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RESEARCH ON THE DYNAMICS OF THE BOF SLAG REDUCTION PROCESS

BADANIA DYNAMIKI PROCESU REDUKCJI ŻUŻLA KONWERTOROWEGO

The publication presents research on the converter slag reduction process dynamics in an induction furnace. Before the experiments were commenced, the chemical composition of the non-metallic phase in the equilibrium state had been computed. The calculations were carried out with the FactSage package. Thermodynamic databases were used for a provisional determination of the reduction process conditions. Next, a number of experiments were carried out to verify the calculations. The experiments were carried out for various process times.

Bauxite was added to reduce the process temperature. As a result of the experiments performed, a material of similar chemical composition as clinker with an increased Al_2O_3 content was obtained. The test results were compared to similar findings obtained abroad.

W publikacji przedstawiono badania dynamiki procesu redukcji żużla konwertorowego w piecu indukcyjnym. Przed rozpoczęciem eksperymentów dokonano obliczeń składu chemiczne otrzymywanej fazy niemetalicznej w stanie równowagi. Obliczenia prowadzono przy pomocy komputerowego pakietu FactSage. Wykorzystując termodynamiczne bazy danych dokonano wstępnego określenia warunków prowadzenia procesu redukcji. Następnie przeprowadzono szereg eksperymentu w celu zweryfikowania przeprowadzonych obliczeń. Eksperymenty prowadzono dla różnych czasów trwania procesu. W celu obniżenia temperatury procesu stosowano dodatki boksytu. W wyniku wykonanych eksperymentów otrzymano materiał o składzie zbliżonym do składu chemicznego klinkieru o podwyższonej zawartości Al₂O₃. Uzyskane wyniki badań porównano z podobnymi wynikami uzyskiwanymi w świecie.

1. Introduction

Research with the objective of finding a method of utilizing the ever-growing mass of steelmaking slag has been carried out for many years across the world. The BOF slag reduction process is a utilization method of the waste material. The non-metallic phase obtained in this way may be used as Portland clinker, calcium-magnesium fertilizer or slag-forming material for the secondary steelmaking. Conditions of the BOF slag reduction process have as yet not been clearly determined, preventing the method's effective use in the industry.

During the reduction process with the participation of a carbon reducer, carbon monoxide bubbles form at the interface, causing the phase reduced to foam. The reaction rate depends on the physical and chemical characteristics of the material reduced as well as the temperature and reducer type. However, it is assumed that the diffusion of the Fe^{+2} and O^{-2} ions in the foamed slag phase is the slowest stage of the process. Along with the increase in the temperature, the dynamic viscosity of the material reduced declines, thus improving the conditions for ion movement in the phase.

In the study the authors have presented their findings on the converter slag reduction process conditions and dynamics on the basis of the FactSage software thermodynamic databases. The computations conducted have been used to determine the impact of the bauxite addition and the reducer participation on the non-metallic phase obtained. The computations have been verified by experiments in an induction furnace.

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2. The non-metallic phase characteristics depending on its intended use

The BOF slag liquidus temperature is usually above 1800°C due to the low Al_2O_3 content in relation to CaO and SiO₂. Therefore it is advisable that the reduction process of the slag be carried out in electric arc furnaces. If an induction furnace were used, the process would have to be carried out in the solid state, which would significantly extend the reaction time. The addition of bauxite, which is an Al_2O_3 carrier, to the charge reduced may be an intermediate solution, as a small addition of bauxite (about 10% of the reduced slag mass) enables the liquidus temperature to be lowered to below 1600°C. Due to economic considerations the bauxite addition in the charge should be as small as possible.

The Portland clinker may be one of the materials resulting from the reduction process. Its chemical composition is: $63 \div 67\%$ CaO, $21 \div 24\%$ SiO₂, $4 \div 7\%$ Al₂O₃, $1,8 \div 3,7\%$ FeO, max. 5% MgO, max. 2% P and max. 1,5% SO₃ [1]. The non-metallic phase from the reduction reaction intended for Portland clinker must not undergo a self-disintegration, which results from the allotropic transformation of the $\beta 2CaO \cdot SiO_2$ into the γ phase. The structure of the γ form is more symmetric than of the β phase. Less symmetry planes in the β form favours the penetration of water particles deep into the crystal planes, which improves the hydraulic bounding characteristics of the β form as compared to the γ phase.

