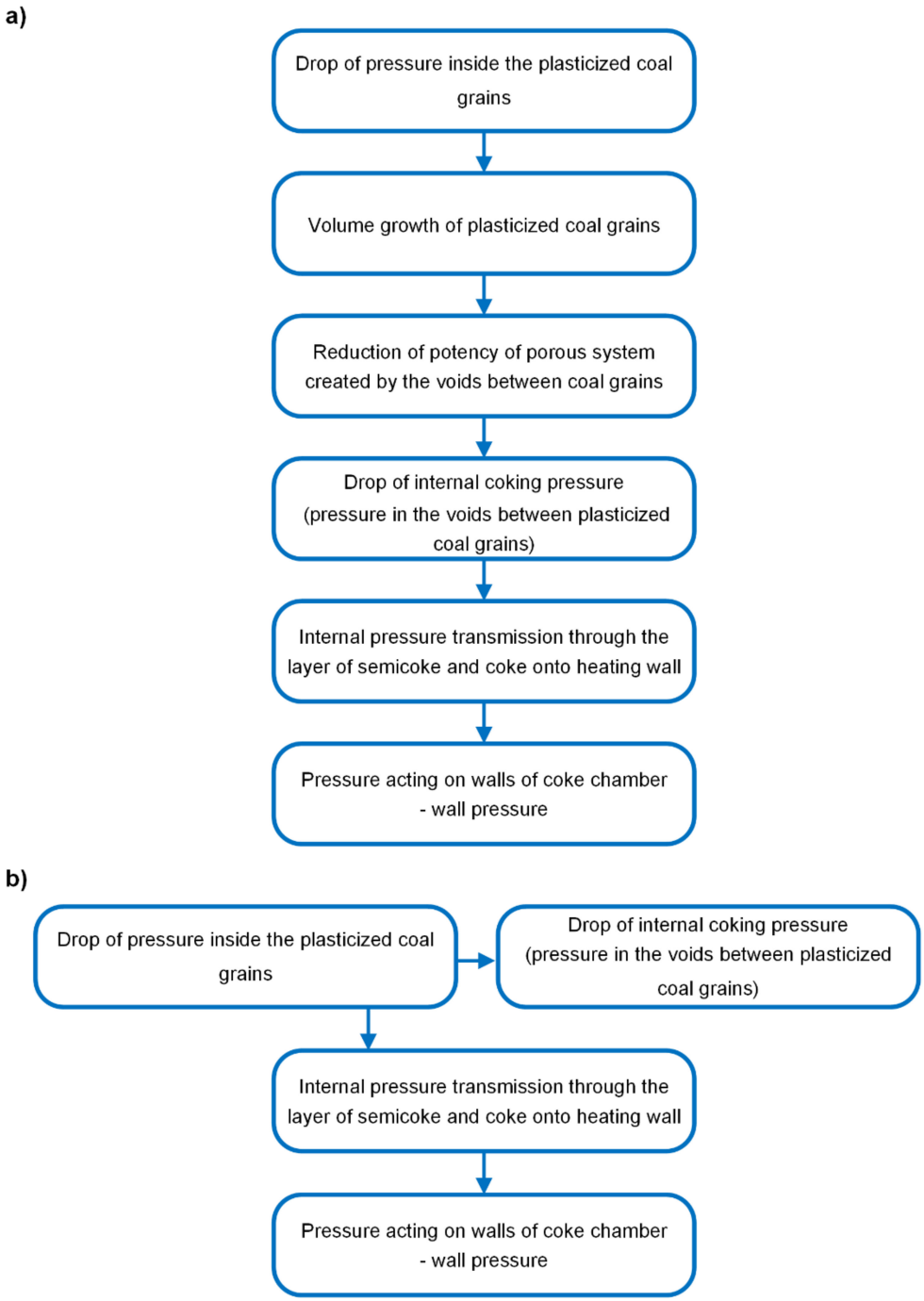

Figure 1. Mechanism of coking pressure generation by the plastic layer: (**a**) hypothesis of limited gas permeability of the plastic layer as the direct source of the wall pressure; (**b**) hypothesis of plasticized coal grains swelling under conditions of limited expansion as the direct source of the wall pressure.

Without deciding which of these two hypotheses is right, it should be emphasized that many aspects of coking pressure can be explained in a satisfactory manner assuming

that the volume growth of plasticized coal grains constitutes the primary source of coking pressure. Among the aforementioned aspects, the most important ones are as follows:

- Similar impact of such factors as the size of grains and the degree of coal oxidation on both phenomena (i.e., coking pressure and plasticized coal grains swelling);
- Convergence of the ability to generate a high coking pressure with a large growth of coal grains volume within the temperature range of coal plasticity which is recorded for coking coals of low volatile matter contents;
- Increase of generated coking pressure along with the growth of bulk density of the coal charge which can be attributed to the decreased volume of voids between coal grains that can be filled with swelling coal grains;
- Lack of lateral shrinkage of the charge inside the coke chamber at the initial stage of coking, typical for coal possessing the ability to generate an extremely high wall pressure;
- Small size of pores and the low total porosity of coke from coal blends generating a high coking pressure [66]; it can be attributed to the lack of, or a significantly limited swelling possibility of, plasticized grains of such coals. Moreover, adverse conditions for the growth of such grains make the pressure inside them very high;
- Specific course of pore development for the semicoke resulting within the temperature range of coal plasticity [66] which is typical for coals with the ability to generate a high coking pressure. For such coals, within the whole range of their plasticity, the porosity continuously increases due to the formation of new pores with small dimensions and, consequently, a high inner pressure. In the case of the remaining coals, the porosity of the resulting coals reaches the maximal value near the temperature of maximal coal fluidity and the pores are of large dimensions, and, thus, the inner pressure inside them is low. Therefore, along with a further increase of temperature, they can easily be compressed and, consequently, their volume does not grow;
- Beneficial mechanical properties of coke produced from coal blends generating a high coking pressure due to both their low porosity (as it was already mentioned), and a very good agglomeration of coal grains being in close contact with one another. The latter is a result of the limited possibility of swelling of the plasticized coal grains and a high pressure inside the grains, as well as elimination of microcracks and microgaps in the resulting semicoke due to their being filled with plasticized coal matter.

