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SINTER REDUCTION IN SIMULATED BLAST-FURNACE CONDITIONS IN THE PRESENCE OF COKE WITH VARIOUS REACTIVITY

REDUKCJA SPIEKU W SYMULOWANYCH WARUNKACH WIELKIEGO PIECA Z UDZIAŁEM KOKSU O RÓŻNEJ REAKCYJNOŚCI

The examinations of the extent of sinter reduction were carried out in simulated conditions of a blast – furnace at the temperatures of 950 and 1100 °C. Two cokes with different reactivity were applied. An analysis was carried out to determine whether it was possible for the CO_2, formed *in situ* as a result of sinter reduction by means of CO, to react with coke. It was shown, that at the temperature of 950 °C the reaction of CO_2 with coke did not occur. The reduction of iron – bearing materials at that temperature occurs by way of indirect reduction without the participation of coke carbon. With the growth of temperature up to about 1100 °C the reaction $CO_2 + C = 2CO$ becomes an important process influencing the course of the reduction and coke consumption. The effect of coke reactivity on the course of direct reduction was also analysed. Increasing of coke reactivity results an increase in the amount of coke for the reaction $CO_2 + C = 2CO$. The type of packing of the reduced sinter and coke influences the rate of the material reduction.

Przeprowadzono badania redukcji spieku w symulowanych warunkach wielkiego pieca w temperaturach 950 i 1100°C. Stosowano dwa koksy o różnej reakcyjności. Analizowano możliwość reakcji dwutlenku węgla, powstającego *in situ* w wyniku redukcji spieku tlenkiem węgla, z koksem. Wykazano, że w temperaturze 950°C nie obserwuje się reakcji dwutlenku węgla z koksem. Redukcja tworzyw żelazonośnych w tej temperaturze odbywa się na drodze redukcji pośredniej bez udziału węgla koksu. Ze wzrostem temperatury do ok. 1100°C reakcja $CO_2 + C = 2CO$ staje się istotnym procesem wpływającym na przebieg procesu redukcji i zużywanie koksu na redukcję bezpośrednią tlenków żelaza. Analizowano wpływ reakcyjności koksu na przebieg redukcji bezpośredniej. Obserwowano zwiększenie zużycia koksu na redukcję bezpośrednią ze wzrostem jego reakcyjności. Zmiana wzajemnego ułożenia redukowanego spieku i koksu zwiększa intensywność redukcji tworzywa.

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1. Introduction

Blast furnace coke is one of the most important factors influencing the economic efficiency of the steel production process. The decreased coke consumption, as well as the use of cheaper substitutes of blast furnace coke, make coke quality especially important. The mechanical properties of coke, its granulation and reactivity as the important criteria determining the coke suitability for the blast furnace process have been discussed in a number of works [1–6]. Coke reactivity, particularly when determined by the Nippon Steel Co. method (CRI index), and its mechanical properties constitute equally important factors determining its quality. An evaluation of coke reactivity makes it possible to draw conclusions concerning the rate of the reaction of CO_2 with carbon contained in coke and, consequently, the possibility of the reduction of iron oxides by way of direct reduction:

$$\mathrm{FeO} + \mathrm{CO} = \mathrm{CO_2} \tag{1}$$

$$\underline{\mathrm{CO_2} + \mathrm{C} = 2\ \mathrm{CO}} \tag{2}$$

$$\mathrm{FeO} + \mathrm{C} = \mathrm{CO} + \mathrm{Fe}\,. \tag{3}$$

The direct reduction is on the one hand an endothermic reaction requiring a significant amount of heat; on the other hand, it utilises the reduction potential of carbon more efficiently. Attempts to reduce coke consumption in the blast furnace process led to a conclusion that, for each variant of blast furnace operation, there exists an optimal contribution of direct reduction to the process of oxygen removing from the iron-bearing materials, for which there occurs the lowest coke consumption. This optimum exists for such a contribution of the direct reduction for which the carbon consumption covering the heat request and that required to remove oxygen from oxides are equal. This contribution usually falls within the range of 20 ÷ 30 %. The ratio between the contribution of the direct reduction and that of the indirect reduction (eq.1) depends also on the utilisation of the reduction potential of gas in the upper part of a blast furnace as well as on reducibility of iron – bearing materials. Moreover, the reduction capacity of gas depends, to a large extent, on reactivity and size of coke lumps.