As a result of research carried out for years it has been found that the stabilization of the β form depends on the chemical composition of the resulting phase and conditions of its cooling. Ferrous and manganese oxides present in the slag stabilize the β form. However, the most important stabiliser is P₂O₅, which prevents the allotropic transformation of the β phase into the γ form if its content exceeds 0.3% molar [1]. If there are no stabilisers, intensive cooling is necessary.

The obtaining of the Portland clinker as a result of the converter slag reduction requires to carry out the process at very high temperatures, due to the high liquidus temperature of the slag. Therefore, also attempts to obtain clinker with an increased Al_2O_3 content from a reduced mixture of BOF slag and bauxite have been made. It allows the reduction process temperature to be lowered under 1600°C [2, 3].

Another material that can be obtained as a result of the converter slag reduction process is calcium-magnesium fertiliser [1]. It is assumed that as a rule materials containing less than 5.5% FeO and 2.0% MaO are accepted as fertilisers by authorized bodies. However, it should be expected that ever growing environmental requirements would cause a further restriction

of FeO and MnO content in the materials applied. It is also vital that the reduced phase self-disintegrate after the reduction process, thus allowing the material to be used as a fertiliser without the need of additional fragmentation. Theoretically, it is possible that the non-metallic phase self-disintegrates during an attempt to obtain the Portland clinker. Although the material obtained this way is no longer suitable for Portland clinker, it can be successfully used as a calcium-magnesium fertiliser.

Another method for convertor slag utilisation is its reduction with a bauxite or lime addition to obtain a refining slag-forming mixture used in the secondary steelmaking. The refining slag resulting from the reduction should be characterised by a low viscosity and a liquidus temperature of about 1350°C [4]. The most favourable materials of this type are synthetic calcium-aluminium slags formed by melting lime and aluminium oxide carriers in an electric arc furnace or by sintering them in a gas-fired rotary furnace. Because of the high price of pure aluminium oxide, bauxite is used commercially, which apart from Al₂O₃ also brings into the slag-forming mixture significant amounts of other compounds such as SiO₂, FeO and TiO₂. Ferrous and titanium oxides present in bauxite lower the refining qualities of slag. Consequently, research aimed at the production of the slag-forming refining mixture of as low FeO and TiO₂ content as possible has been carried out abroad [5, 6] for many years.

The non-metallic phase resulting from the reduction process of ladle slags containing high amounts of Al_2O_3 with low liquidus temperatures may be an alternative to the material obtained as a result of the reduction of BOF slag with a bauxite addition. The application of such a solution would enable the cost of obtaining a slag-forming mixture for secondary steelmaking to be additionally reduced.

3. Research on the dynamics and conditions of conducting the BOF slag reduction process

To determine the dynamics and conditions of the BOF slag reduction process, a number of experiments were carried out in laboratory conditions with the use of an induction furnace. Five series of heats were made, the bauxite addition and the carbon reducer share in the phase reduced effect on the process dynamics were examined, the contents of bauxite and carbon reducer were adjusted on the basis of thermodynamic databases of the FactSage package for the temperature of 1650°C. The mass of the base BOF slag reduced in each sample was 500g +/-1g plus a bauxite addition of 10-20%, of chemical composition: 88% Al₂O₃, 6% SiO₂, 3,85% TiO₂, 1,5% FeO and 0,3% CaO. The reduction process of the

resulting mixtures was carried out at a temperature of ca. $1650^{\circ}C$ +/- $20^{\circ}C$ for various time intervals Δt , understood as the time period between the induction furnace being turned on and the moment at which the crucible is taken out of the furnace. Having finished the experiment, the samples were left for self-cooling at room temperature and they were then crushed and ground in order that chemical analysis be performed. The chemical

composition of the base BOF slag and its mixtures with bauxite is presented in table 1. The liquidus temperature of the BOF slag applied was above 1800°C, the liquidus temperature of the mixtures about 1530°C. Increasing the bauxite share applied did not cause any significant decrease in the liquidus temperature of the mixtures examined.