Therefore, the presented research was mainly devoted to evaluating the impact of the possibility of expansion of the plastic layer on the coking pressure generated by this layer.

2. Materials and Methods

For the examinations of pressure generated by the plastic layer, the laboratory equipment presented in Figure 2 was applied. A vertical electric oven equipped with a steel retort constituted the main part of the laboratory equipment. The retort was closed from above with a cover and from below with a threaded closure. Pyrolytic gases were evacuated through an outlet located in the cover. In the middle of the cover there was a threaded sleeve with a screw-on force sensor housing. This sleeve ensured the correct location of the measuring piston in relation to the surface of the examined sample (enabling the required expansion of the carbonized sample), and at the same time acted as a measuring piston guide. The piston conveyed the pressure from the plasticized coal sample onto the force sensor. The C9B force transducer was applied, with strain gauge measuring system, made by HBM Mess- und Systemtechnik GmbH (Darmstadt, Germany), of the accuracy class from 0.5 (for nominal force within the range: 0.5–20 kN) to 1 (for nominal source: up to 50 kN), nominal sensitivity 1 mV/V, and relative sensitivity deviation $\leq$1%. For measuring force, a system consisting of the abovementioned tensometric sensor and a signal amplifier was used. The heating system consisted of three resistance heaters with individual power control. It enabled obtaining a fairly even temperature within the space where the sample was found—during the test, temperature differences along and across the examined sample did not exceed 5 degrees. Thanks to the use of such a solution in the applied equipment,

after reaching the temperature of coal softening, the entire volume of the coal sample was plasticized and, therefore, the sample could be treated as a part (slice) of the plastic layer formed inside the charge carbonized in the coke chamber.

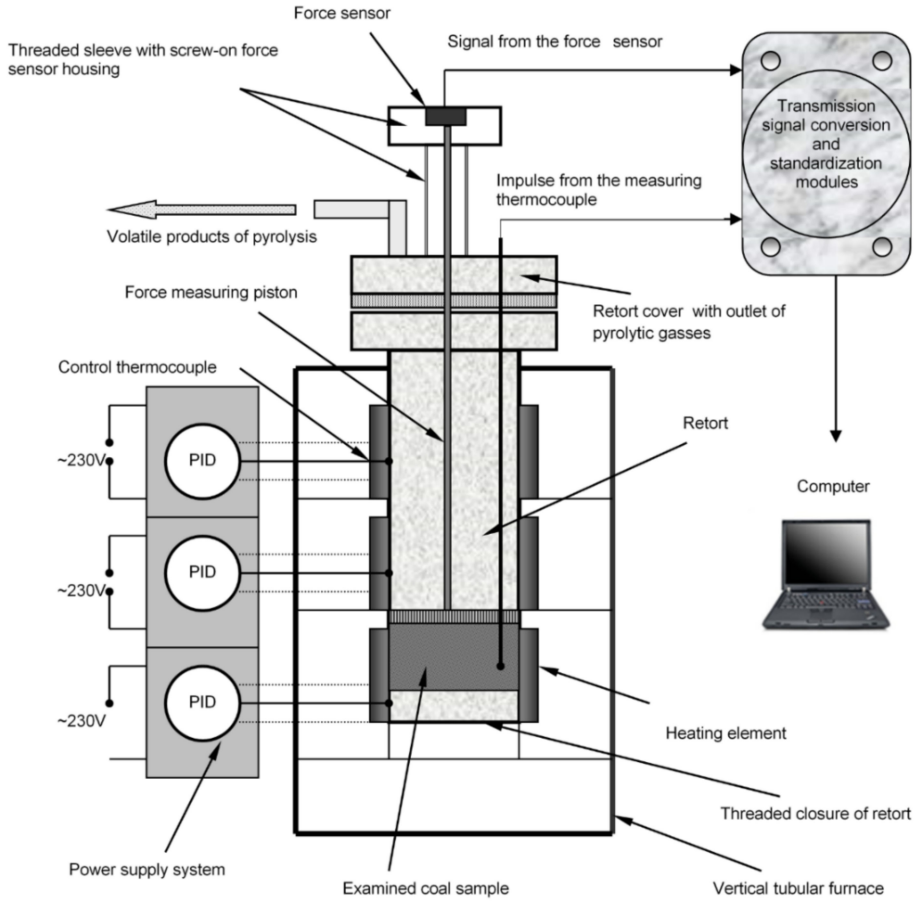

Figure 2. Laboratory equipment for examinations of coking pressure generated by the layer of plasticized coal grains during the coking process.

The temperature within the heated sample was measured with the use of a sheathed thermocouple. Both the force and the temperature pulses were transmitted to the transmission signal conversion and standardization modules and, through them, to a computer set used for the visualization and storage of measurement data.