Ferric oxide ($Fe_{1-y}O$) entering the production zone of a blast furnace is reduced there to Fe. It can be reduced both by means of CO and, in two stages, with production of CO in the Boudouard – reaction, by means of coke carbon. As the blast furnace charge progresses in the direction of increasing temperatures, the direct reduction becomes more significant [7]. The model calculations for the blast furnace operation showed that at the temperatures of 1000 °C up to 1400 °C about 40 ÷ 45 % of $Fe_{1-y}O$ is reduced by way of direct reduction [8].

The direct reduction of iron oxides depends on the occurrence of the reaction: $CO_2 + C = 2CO$. The course of this reaction in a blast furnace is, however, different from the clearly established thermodynamic relationships concerning the state of equilibrium. In the area situated above the tuyere zone of a blast furnace the carbon dioxide can appear only as a result of the reduction of iron oxides by CO, and, therefore, its concentration in gas will depend on the rate of reaction (1). The rate of reaction (2) is determined by: CO_2 – concentration, gas – solid heat exchange, temperature, pressure and rate of gas flow in the blast furnace. It is assumed that the required thermodynamic and kinetic conditions and, consequently, the zone of direct reduction, occur at the temperature exceeding 900 °C.

The rate of the reaction (2) increases with the temperature growth, reaching reasonable values at temperatures exceeding 1000°C [9]. The rate of the reaction: $CO_2 + C = 2\ CO$ in a blast furnace depends, however, not only on temperature but also on the possibility of contact of coke with the streaming gas [10]. The contact occurs fairly easily in the solid material area. Difficulties arise in those areas of the blast furnace in which there occurs the liquid phase, i.e. in the cohesion and sub-cohesion zones. In the state of softening -melting of the sinter the rate of direct reduction decreases. It is assumed that the distribution of the rate of direct reduction is similar to the normal distribution with the rate maximum at the temperature of the upper limit of the cohesion zone. This temperature depends, among other things, on the temperature of softening of the materials used in the blast furnace process. Data quoted in the literature make it possible to conclude that for sinters this temperature is equal to about 1225–1250°C [11]. After the sinter conversion into the liquid phase, the direct reduction rate can grow again as a result of, among others, an increased area of contact with coke [12].

An evaluation of coke reactivity can, therefore, be a hint concerning the possibility of coke gasification and the amount of coke being used for the purposes of direct reduction. The conditions of the laboratory examinations of coke reactivity are, however, significantly different from those existing in a blast furnace. This concerns, in particular, the CO_2 – content in the gas reacting with coke as well as the temperature of laboratory examination. Some works, taking into account a smaller concentration of CO_2 in gas indicate that the role of coke reactivity in the blast furnace process is less significant than that of its mechanical properties [13]. For a complete evaluation of coke suitability, the laboratory examinations of coke reactivity should be carried out under conditions as closely corresponding to the ones in the blast furnace as possible. This concerns, in the first place, the CO_2 – concentration in gas, temperature, pressure as well as the state and the type of packing of the raw materials.

2. Subject and scope of the examinations

The presented work aimed at investigating sinter reduction under simulated conditions of a blast furnace with the participation of cokes with different reactivity within the temperature range of 950 ÷ 1100°C. Another objective of the investigations was to determine whether it was possible for CO_2, formed *in situ* as a result of the sinter reduction by CO, to react with the coke carbon, i.e. whether it was possible for the direct sinter reduction to occur. Moreover, the influence of the reactivity of coke used for the examinations on the course of the sinter reduction was analysed. The influence of the type of packing of the reduced materials and coke on the course of the reduction was also examined.

In the laboratory examinations simulating the conditions of the blast furnace process pains were taken to preserve the structure of the charge – i.e. similarities relating to the mass proportions of coke to sinter, granulation and packing of sinter and coke as well as the flow rate of the gaseous reducer and its composition. The examinations were limited to the sinter as an iron – bearing material and the carbon monoxide as the reducing agent.