TABLE	1

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No	Mixture	Bau	xite				Chemical	l composi	ition befo	re reduc	tion, %			
110.	g g	g	%	MnO	FeO	MgO	Al_2O_3	SiO ₂	CaO	TiO ₂	K ₂ O	Cr ₂ O ₃	P_2O_5	S
b.s.	500	0	0	2,36	20,75	4,56	0,84	17,76	47,79	0,18	0,80	1,60	1,07	0,05
							Series 1							
1	550	50	10	2,14	19,00	4,14	8,75	16,69	43,47	0,51	0,72	1,45	0,97	0,05
2	600	100	20	1,96	17,54	3,80	15,35	15,80	39,87	0,79	0,66	1,33	0,89	0,04
							Series 2							
3	560	60	12	2,11	18,69	4,07	10,17	16,50	42,70	0,57	0,71	1,43	0,96	0,04
4	565	65	13	2,09	18,54	4,04	10,86	16,41	42,33	0,60	0,71	1,42	0,95	0,04
5	570	70	14	2,07	18,39	4,00	11,54	16,32	41,96	0,63	0,70	1,40	0,94	0,04
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The chemical composition of the BOF slag and mixtures reduced

b.s. – BOF slag

The Equilib software of the FactSage package was used for modelling the non-metallic phase in the state of equilibrium. The reducer share for series 1 was 6% of the mass of the slag reduced and was adjusted so as to the amount of solid graphite precipitates in the theoretical stationary state was 0, and the amount of MnO and FeO was as low as possible for a set bauxite addition. The calculated mass of solid precipitates in the equilibrium state was about 11% for mixture no. 1 and 0 for mixture no. 2. The chemical composition of the non-metallic phase obtained for various time intervals is presented in tables 2 and 3. The chemical composition examples of the metallic phase obtained at the same time are presented in tables 4 and 5.

TABLE 2

The chemical composition of the non-metallic phase obtained during the reduction of the mixture no. 1 carried out with the carbon reducer share of 6% of the BOF slag mass

Δt,	Mass,		Chemical composition, %										
min	g	MnO	FeO	MgO	Al_2O_3	SiO ₂	CaO	TiO ₂	K ₂ O	Cr_2O_3	P_2O_5	S	
11	468	2,02	9,44	4,43	10,28	19,60	51,06	0,59	0,54	1,05	0,73	0,05	
15	460	1,94	8,01	4,50	10,46	19,95	51,96	0,60	0,47	0,91	0,78	0,05	
19	433	1,51	2,90	4,79	11,12	21,22	55,27	0,64	0,41	0,77	0,41	0,06	
25	424	1,08	1,41	4,89	11,36	21,66	56,41	0,65	0,31	0,55	0,34	0,06	
30	420	0,80	0,73	4,94	11,47	21,88	56,99	0,66	0,23	0,37	0,22	0,06	
St.	403	0,09	0,02	5,14	11,95	22,76	59,26	0,68	0,05	0,01	0,00	0,00	

St. – stationary state

TABLE 3

Δt,	Mass,				Chemical composition, %							
min	g	MnO	FeO	MgO	Al_2O_3	SiO ₂	CaO	TiO ₂	K ₂ O	Cr_2O_3	P_2O_5	S
10	550	1,95	13,22	3,98	16,73	17,10	43,48	0,81	0,50	0,95	0,82	0,05
12	524	2,01	9,21	4,18	17,58	17,97	45,69	0,85	0,47	0,88	0,61	0,05
14	497	1,91	4,65	4,41	18,54	18,95	48,17	0,89	0,44	0,80	0,50	0,05
17	494	1,86	4,19	4,43	18,64	19,05	48,44	0,90	0,39	0,69	0,52	0,05
19	477	1,62	1,31	4,58	19,28	19,71	50,11	0,93	0,36	0,61	0,34	0,05
21	476	1,55	1,13	4,59	19,33	19,76	50,24	0,93	0,33	0,54	0,31	0,05
St.	453	0,20	0,01	4,83	20,32	20,77	52,72	0,98	0,11	0,01	0,00	0,00

The chemical composition of the non-metallic phase obtained during the reduction of the mixture no. 2 carried out with the carbon reducer share of 6% of the BOF slag mass

TABLE 4

Examples of the chemical composition of the metallic phase obtained during reduction of mixture no. 1