The mass of coal samples depended on the applied bulk density and height of the samples. For example, in the case of a sample with the height of 40 mm and bulk density of 0.750 g/cm^3, its mass was equal to 60 g. The heating rate of a coal sample was ca. 3 °C/min. During the test, the temperature was recorded every 15 s and the force was recorded within the temperature range of plasticity of the examined coal in a continuous way. The standard deviation of the recorded maximal values of the generated pressure, determined on the basis of a series of repeated measurements (samples of the same coal examined under conditions of the same bulk density and the same expansion possibility of the carbonized sample) did not exceed 18.0 kPa. A sample course of changes in the force exerted on the measuring piston during the carbonization test is presented in Figure 3. Detailed characteristics of all the examined coals are presented in Supplementary Materials: Tables S1–S3, S8–S12 and S15.

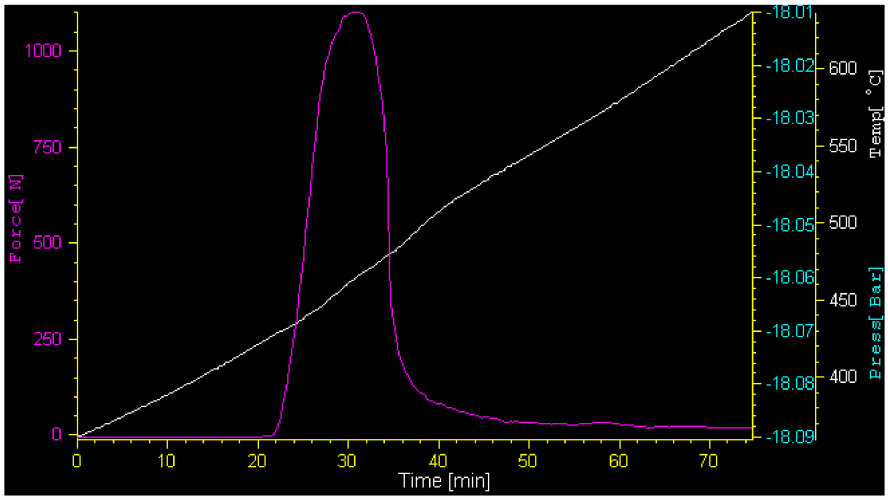

Figure 3. Sample visualization of the course of pressure measurement.

The volume changes of plasticized coal grains were examined under conditions of their free swelling with the use of a specially adjusted high-temperature Leitz microscope and the nitrogen atmosphere—Figure 4. The examinations were performed with the use of the type 301-200.301 microscope made by the Märzhäuser Wetzlar Company (Wetzlar, Germany). Within the framework of these examinations, twelve grains of each coal were sampled at random from each size fraction and then carbonized with the heating rate of 5 °C/min. The contour changes of the carbonized grains were recorded photographically at 12 different temperatures. The first photograph was taken at the temperature of 350 °C and the subsequent ones within the whole temperature range of coal plasticity for the analyzed coal. The photographs were scanned and digitalized. The cross-section areas of the carbonized coal grains were determined at different temperatures Assuming the spherical shape of the grains, their diameter and volume were estimated. On this basis, the indices of the relative increase of grain volume (in relation to its initial volume at 350 °C) were calculated for each of the examined temperature.

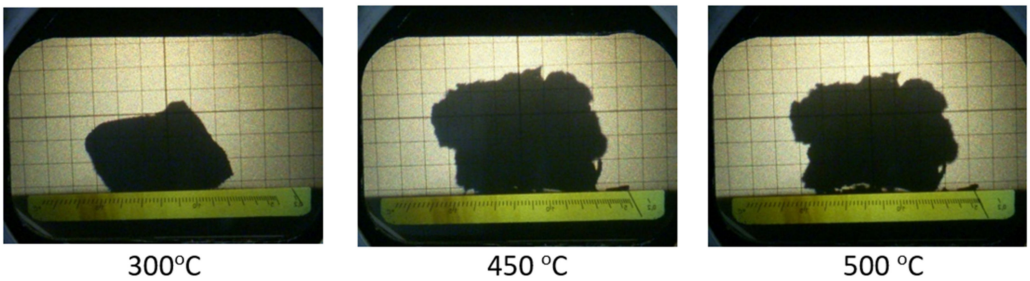

Figure 4. Sample changes in the volume of a coal grain during its carbonization (coal: Budryk; initial diameter of examined coal grain ca. 3 mm).

In order to verify the relationship between the phenomenon of free swelling of the plasticized coal grains and coking pressure generation by the layer of such grains, a statistical analysis was carried out. To that end, the maximal values of the relative increase of coal grains volume during the examination and the values of maximal coking pressure under conditions of the constant volume of carbonization space (0% of expansion of the carbonized sample) were compared. The statistical analysis was conducted for two levels of the bulk density of a coal sample (0.720 and 0.800 g/cm^3), four coking coals and three grain size fractions (0.4–0.6 mm; 1.4–1.5 and 2.0–3.15 mm).

3. Results and Discussion

3.1. Impact of the Plastic Layer Expansion on the Generated Coking Pressure

Within the framework of preliminary examinations, the impact of the possible expansion of the carbonized coal sample (within the range: 0–20%) on the generated pressure was determined. Three coking coals with the same grain size distribution (size fraction: 0–5 mm) and bulk density of the sample was equal to 0.625 g/cm^3 were tested. The selection of the coals was made, taking into consideration the great variety of their plastic and dilatometric properties. Characteristics of the examined coal samples are presented in Supplementary Materials—Table S1. The obtained results indicate a significant impact of the expansion of the carbonized sample on the generated coking pressure—Figure 5. At the next stage, the examinations for four selected size fractions of the abovementioned coals (2.5–3.15 mm; 1.0–1.2 mm; 0.5–0.63, and 0.2–0.315 mm) were carried out—Figure 6. The results of these examinations confirmed the strong impact of the size of grains on the coking pressure. It should be noted that in the case of the size fraction of 0.2–0.315 mm, none of the examined coals generated coking pressure. This results from the fact that, for coal grains with small diameter, the phenomenon of their swelling is not observed, which results from a relatively short route of migration of volatile products of pyrolysis. Therefore, in the conditions of a limited expansion, such grains do not generate the coking pressure [66].