Two kinds of coke were examined (coke 1 and coke 2) which differed in physico-chemical properties, including reactivity. Reactivity (CRI) and strength after reaction (CSR), determined by the Nippon Steel Co. method, were equal to:

- coke 1: CRI = 27.9 % and CSR = 60.2 %
- coke 2: CRI = 47.2 % and CSR = 40.8 %

Coke grains with the diameters from 0.5 to 0.8 cm were used. The sinter was featured by the basicity of 1.32 and grain size of 0.3 to 0.5 cm. The iron content in the examined sinter is given in Table 1.

TABLE 1

Iron content in sinter

Run	Sinter	$Fe_{tot.}$, %	FeO, %	Fe_2O_3, %	$Fe_{metal.}$, %
1	2	3	4	5	6
0	initial	56.54	7.70	72.07	0.15
2	reducced 957°C, coke 2	56.57	61.40	0.91	8.22
3	reduced 1109°C, coke 2	56.57	62.32	0.76	7.62
4	reduced 1109°C, without coke	56.46	60.93	1.36	8.17
5	reduced 958°C, coke 1	56.79	59.39	2.10	8.81
6	reduced 1104°C, coke 1	56.06	61.27	1.80	7.19
7	reduced 1100°C, coke 1	56.74	63.44	1.50	6.38
9	reduced 1100°C, coke 2	56.42	57.30	0	11.90
10	reduced 1100°C, coke 1	56.52	60.49	1.04	8.74
11	reduced 1100°C, coke 1	56.50	70.30	1.94	0.51

The examinations were carried out in the flow system of the steel reactor in the form of a cylinder with 6.8 cm in diameter and 25.0 cm in height. The mass of the examined materials (coke and sinter) was selected to match the amount and packing of the materials as well as the flow rate of gas in the blast furnace. The weighed portions of about 0.3 kg of sinter and 0.095 kg of coke were used. These amounts correspond to the charge of blast furnaces expressed by the ratio: R/K= 3.2 (C/Fe = 0.50). Two kinds of experiment were carried out. The first one involved the examined materials being placed in the reactor in layers: sinter as the lower layer subjected to the first contact with the gas reducer, and coke as the upper layer situated on the surface of the sinter (runs 2 ÷ 7). The second kind of experiment entailed the sinter previously mixed with coke (runs 9 ÷ 11).

The experiments were carried out under isothermal conditions at the temperatures of about 950 and 1100°C. After heating the charge up to the temperature of the examination under the nitrogen atmosphere, the gaseous reducer, containing 32.4 % of CO and 67.6 % of N_2 was introduced. The flow rate of gas was equal to 5 dm^3/min. The duration of a single run was limited to 60 minutes but in two runs this time was shorter (run 7 – 50 minutes and run 11 – 30 minutes).

For comparison, a measurement of the rate of reduction of the sinter alone was carried out at the temperature of 1109 °C. The remaining experimental conditions were identical to those in experiments involving both sinter and coke.

The course of the reaction of solid materials with the gaseous reducer was determined by analysing the CO_2 and CO – concentration in the outlet gas. It should be pointed out, that the changes in gas volume resulting from the formation of additional amounts of CO (reaction 2) were taken into consideration. The mass of the sinter and of the coke obtained as a result of the experiment were applied as reference values. The content of iron in the form of oxides and Fe in the sinter after reduction was also analysed. The obtained results are shown in Table 1. These values refer to the whole sample of the applied sinter.

3. Sinter reduction

The amount of oxygen removed from the sinter during reduction was determined by the chemical analysis of iron compounds concentration in the sinter prior to and after the reaction. The results of the analysis are given in Table 1. The calculated mass of the oxygen removed from the sinter (ΔO_2) is given in Table 2 (Column 6). This mass of oxygen was recalculated to the CO_2 – volume which would be produced if the reduction occurred only according to reaction (1), i.e. by way of indirect reduction without the participation of carbon (Column 7).