Δt,		on, %				
min	Fe	Mn	Cr	Ni	Pb	С
15	91,80	0,35	0,12	0,45	0,35	>4,50
19	93,30	0,60	0,12	0,25	0,22	>4,50
25	92,20	0,77	0,16	0,34	0,33	>4,50
30	92,20	1,60	0,51	0	0	3,16
St.	78,77	8,08	5,29	Nz	Nz	4,96

TABLE 5

Examples of the chemical composition of the metallic phase obtained during reduction of mixture no. 2

Δt,		Chen	nical co	mpositio	on, %	
min	Fe	Mn	Cr	Ni	Pb	С
17	93,20	0,43	0,12	0,28	0,25	>4,50
21	92,00	0,86	0,14	0,34	0,29	>4,50
St.	79,05	7,60	5,27	Nz	Nz	4,87

Fine metal drops suspended in various parts of the material reduced were obtained in the experiments. Where most of the reduced drops formed a uniform mass at the crucible bottom, its chemical composition was determined. It was possible when longer reduction times were used.

In series 2, the effect of the carbon reducer share on conditions and dynamics of the BOF slag reduction process was tested. Thermodynamic databases were used for matching such pairs of reducer-bauxite additions that the sum of solid non-metallic phase precipitates in the equilibrium state at the temperature of 1650°C was 0, the sum of FeO and MnO oxides did not exceed ca. 1%, and the bauxite addition was as low as possible. The values obtained are presented in table 6. Next the calculated oxide contents were compared with the values obtained in the process of reduction of mixtures no 3, 4 and 5. Tables 7, 8 and 9 present the chemical compositions of the non-metallic phases obtained.

TABLE 6

Impact of the reducer on the MnO and FeO content in the theoretical state of equilibrium of the reduced phase in series 2

Series	Bauxite addition, %	Carbon reducer share, %	MnO	FeO	MnO+FeO
3	12	5	0,91	0,14	1,05
4	13	5,5	0,44	0,05	0,49
5	14	6	0,14	0,01	0,15

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TABLE 7

The chemical composition of the non-metallic phase obtained during the reduction of the mixture no. 3 carried out with the carbon reducer share of 5% of the BOF slag mass

Δt,	Mass,		Chemical composition, %										
min	g	MnO	FeO	MgO	Al_2O_3	SiO ₂	CaO	TiO ₂	K ₂ O	Cr_2O_3	P_2O_5	S	
12	455	1,99	4,45	4,95	12,52	20,31	52,57	0,70	0,49	0,86	0,73	0,05	
15	452	1,79	4,01	4,98	12,61	20,46	52,95	0,71	0,44	0,72	0,73	0,06	
18	443	1,80	2,27	5,08	12,84	20,84	53,93	0,72	0,39	0,58	0,68	0,06	
21	436	1,44	1,08	5,16	13,07	21,20	54,86	0,73	0,33	0,44	0,53	0,06	
25	435	1,34	0,99	5,17	13,09	21,24	54,97	0,73	0,26	0,25	0,50	0,06	
St.	420	0,91	0,14	5,36	13,56	22,00	56,86	0,76	0,17	0,01	0	0	

TABLE 8

The chemical composition of the non-metallic phase obtained during the reduction of the mixture no. 4 carried out with the carbon reducer share of 5,5% of the BOF slag mass

Δt,	Mass,		Chemical composition, %										
min	g	MnO	FeO	MgO	Al_2O_3	SiO ₂	CaO	TiO ₂	K ₂ O	Cr_2O_3	P_2O_5	S	
12	469	1,87	6,11	4,73	13,07	19,73	50,98	0,72	0,49	0,89	0,59	0,05	
15	450	1,71	2,56	4,92	13,61	20,56	53,12	0,75	0,43	0,76	0,52	0,06	
18	444	1,62	1,31	5,00	13,81	20,85	53,89	0,76	0,38	0,63	0,36	0,06	
21	445	1,30	1,92	4,98	13,77	20,79	53,72	0,76	0,32	0,49	0,33	0,06	
25	438	0,89	0,79	5,06	14,00	21,13	54,61	0,77	0,25	0,32	0,29	0,06	
St.	423	0,44	0,05	5,24	14,49	21,88	56,84	0,80	0,12	0,01	0,00	0,00	

TABLE 9

The chemical composition of the non-metallic phase obtained during the reduction of the mixture no. 5 carried out with the carbon reducer share of 6% of the BOF slag mass