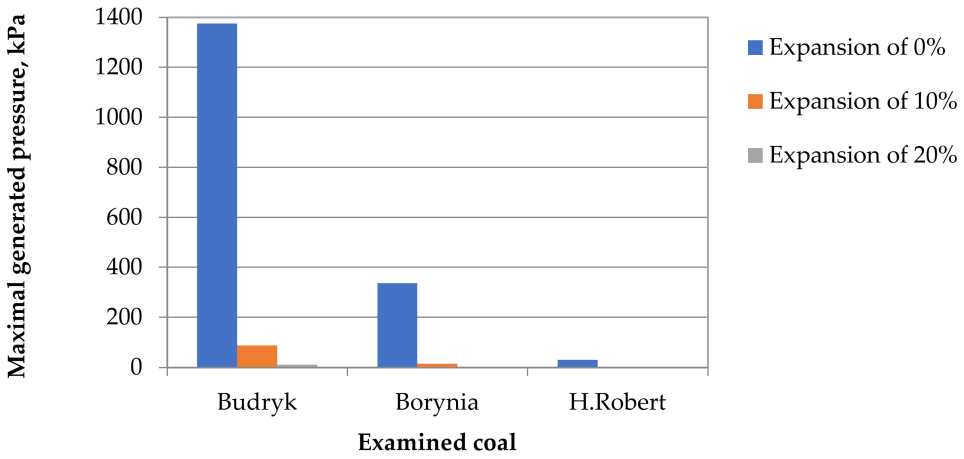

Figure 5. Impact of the expansion rate of the examined coal samples on the generated pressure (grain size fraction: 0–5 mm).

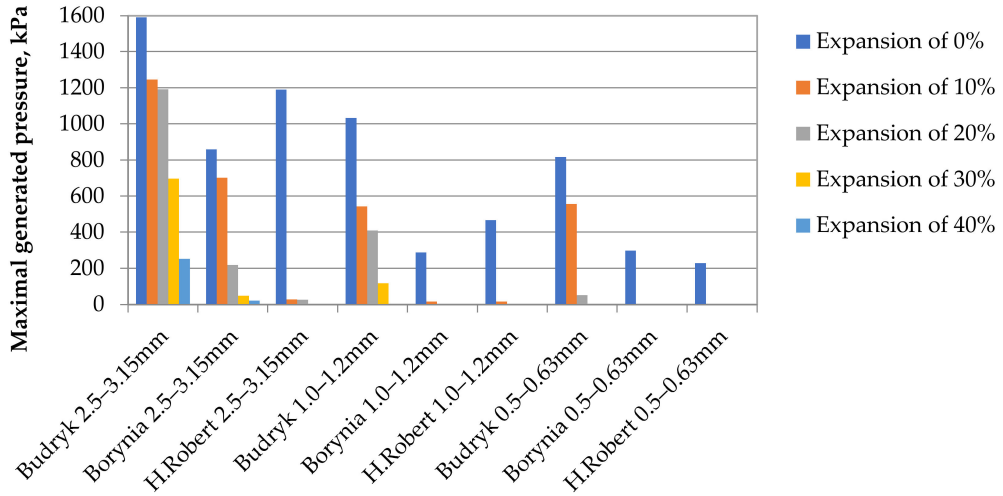

Figure 6. Impact of grain size of the examined coal samples on the generated coking pressure.

On the basis of these results, a statistical assessment of the significance of the following factors was carried out: coal (three coals), grain size (four size fractions), and the rate of expansion of the carbonized sample (five expansion levels: 0%, 10%, 20%, 30%, and 40%) for the pressure generated by the plasticized coal grains. For this assessment, one of the methods of variance analysis, i.e., the model of simple classification with subgroups, was applied [69].

The input data for this analysis is presented in Supplementary Materials Tables S2 and S3, while its course and final results are shown in Table 1. Based on the result of this analysis, it can be stated that it is the expansion possibility of the examined coal sample that exerted the strongest impact from among the analyzed factors.

Table 1. Final results of statistical verification of the correlation between the analyzed factors and the coking pressure generated by coal within the temperature range of its plasticity.

Factor	Number of Degrees of Freedom	Mean Square	Value of F-Test:		Significance of the Analyzed Factor
			Calculated	Critical Value for Significance Level 0.05	
Expansion level	4	1,810,987	11,852.01	2.52	yes
Coal	10	547,648	3584.08	1.99	yes
Grain size	45	165,830	1085.27	1.58	yes
Error	60	153			
Total	119				

3.2. Relationship between the Phenomenon of Free Swelling of Coal Grains and the Coking Pressure Generated by Their Bed

Despite a fairly good compliance of the temperature of maximal coal plasticity (as well as the dilatation temperature) with the temperature at which the maximal pressure is generated (Table 2), the data presented in Figure 7 indicate a lack of direct relationship between both of the analyzed phenomena, i.e., the free swelling of coal grains and coking pressure generated under conditions of constant carbonization space. The lack of such a relationship was also confirmed on the basis of the results of the statistical analysis. The detailed results of the examinations and the course of their statistical analysis are presented in Supplementary Materials—Tables S4–S7.