TABLE 2

CO_2 volume emitted during reduction of sinter

Run	Reagent	Temperature °C	Time, min.	O_2 loss from sinter (ΔO_2), chemical analysis, g	CO_2 volume, corresponds to ΔO_2 dm^3	CO_2 volume, gas analysis, dm^3	ΔCO_2, dm^3
1	2	3	4	6	7	8	9
4	sinter	1109	60	32.71	45.79	45.60	0.19
1	coke 2	1100	60	–	–	–	–
2	coke 2 + sinter	957	63	33.42	46.79	46.28	0.51
3	coke 2 + sinter	1109	62	32.34	45.28	25.80	19.48
9	coke 2 + sinter	1100	60	36.80	51.52	30.22	21.30
8	coke 1	1100	60	–	–	–	–
5	coke 1 + sinter	958	63	33.00	46.20	46.07	0.13
6	coke 1 + sinter	1104	60	32.20	45.09	32.67	12.42
7	coke 1 + sinter	1100	50	30.97	43.36	30.74	12.62
10	coke 1 + sinter	1100	60	33.43	46.80	32.69	14.10
11	coke 1 + sinter	1100	30	21.43	33.26	22.07	11.19

The recorded contents of CO_2 (in %) in the gas emitted during the reduction of sinter are represented in Figures 1 and 2. Figure 1 represents the CO_2 – content during the sinter reduction with coke 1 and 2 at the temperatures of about 950°C and about 1100°C. Figure 2 shows changes in CO_2 – content during the reduction of the sinter alone and the reduction of the sinter with coke 1 and 2 at the temperature of about 1100°C.

The total volume of CO_2 released during particular experiments, calculated on the basis of an analysis of the CO_2 – content in the outlet gas, is given in Table 2 (Column 8). Column 9 in this table shows the CO_2 – content (ΔCO_2) which constitutes the result of subtraction of the volume corresponding to the loss of oxygen in the sinter from the volume determined by way of an analysis of the outlet gas.

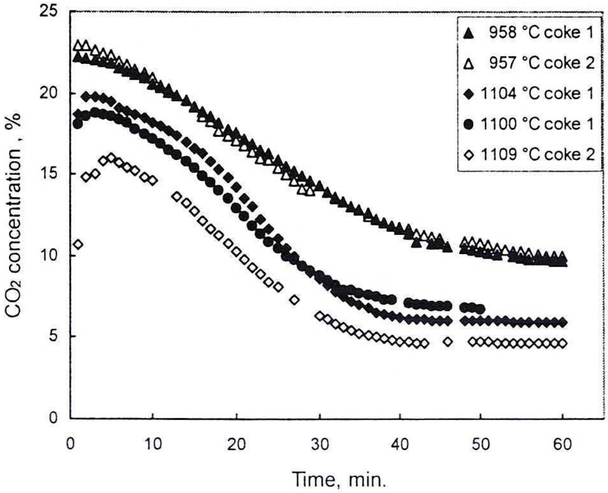

Fig. 1. Dependence of sinter reduction on temperature and coke reactivity

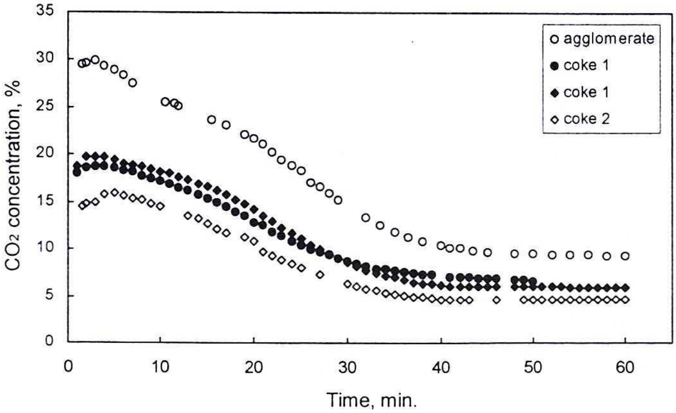

Fig. 2. Dependence of sinter reduction on coke reactivity at temperature 1100°C

3.1 Influence of temperature and coke reactivity on the course of the sinter reduction

The course of the sinter reduction is significantly different for both the examined temperatures. This concerns changes in CO_2 – concentration in time as well as its total volume removed with the outlet gas.

At the temperature of about 1100°C the CO_2 – concentration in gas during reduction is significantly lower. One can also observe different CO_2 – concentration in the outlet gas depending on the kind of coke. During the reduction with the participation of coke 2 the CO_2 – concentration in gas is significantly lower than during the reduction with the participation of coke 1. The difference in CO_2 – concentration ranges from about 5 to about 1.5 % depending on time.