Δt,	Mass,		Chemical composition, %										
min	g	MnO	FeO	MgO	Al_2O_3	SiO ₂	CaO	TiO ₂	K ₂ O	Cr_2O_3	P_2O_5	S	
12	473	1,92	6,26	4,52	13,88	19,59	50,56	0,73	0,49	0,92	0,64	0,05	
15	460	1,75	3,97	4,64	14,26	20,13	51,97	0,75	0,43	0,80	0,45	0,05	
18	445	1,52	1,10	4,80	14,75	20,81	53,73	0,77	0,38	0,69	0,29	0,05	
21	444	1,33	1,08	4,81	14,78	20,86	53,85	0,77	0,33	0,57	0,36	0,05	
25	442	1,26	1,04	4,83	14,84	20,95	54,08	0,78	0,26	0,41	0,30	0,05	
St.	423	0,14	0,01	5,05	15,52	21,90	56,40	0,81	0,08	0,01	0,00	0,00	

Obtaining a non-metallic phase of a composition as similar to the stationary state as possible, in practice turned out to be difficult to be achieved. To this end, it would be necessary to extend the reduction process significantly, which would lead to an increase in the reduction degree and result in the allotropic transformation of the β 2CaO·SiO₂ into the γ form. A stable β phase form can be obtained in the laboratory conditions by conducting the reduction process for about 21-25 min. At the same time, a low ferrous and manganese oxide content may be obtained, the total not exceeding 2.5%. The reducer share and bauxite addition should be adjusted on the basis of calculations performed for the state of equilibrium, so that the sum of MnO and FeO does not exceed 1%.

The metallic phase examinations show that a uniform mass can only be obtained if the reduction time is extended. If reduction times in the non-metallic phase are short, many fine solidified particles of metal drops, which did not manage to fall onto the crucible bottom, are obtained. This necessitates the crushing of the non-metallic phase obtained and the separation of the solidified metal drops. If the reduction times are longer and a uniform metal mass at the crucible bottom is formed, there is no need for it to be separated from the resulting non-metallic phase. The main component of the metallic phase obtained was iron, and its content exceeded 90%. This phase contained a small amount of manganese and usually over 4% of carbon.

The experiments conducted indicate that the change in the temperature during the process has a significant impact on the dynamics of the ferrous oxide reduction. Due to the gradual heating of the induction furnace during consecutive heats, the physical conditions of the reduction reaction change. The problem is additionally complicated by the fact that the whole process is controlled by an operator, which manually sets the furnace power on the basis of the subjectively determined charge temperature. The use of optical temperature measurement and a computer to control the process in real time would allow more predictable and repeatable results to be obtained by minimising the human factor in the process. If the temperature cannot be measured the benchmark enabling the reduction process time to be determined is the end of the intensive slag foaming period. After the foam has completely disappeared, the process should still be continued for about 3-5 minutes.

4. Examinations of possibilities for obtaining clinker with an increased Al2O3 content as a result of the BOF slag reduction process

In order to determine the possibilities for obtaining clinker with an increased Al_2O_3 content, series 3 of heats was produced to examine the BOF slag reduction reaction. Mixture no. 1 of a composition presented in table 1 was used as the charge. The objective of the experiment was to obtain the non-metallic phase with its composition similar to the clinker obtained in the Belgian research [2] and the verification of the repeatability of own research findings. The reduction process was conducted in an induction furnace at a temperature of about 1650°C +/-20°C. The mixture had been blended with the carbon reducer before the reduction process started. The reducer share in all tests conducted was 6%of the BOF slag mass brought in by the mixture. The mass of the charge reduced was 550g. Table 10 presents the chemical composition of the output BOF slag and the non-metallic phase obtained in the Belgian research. Own findings are presented in table 11. The process time was similar for each heat and was about 22 min. from the moment of the induction furnace being turned on to the moment at which the crucible is taken out of the furnace. The reaction completion time was from 16 to 18 min and varied for each sample.