Table 2. Comparison of the temperature of maximal plasticity (and the temperature of dilatation) with the temperature at which the maximal pressure is generated.

Coal Sample	Size Fraction, mm	t_{II}, °C	t_{max}, °C	t_{pmax}, °C
Budryk	0.4–0.6	417	438	441
	1.4–1.5	419	439	443
	2.5–3.15	419	438	444
Zofiówka	0.4–0.6	432	453	430
	1.4–1.5	432	457	465
	2.5–3.15	442	456	468
Consolidation	0.4–0.6	466	475	495
	1.4–1.5	466	474	488
	2.5–3.15	466	474	493
Peak Downs	0.4–0.6	454	468	484
	1.4–1.5	457	466	481
	2.5–3.15	469	464	486

t_{II}—temperature of coal dilatation; t_{max}—temperature of maximal fluidity of coal by Gieseler; t_{pmax}—temperature corresponding with maximal coking pressure generated by plastic layer.

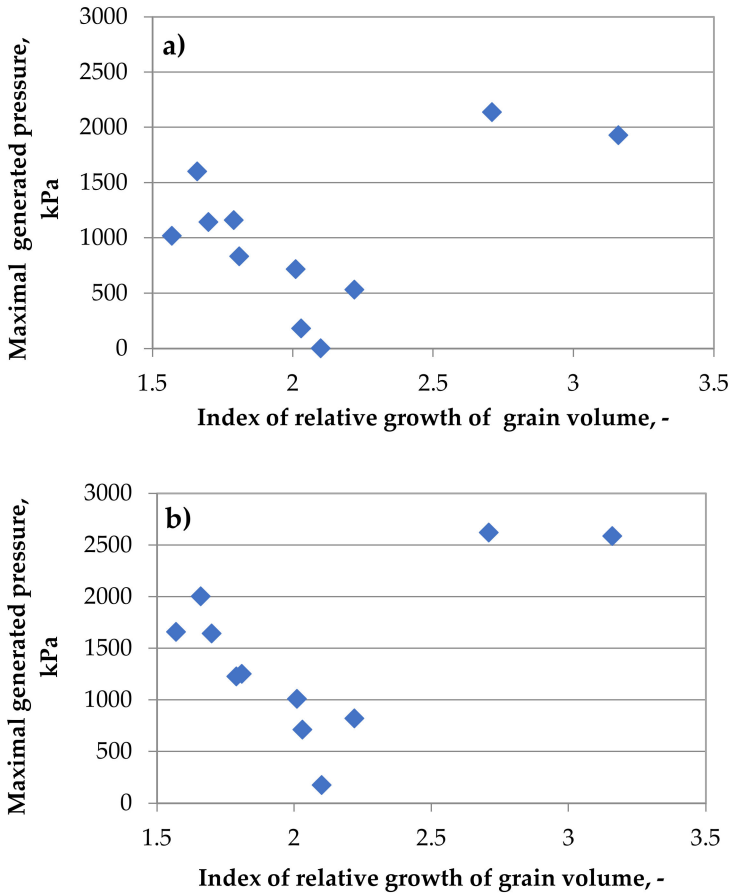

Figure 7. Comparison of maximal coking pressure generated by the plastic layer of the examined coal sample with its index of specific volume increase within the temperature range of coal plasticity: (**a**) bulk density of coal sample: 0.720 g/cm^3; (**b**) bulk density of coal sample: 0.800 g/cm^3.

It is the authors' belief that such results do not demonstrate the total lack of correlation between the free swelling of the plasticized coal grains and coking pressure generated under conditions of constant volume, but rather demonstrate the strong influence of other factors, e.g., the impact of volume expansion of the plasticized coal grain on the pressure generated inside it, different for the particular examined coals.

3.3. Impact of Plastic Layer Thickness on the Generated Coking Pressure

The impact of coal sample height on the generated coking pressure was determined. Coal samples of a coal blend with the grain size below 3 mm and bulk density equal to 0.75 g/cm^3 (detailed characteristics of the coal blend are presented in Supplementary Materials—Table S8) were examined under conditions of both constant volume and 20% expansion of the carbonized sample. The height of coal sample ranged from 2 up to 16 cm. The results of these examinations, presented in Table 3, indicate the lack of a significant influence of coal sample height (i.e., the thickness of the plastic layer) on the generated coking pressure—the value of the standard deviation of the maximal pressure for various heights of the coal sample did not exceed the value of SD determined on the basis of the aforementioned series of measurements repeated several times for the same sample.

Table 3. Values of the maximal pressure generated by the samples of the examined blend of various heights.

Height of Sample, cm	Maximal Pressure (kPa) Generated under Conditions of:	
	Constant Volume	Expansion of 20%
2	1115	945
3	1084	913
6	1083	915
9	1116	916
12	1084	947
16	1116	944
SD *	17.5	16.9

* The standard deviation value determined on the basis of the series of measurements repeated several times for the same sample was equal to 18.0 kPa.

The obtained results can be useful for explaining the mechanism of coking pressure generation by the layer of plasticized coal grains. As it was already mentioned, the hypotheses pointing at the plastic layer as the place of coking pressure generation are treated as the most reliable. As the direct source of pressure, these hypotheses assumed either the difficult flow of pyrolytic gases through the plastic layer, or the volume growth of the plasticized coal grains (caused by the gas blisters formed within the plasticized grain by the volatile products of pyrolysis).