A comparison of the amount of CO_2 released during the reduction and that measured by way of an analysis of the outlet gas makes it possible to state that in the case of the reduction of sinter alone (run 4) and the reduction with the participation of both the examined cokes at the temperature of 950°C there is practically no difference between the volume of CO_2 calculated by analysing the sinter and that measured in the outlet gas. Small differences fall within the limit of the analytical error. One can therefore conclude that at the temperature of 950°C the CO_2, formed as a result of reaction of iron oxides with the gaseous reducer, does not react with coke carbon. The entire volume of CO_2, formed as a result of the oxygen being removed during reduction, is removed with the outlet gas. Consequently, the reduction of sinter is, in these conditions, an indirect reduction occurring without the participation of coke carbon.

At the temperature of 950°C the reactivity of both the examined cokes is low. The determined [13] mean value of reactivity index for coke 1 is equal to 0.30 $cm^3/g{\cdot}s$ and for coke 2 about 0.54 $cm^3/g{\cdot}s$. The above mentioned values were determined with the use of the gas containing 100 % of CO_2 at the temperature of 1000°C. A decrease in the CO_2 – content in the gas to about 20 % results in a drop in the reactivity index of coke 1 to 0.13 $cm^3/g{\cdot}s$ and of coke 2 – to 0.21 $cm^3/g{\cdot}s$. In the experiments discussed in the paper the CO_2 – content in gas reaches the value of about 22 % at the initial stage of the reduction and decreases to about 11 % at the final stage. Therefore, the reactivity of the examined cokes will be significantly lower – approximately 0.02 $cm^3/g{\cdot}s$ for coke 1 and 0.05 $cm^3/g{\cdot}s$ for coke 2. Consequently, the rate of the Boudouard-reaction (reaction 2), though possible in terms of thermodynamics, is negligible in the conditions of the experiment.

Figure 2 represents the differences in the course of the reduction of sinter with and without the participation of coke at the temperature of 1100°C. The reduction of sinter without the participation of coke can only occur as an indirect reduction. Consequently, the amount of the emitted CO_2 corresponds to the mass of the oxygen removed from the sinter. During the reduction of sinter with the participation of coke the CO_2 – volume in the outlet gas is significantly smaller than during the reduction of sinter alone. Differences in CO_2 – concentration amount to 10 ÷ 15 % at the beginning of the experiment and 5 ÷ 7 % at the final stage. The lower concentration of CO_2 in gas observed during the reduction with coke can only result from a reaction of a part of the emitted CO_2 with coke carbon. Consequently, in these conditions one should expect a mixed reduction, i.e. indirect and direct reduction.

The CO_2 – volume removed with the outlet gas during the experiment with the participation of coke is significantly smaller than that corresponding to the loss of oxygen in the sinter. This volume is equal to about 33 dm^3 and 26 dm^3 for coke 1 and coke 2 respectively, which constitutes 72 and 57 % of the amount of the CO_2 which would be found in the outlet gas in the case when the indirect reduction was the only one to occur. As a result of the experiment a decrease in the mass of coke was also observed. It was equal to about 7 and 11 % for coke 1 and coke 2 respectively. This decrease corresponds to the stoichiometric amount of carbon which can react with the amount of CO_2 constituting the result of subtraction of the CO_2 released during the reduction from that included in the outlet gas (Δ CO_2 – Table 2). Therefore, it can be concluded that the CO_2 formed as a result of the reaction of CO with the sinter undergoes a consecutive reaction with coke. The occurrence of such a reaction is facilitated by a significant increase in coke reactivity with the growth of temperature [13].