TABLE 10

The composition of the BOF slag and the non-metallic phase obtained in the industrial conditions in the Belgian research

			Chemical composition, %										
No.	materiał	MnO	FeO	MgO	Al_2O_3	SiO ₂	CaO	Cr_2O_3	P_2O_5	S			
1	BOF slag	5,70	27,66	3,00	0,90	12,90	46,40	0,10	2,00	0,10			
2	Non-metallic phase	2,20	4,11	4,90	11,10	21,40	53,20	-	0,70	0,10			

TABLE 11

The chemical composition of the mixture reduced and the resulting non-metallic phases

			Chemical composition, %									
No.	Material	MnO	FeO	MgO	Al_2O_3	SiO ₂	CaO	TiO ₂	K ₂ O	Cr_2O_3	P_2O_5	S
2	Mixture 1	2,14	19,00	4,14	8,75	16,69	43,47	0,51	0,72	1,45	0,97	0,05
4	N.p. 1	1,14	1,25	4,84	11,31	21,54	56,52	0,66	0,34	0,60	0,27	0,06
5	N.p. 2	1,08	0,82	4,82	11,40	21,72	58,48	0,67	0,38	0,63	0,36	0,06
6	N.p. 3	1,44	1,13	4,75	11,29	21,57	56,24	0,64	0,30	0,57	0,42	0,06
7	Stationary state	0,09	0,02	5,14	11,95	22,76	59,26	0,68	0,05	0,01	0,00	0,00

N.p. – Non-metallic phase

The chemical composition of the non-metallic phase obtained in our own research intended for clinker with an increased Al_2O_3 content is similar to the composition of the phase obtained in the Belgian research. In addition, the resulting non-metallic phase is characterised by a lower content of manganese and ferrous oxides. However, the oxide content, including P_2O_5 , was so high that the resulting material would not self-disintegrate during slow cooling.

By comparing the chemical composition of the resulting non-metallic phases with the chemical composition of Portland clinker it has been found that the CaO content in all cases was about 7% lower than the required value for the clinker. At the same time a higher Al_2O_3 content was obtained. Therefore the obtaining of the Portland clinker as a result of the BOF slag reduction requires the addition of calcium to the charge before the reduction process is started or the use of BOF slag with a very high CaO content, much higher than 50%. However, it should be emphasised that the reduction process temperature increases with the increase in the CaO content in the charge [1, 7].

The resulting non-metallic phases may still be used as the base material for metallurgical cement production, containing CaO about 50%, Al_2O_3 over 9%, and SiO_2 even up to 30%. The reduced material may be blended with blast-furnace slag resulting in the metallurgical cement.

5. Conclusion

The reduction process conditions, such as the temperature, reducer share and an appropriate selection of input materials may be provisionally determined with the FactSage computer package. The thermodynamic databases contained in the package allow the desired chemical composition of the non-metallic phase in the equilibrium state to be obtained depending on the reduction process conditions. The shorter the process time the more discrepancies occur between the chemical composition of the actual non-metallic phase and the chemical composition that can be obtained in the state of equilibrium.

The examination on the BOF slag reduction process dynamics enabled the relationship between the reduction reaction rate and the chemical composition of the material and the share of reducer used during the process to be determined approximately. By analysing the laboratory findings it has been found that the reduction process time after completion of the intensive foaming of the phase reduced should depend on its intended use. Also the rate of reaching the set temperature has a significant impact on the process dynamics. It is advantageous to obtain the set temperature as soon as possible, and it is unadvisable to exceed 1700°C in an induction furnace, as this results in a much faster wear of the graphite lining.

The BOF slag reduction process in the liquid state can only be carried out in an EAF, due to the fact that the liquidus temperature of liquid slag usually exceeds 1800°C. If the BOF slag reduction process is to be conducted in an induction furnace, the process temperature needs to be lowered. The simplest method of lowering the temperature is to add some amount of bauxite to the charge. The mass of bauxite needed to lower the process temperature to the assumed value may be calculated with thermodynamic databases contained in the FactSage package.

The conducted experiments show that the reducer share in the charge must be increased for the assumed total amount of ferrous and manganese oxides to be obtained. However, there is a boundary reducer share, above which no significant reduction of the total amount of these oxides in the reduced material can be observed. Further reduction of these oxides contents is only posThe findings of the experiments performed confirm that obtaining Portland clinker as a result of the BOF slag reduction process requires lime to be added to the charge reduced to increase the CaO content in the material obtained. The chemical composition of the non-metallic phase obtained in the BOF slag reduction process corresponds to the chemical composition of the low CaO clinker. Therefore this phase may be used for the metallurgical cement production after blending with the blast furnace slag.

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