For the first case, under conditions of the laminar flow of pyrolytic gasses and homogeneity of their internal sources, the gas flow through the plastic layer is described by the following equation [66,70,71]:

$$\frac{d}{dx}\left[\delta_w \cdot \frac{d\left(\frac{p^2}{2p_0}\right)}{dx}\right] + e_w = 0 \quad (1)$$

where p is pressure at any point inside the plastic layer, Pa; p_0 is nominal pressure of 101,325 Pa; x is distance between the analyzed point and the middle of the plastic layer, m; e_w is capacity of internal gas source per unit volume of the plastic layer, 1/s; δ_w is index of plastic layer gas permeability, s·m^3/kg.

The maximal value inside the plastic layer can be determined by solving the equation below:

$$p_{w(max)} = \sqrt{p_{zw}^2 + \frac{p_0 \cdot e_w \cdot s^2}{4 \cdot \delta_w}} \quad (2)$$

where $p_{w(max)}$ is maximal pressure inside the plastic layer, Pa; s is thickness of the plastic layer, m; p_{zw} is pressure outside the plastic layer, Pa.

According to Equations (1) and (2), along with the increase of plastic layer thickness, the pressure of pyrolytic gasses as well as the pressure generated by this layer should also increase. Therefore, the observed lack of influence of the height of a sample on the generated pressure indicates the second source, rather than the first one, as the direct cause of coking pressure generation, i.e., the lack of, or limited expansion of, the plasticized grains.

3.4. Influence of the Compression of the "Cool" Part of the Coal Charge and the Migration of the Plasticized Coal Matter Outside the Plastic Layer on the Generated Coking Pressure

Assuming that the coking pressure is generated by the plastic layer, the role of the adjacent layers of unplasticized coal grains and semicoke should be taken into account. A part of the plasticized coal matter can migrate to these layers and, therefore, an additional space for swelling coal grains is created. As a consequence, it can reduce the pressure generated by the coal layer.

This view is in line with the characteristics of coal samples taken from various places of the charge and with the shape of the plastic layers formed in coke oven chambers [68] as well

as with the results of X-ray examinations of coal char samples [72–74]. Moreover, as was already mentioned, the pressure generated by the plastic layer can compress the "cool" part of the coal charge. It can increase the space for plastic layer expansion and, consequently, decrease the pressure generated by this layer. In order to confirm the possibility of such a compression of the "cool" part of the coal charge and the possibility of migration of plasticized coal matter outside the plastic layer, as well as to confirm their influence on the generated pressure, a sequence of simulation experiments was carried out. During these experiments, the layers of graphite grains with the thickness of 10 mm functioned as the layers adjacent to the plastic layer formed during the coking process.

In order to examine only the impact of the migration phenomenon on the pressure generated by the plastic layer (without the influence of compression of the "cool" part of the coal charge), the gas-permeable membrane placed between the coal sample and the graphite layer was applied. This membrane prevented the migration of the plasticized coal matter outside the plastic layer.

The obtained results of the pressure measurements were compared with the results obtained without the use of the graphite layer and the gas permeable membrane. The examinations under conditions of the constant volume of a sample were conducted with the use of the Borynia coal samples with the size of 0–5 mm and the bulk density of 0.750 g/cm^3.

The kaolin paper with the thickness of 2 mm was used as the gas-permeable membrane. In order to simulate the migration of the plasticized coal matter in the direction opposite to that of heat flow, the examined coal sample was placed on the layer of graphite grains. In the case of the migration simulation in the direction of heat flow, the graphite layer was placed on the examined coal sample. Within the framework of the abovementioned examinations, four rounds of examinations were carried out:

- Round 1: coking of the coal sample only (i.e., without the graphite layer); during this experiment the plasticized coal matter could not migrate outside the plastic layer and the compression of adjacent layers was impossible (the lack of such layers);
- Round 2: coking of the coal sample with the graphite layer placed on it; during this experiment the plasticized coal matter could migrate (in the direction of heat flow) to the graphite layer and the compression of graphite was possible; in this case, the generated pressure could be hypothetically determined by both the abovementioned migration phenomenon and the compression of the adjacent graphite layer;
- Round 3: coking of the coal sample with the graphite layer placed on it; both layers were separated with a gas-permeable membrane; in this case the generated pressure could be hypothetically determined only by the compression of the adjacent graphite layer (the migration of the plasticized coal matter was impossible);
- Round 4: coking of the coal sample placed on the layer of graphite grains; during this experiment the plasticized coal matter could migrate (in the direction opposite to that of heat flow) to the graphite layer; in this case, the generated pressure could be hypothetically determined by both the abovementioned migration phenomenon and the compression of the graphite layer.

A schematic diagram of the experiments conducted as well as the results obtained for the Borynia coal sample (grain size fraction: 0–5 mm) is presented in Figure 8. These results confirm the fact that that both the compression of the "cool" part of the coal charge as well as the migration of the plasticized coal matter in the direction of heat flow (i.e., into the voids between the unplasticized coal grains) can determine the value of the generated coking pressure. In order to confirm the abovementioned statements, the next stage of examinations was carried out. Samples of two coals (Borynia i H. Robert) and two bulk densities of the graphite layer were examined under conditions of both the constant volume of the carbonized sample and the limited expansion of the sample. The degree of coking pressure reduction due to the compression of the graphite layer and due to the migration of the plasticized coal matter were separately determined. Characteristics of the examined

coals are shown in Supplementary Materials—Tables S9 and S10. The obtained results are presented in Table 4.