The rate of the reaction of CO_2 with coke carbon depends also on coke reactivity. This reactivity varies depending on the way in which the coke is produced. Coke 1, produced with the use of the dry quenching method, is characterised by lower reactivity than coke 2, produced with the use of the wet quenching method. The effect of coke reactivity on the course of the reduction is reflected in the kinetic curves of CO_2 – concentration in the produced gas. These curves are represented in Figure 2. The CO_2 – concentration in gas is lower by $3 \div 5$ % during the reduction with the participation of coke 2. Markedly different is also the CO_2 – volume which reacts with coke. The volume is equal to about 28 and 43 % of the CO_2 – volume, formed during the reduction of sinter without the participation of coke, for coke 1 and coke 2 respectively. Under the conditions of the experiment one can, therefore, observe a significant influence of the kind of coke on its consumption during reduction. However, the above-mentioned remarks cannot be a criterion determining coke suitability for the blast furnace process. Under real conditions of a blast furnace, at temperatures exceeding 1000°C, ferric oxide is the prevailing oxide. Consequently, it can be conjectured that this oxide will be reduced mainly by way of the direct reduction. The effect of coke reactivity on the course of the sinter reduction will therefore be reflected in its influence on the course of the $Fe_{1-y}O$ – reduction.

In the experiments discussed in this paper the reaction of CO_2 with coke occurs within the entire time range. In these conditions, the reduction of both oxides present in sinter (Fe_2O_3 and $Fe_{1-y}O$) is of the mixed type. The reduction of $Fe_{1-y}O$ to Fe is slower than the reduction of Fe_2O_3 to $Fe_{1-y}O$ and, as a rule, occurs as a consecutive reaction to the reduction reactions of higher iron oxides. The course of the kinetic curves represented in Figure 2 shows a significant decrease in the rate of the process after about 40 minutes. This fact as well as the calculations carried out on the basis of the sinter analysis before and after the reduction suggest that at the final stage of the experiment ($40 \div 60$ min.) there occurs, basically, a reduction of $Fe_{1-y}O$ to Fe. Starting from the Fe-content in the sinter after the reduction (7.04 % and 7.53 % for coke 1 and coke 2 respectively) the amount of CO_2 formed as a result of removing oxygen from $Fe_{1-y}O$ during the reduction to Fe was calculated. The amount was equal to 7.56 dm^3 for coke 1 and 8.09 dm^3 for coke 2. The volumes of CO_2 in the outlet gas, for a similar period of time, were equal to 5.80 and 5.02 dm^3. The remaining amount of CO_2, i.e. about 23 and 38 % of the emitted amount, reacted with coke carbon. It can, therefore, be concluded that the reduction of $Fe_{1-y}O$ at 1100°C is a mixed – type reduction with the indirect reduction prevailing. This conclusion is in accordance with the model calculations concerning the contribution of the direct reduction to the reduction of $Fe_{1-y}O$ in the blast furnace process [8]. In these model calculations the contribution of the direct reduction of $Fe_{1-y}O$ was found to reach the level of 40 ÷ 45%. Higher reactivity of coke results in an increased contribution of the direct reduction to the process. It should, however, be pointed out that a comparison of the above mentioned values ought to be treated as an approximate assessment. It cannot be assumed that the entire amount of reduced iron was formed at the final stage of the reduction process.

3.2 Influence of the type of packing of materials on the reduction

The influence of the type of packing of the sinter and coke inside the reactor on the course of the reduction at 1100°C was analysed by comparing experiments in which the sinter was mixed with coke (runs 9 and 10) with those, in which the sinter and coke were packed in layers (runs 3, 6 and 7).

The CO_2 – concentration in the outlet gas recorded during the reduction with the participation of coke is represented in Figures 3 and 4 for coke 1 and coke 2, respectively. It can be seen that the influence of mixing the sinter with coke 1 on the course of the reduction is rather small. The CO_2 – concentration in the outlet gas is similar to that in experiments in which the sinter and coke were packed in layers. Certain differences occur in the experiment with the participation of coke 2. At the beginning of the process, the CO_2 – concentration in the outlet gas is higher in the case when the coke is mixed with the sinter. This points to the lower rate of the reaction: $CO_2 + C = 2CO$. As the reduction progresses the rate of this reaction increases (a lower concentration of CO_2 in the outlet gas).

An analysis of the values shown in Table 2 enables a more precise definition of the influence of the packing type of the materials in the reactor on the course of the reduction. As a result of mixing coke 1 and the sinter, the extent of sinter reduction grew by 3.7 % (cf. runs 6 and 10). As the amount of CO_2 removed with the outlet gas did not change, an additional amount of CO_2 formed during reduction (1.71 dm^3) fully reacted with coke carbon. Mixing coke 2 with the sinter resulted in an increase in the extent of sinter reduction by 13.8 % (cf. runs 9 and 3). The additional amount of CO_2 (6.24 dm^3), which was formed, reacted with coke only in part (1.82 dm^3). The remaining amount was removed with the outlet gas.

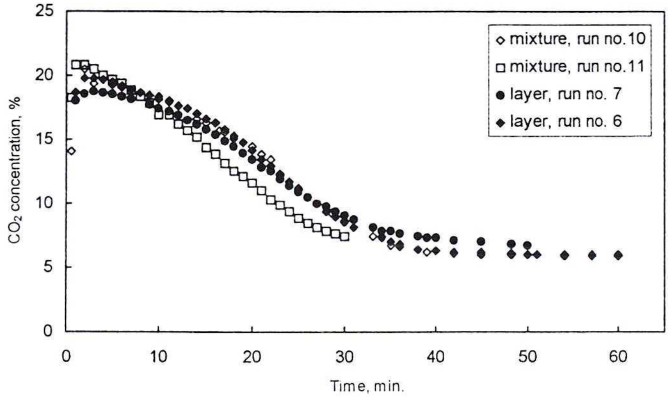

Fig. 3. Influence of the packing type of materials (coke 1 and sinter) on reduction at temperature 1100 °C

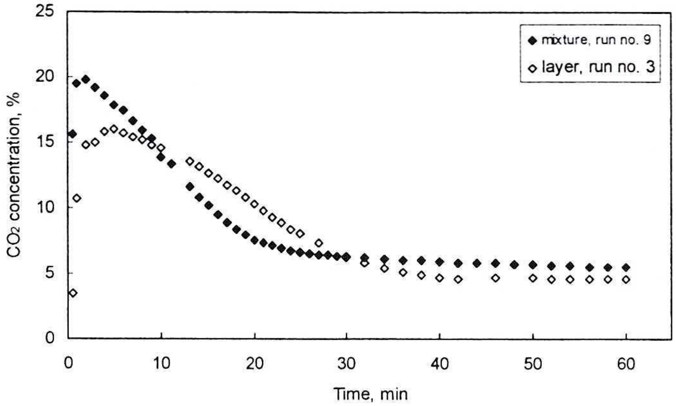

Fig. 4. Influence of the packing type of materials (coke 2 and sinter) on reduction at temperature 1100 °C

The above-mentioned analysis indicates that mixing coke with the sinter influences, in the first place, the progress of the sinter reduction. Lumps of coke, bigger than the sinter grains, cause an increase in the gas – permeability of the blast furnace charge, and, consequently, facilitate access of the gaseous reducer to the reduced material. An increase in the rate of the reduction may be achieved both by way of an indirect and a direct reduction. A small change in the contribution of the direct reduction to the course of the process seems to be independent from coke reactivity.

It should, however, be pointed out that mixing the coke with the sinter can be accidental. Therefore, drawing conclusions concerning its influence in connection with coke reactivity may also be unfounded.

4. Conclusions

1. At the temperature of 950°C the reaction of CO_2, formed as a result of the reduction of iron oxides with coke, does not occur. The reduction of iron-bearing materials occurs by way of the indirect reduction, without the participation of coke carbon. Such a course of the reduction is accounted for by low coke reactivity caused by temperature and low CO_2 – concentration in the gaseous phase.
2. With the growth of temperature up to about 1100 °C, the reaction: $CO_2 + C = 2CO$ becomes an important process influencing the course of the reduction and coke consumption for the direct reduction.
3. There can be observed an increase in coke consumption for the purposes of the reduction with the growth of its reactivity. However, the final extent of sinter reduction and the amount of resulting Fe seem to depend on coke reactivity only to a small degree.
4. The mixing of the sinter with coke increases the rate of the sinter reduction. A change in the contribution of the direct reduction to the process seems to be independent from coke reactivity.

5. The amount of reduced iron (about 8%), formed as a result of the reduction at both the examined temperatures, seems to be independent from the type of the reduction process. It can be, however, connected with the formation of an iron layer of different structure.

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