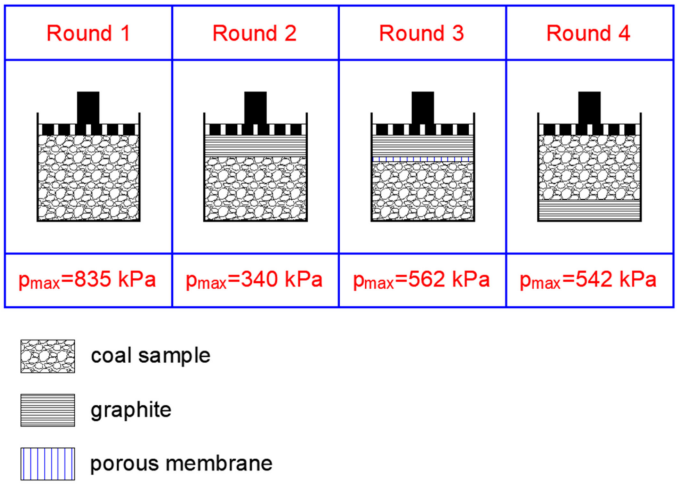

Figure 8. Schematic diagram of the experiment simulating various possibilities of the compression of the unplasticized part of the coal charge and the migration of the plasticized coal matter into layers adjacent to the plastic layer (explanations concerning particular rounds have been given in the text).

Table 4. Influence of the migration effect of the plasticized coal matter and the bulk density of the graphite layer on the maximal value of coking pressure registered during the coking test of samples of the Borynia and H. Robert coals.

Coal Sample	Bulk Density of Graphite, g/cm^3	Expansion of Coal Sample, %	P_{max} (without Compression and Migration), kPa	Decrease of Maximal Value of Generated Pressure:				
				Total	Due to Compression		Due to Migration	
				kPa	kPa	%	kPa	%
Borynia	0.75	0	835	495	273	55	222	45
		10	682	386	343	89	43	11
		20	281	234	234	100	0	0
	1.00	0	835	325	198	61	127	39
		10	682	388	388	100	0	0
		20	281	157	157	100	0	0
H. Robert	0.75	0	884	638	540	85	98	15
		10	281	203	181	89	22	11
		20	42	19	11	58	8	42
	1.00	0	884	458	365	80	93	20
		10	281	152	125	82	27	18
		20	42	23	23	100	0	0

The results presented in this table confirm, in general, the previous statements. In the case of the presented examinations, the compression constituted 55–100% of the total reduction of the generated coking pressure. The obtained results lead to the following conclusions:

- The compression of the coal grains forming the "cool" part of the coal charge in a coking chamber caused by the pressure generated within the plastic layer may result in an increase of plastic layer volume and, consequently, in a decrease of the generated coking pressure;
- The decrease of coking pressure due to "cool" charge compression depends on both the individual properties of coal (the ability to generate high pressure) and on the bulk density of the coal charge;

- The phenomenon of plasticized coal matter migration in the direction of heat flow may have an important impact on the coking pressure generated by the plastic layer. This impact, however, may be observed only in the case of a sufficiently high pressure generated by this layer. In the case of an insufficiently high pressure, the plasticized coal matter is not able to fill the voids between coal grains of the "cool part" of the coal charge. Such different behaviors were noticed in the laboratory examinations under conditions of the various expansion possibilities of the examined coal samples;
- The impact of plasticized coal matter migration on the generated coking pressure depends on both the individual properties of coal (such as the ability to generate a high internal coking pressure, and the fluidity of the plasticized coal matter) and the bulk density of the coal charge; in the case of coals forming the plasticized coal matter of a sufficiently high fluidity, the compression effect can be reduced as a result of filling the voids between grains of the "cool" part of the charge with the plasticized coal matter.

An additional comparative analysis was carried out in order to identify the factors influencing the abovementioned migration. The effects of grain size fraction, the fluidity of the plasticized coal matter, the bulk density, and various degrees of expansion on the difference between the pressure generated under conditions of the migration of the plasticized coal matter and the pressure generated under conditions of the lack of migration possibility were statistically verified.

The design of the experiment based on the Greek–Latin square scheme with repetitions was applied. The design of the experiment and the levels of the analyzed factors are shown in Table 5. Characteristics of the examined samples are shown in Supplementary Materials—Tables S11 and S12. The detailed results of the measurements and the course of the statistical analysis are given in Supplementary Materials—Tables S13 and S14. The final result of the analysis demonstrates that all the analyzed factors, i.e., grain size fraction, bulk density, level of the possible expansion of the coal sample, and fluidity of the plasticized coal matter, have a significant influence on the migration phenomenon and, therefore, on the value of the generated coking pressure. It should be pointed out that the factors exerting the strongest influence on the coking pressure are the bulk density, followed by the fluidity of the plasticized coal matter.

Table 5. Design of the experiment.

Levels of Factor	I	II	III
1	*A; α*	*B; γ*	*C; β*
2	*B; β*	*C; α*	*A; γ*
3	*C; γ*	*A; β*	*B; α*

1, 2, 3—levels of grain size fraction of coal sample: <5 mm; <3 mm; <2 mm; I, II, III—levels of maximal coal fluidity, Fmax: 5208 ddpm; 2949 ddpm; 2185 ddpm; A, B, C—levels of bulk density: 0.750 g/cm^3; 0.850 g/cm; 0.950 g/cm^3; α,β,γ—levels of coal sample expansion during examination: 0%; 10%; 20%.

3.5. Preliminary Assessment of the Usefulness of Laboratory Measurements for the Identification of Dangerous Coals (in Respect of Generation of an Excessively High Wall Pressure)

A high value of the coking pressure generated by the plastic layer under conditions of its constant volume does not necessarily indicate that this coal will also generate an excessively high wall pressure in industrial coke chambers, since the wall pressure is determined also by a number of phenomena that occur in the remaining areas of the coal charge, i.e., in the "cool" charge, in semicoke, and in coke.

Taking into account the abovementioned facts, another round of examinations was carried out (detailed characteristics of coal blend are presented in Supplementary Materials—Table S15). The pressure generated by the plastic layer of selected coals was measured during carbonization both under conditions of the constant volume and under conditions of the limited expansion of the carbonized sample. The limited expansion for particular coals corresponded to the coal shrinkage during carbonization determined by the Sapozhnikov

method [75]. Table 6 presents the obtained values of this shrinkage. This table also contains the obtained values of pressure generated by the plastic layer during carbonization both under conditions of the sample expansion corresponding to the shrinkage of the resulting semicoke, as well as under conditions of the constant volume of the carbonized sample. The results presented in this table confirm that the high coking pressure generated by the plastic layer under conditions of the constant volume of the carbonized sample does not necessarily indicate that the examined coal will generate an excessively high wall pressure in industrial coke ovens.

Table 6. Comparison of the coking pressure generated by the plastic layer during carbonization under conditions of the limited expansion (corresponding to the coke shrinkage of the examined coals) and under conditions of the constant volume of the carbonized sample).

Coal Sample	Shrinkage by Sapozhnikov		Maximal Pressure (kPa) Generated under Conditions of:	
	mm	%	Constant Volume	Limited Expansion
Budryk	34	68	1594	0.2
Szczygłowice	28	56	752	0
Pniówek	28	56	1070	0
Borynia	24	48	863	0
Zofiówka	10	20	717	0
Burton	15	30	210	0
H. Robert	5	10	1189	21

For the purposes of the identification of coals generating a dangerous wall pressure, the laboratory scale methods may be useful; however, the tests should be conducted under conditions of the expansion of the carbonized samples corresponding to the coal shrinkage of the resulting coke.

According to [76,77], significant differences between particular coals are observed for temperatures lower than the temperature of plasticized coal matter resolidification; while above this temperature, the shrinkage of the resulting coke is similar for all coals.

The results of the Sapozhnikov test fairly well describe the diversified shrinkage of particular coals [75]. Therefore, the authors believe that the applied laboratory method combined with the Sapozhnikov test of the coal matter shrinkage during carbonization can be useful for the preliminary identification of dangerous coals (in respect of the generation of an excessively high pressure). For the preliminary verification of this belief, the wall pressure measurements for four selected coals were carried out with the use of a test oven with a moving wall—Table 7. The final conclusion concerning the usefulness of such a method for the dangerous coals identification requires, however, obtaining examination results for a much larger number of coals.

Table 7. Comparison of the coking pressure measured in the laboratory equipment under conditions of the expansion corresponding to the shrinkage determined by the Sapozhnikov method with the wall pressure measured in a test oven with a moving wall.

Coal Sample	Expansion Degree of the Examined Coal Sample, %	Maximal Pressure Exerted on the Measuring Piston, kPa	Pressure Exerted on the Wall of the Test Oven with a Moving Wall, kPa
Borynia	48	0	0
Burton	30	0	0
Zofiówka	20	0	0
H. Robert	10	21	21

4. Conclusions

(1) The coking pressure generated by the plastic layer is a result of the increase of plasticized coal grains volume under conditions of the limited possibility of their swelling. Within the whole temperature range of coal plasticity, coal grains preserve their shape (they do not melt), although they change their sizes. For this reason, they do retain their pressure generation ability.
(2) The expansion ability of plasticized coal grains is a crucial factor determining the value of the coking pressure generated by the plastic layer.
(3) The increase of plastic layer volume is possible due to both the compression of the "cool" part of the coal charge and the migration of the plasticized coal matter in between coal grains of this part of the coal charge.
(4) The compression rate of the "cool" part of the coal charge depends on its bulk density as well as on the value of the coking pressure generated by the plastic layer.
(5) The ability of the plasticized coal matter to migrate in between coal grains of the "cool" part of the coal charge depends on such factors as the bulk density of the coal charge, the fluidity of the plasticized coal matter, and, subsequently, the size of coal grains.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/en15062044/s1, Table S1: Characteristics of the examined coals; Table S2: Maximal values of pressure generated by the examined coal samples; Table S3: Measurement results of maximal pressure (kPa) generated within the temperature range of coal plasticity by selected grain size fractions—input data for the conducted statistical analysis; Table S4: Characteristics of the examined coals; Table S5: Comparison of indices of the relative grain volume increase of examined coals (kV) with values of the maximal generated pressure (kPa) for different bulk densities of samples; Table S6: Course and final results of significance analysis of linear correlation (y = bx + a); Table S7: Course and final results of the Spearman test—verification of the correlation between the index of relative increase of plasticized coal grain and maximal pressure generated by the bed of such grains (for bulk density of coal sample equal to 0.72 and 0.80 g/cm3); Table S8: Characteristics of the examined coal blend; Table S9: Characteristics of the coals used for examinations; Table S10: Grain size distribution of the examined coals; Table S11: Characteristics of the examined coals; Table S12: Grain size distribution of the examined coals; Table S13: Results of coking pressure examinations (kPa) according to the design of the experiment; Table S14: Statistical significance of the analyzed factors influencing the migration phenomenon in respect of the value of the generated coking pressure; Table S15: Characteristics of the examined coals (size fraction: 0–3.15 mm).

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