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Complex solid-phase reduction in a blast furnace of self-healing pellets of cold agglomeration containing by-product carbon materials of metallurgical production

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Комплексне твердофазне відновлення в доменній печі самовідновлювальних окатишів холодної агломерації, що містять побічні вуглецеві матеріали металургійного виробництва

Abstract. *The reactions of direct and indirect reduction occurring during the heat treatment of self reducing pellets (SRP) have been studied. In this investigation Blast furnace (BF) sludge which contains particles of coke, has been included in the SRP blend as a source of solid reductant. In the SRP as a part ot the blast furnace burden occur the reactions simultaneously: inside of SRP-direct reduction by* C_{solid}*; gasification of carbon and indirect reduction by CO; and outside of SRP-indirect reduction of iron bearing oxides by reducing gas coming from the hearth of blast furnace through the column of charged materials. The experiments was performed continuously from the start temperature (~200 ˚C) to the experimental temperature (500 ˚C; 700 ˚C; 900 ˚C; 1100 ˚C) in argon free environment. Upon reaching the desired temperature argon was replaced by hydrogen during 30 minutes. After that the reduced probe of SRP was cooled in argon. The objective of the present work is to research a quantitate ratio of degree direct reduction inside of SRP and degree of indirect reduction outside of SRP on the top of the blast furnace.*

Key words: *self reducing pellets; direct and indirect reduction degree; degree of metallization.*

Анотація. *Процеси доменної печі відрізняється від процесів виробництва заліза прямим відновленням. У твердих і самовідновлювальних брикетах (СВР) у складі шихти доменної печі одночасно відбуваються реакції: всередині СВР – пряме відновлення вуглецем (твердим); газифікація вуглецю та непряме відновлення монооксидом вуглецю, а зовні СВР – непряме відновлення відновлювальним газом, що надходить з поду доменної печі. Інтегральний ступінь відновлення СВР, а саме пряме відновлення твердим вуглецем, що міститься в СВР, та непряме відновлення відновлювальним газом – воднем зовні СВР, досліджувався при температурах 500; 700; 900 та 1100°С.. Були оцінені ступені відновлення СВР твердим вуглецем (всередині зразка) в атмосфері аргону воднем (зовнішня поверхня зразка) за допомогою експерименту з відновлення. Отримано наступні результати. Відновлення F2O3 до Fe3O4 відбувається прямим відновленням (ступінь прямого відновлення становить 46,06%) та непрямим відновленням воднем (ступінь відновлення 53,94%) за температури 500 °С. Вміст кисню в F2O3 для переходу на Fe3O4 дорівнює 15,8%, що відповідає інтегральному ступеню відновлення – 16,5%. Аналогічно, інтегрований ступінь відновлення при прямій температурі 700 °С дорівнює 97,1%, що включає 34,9% за прямої температури 700 °С; та 100%, що включає 48,7% за прямої та 51,3% за непрямої відновлення за температури 900 °С. Інтегрований ступінь відновлення дорівнює 100%, що включає 98,6% за прямої відновлення твердим вуглецем за температури 1100 °С. Хімічний аналіз відновленого полімерного волокнистого матеріалу (SRP) показав зміну ступеня інтегрального відновлення з 85,79 % (900 °С) до 92,50 % (1000 °С) та 84,6 % (1100 °С) та металізації 83,30 % (900 °С), 89,90 % (1000 °С), 80,75 % (1100 °С). Ці дані відповідають результатам залежності ступеня відновлення СВР від температури. Відновлений зразок містить металеву фазу, що утворює найтонші дендрити у склоподібній масі шлакоутворюючих компонентів. Таке перетворення в СВР забезпечує значне підвищення міцності завдяки утворенню металевого каркаса.*

Ключові слова: *самовідновлювальні брикети, температура, вуглець, ступінь відновлення, металевий каркас, міцність.*

Introduction

Metallurgical coke, produced from coking coal, is the primary fuel used in blast furnaces for iron smelting. The cost of coke plays a decisive role in the overall cost of iron production. Furthermore, for use in blast furnaces, coke must meet certain quality criteria, including chemical composition, particle size distribution, mechanical strength, and metallurgical properties. Among these properties, fuel reactivity is particularly important, as it directly affects the temperature of the blast furnace's thermal reserve zone, thereby influencing its operational efficiency [1-3].

To improve the efficiency of the iron smelting process, self-reducing agglomerates and pellets are widely used in the iron charge. These materials offer a number of advantages, primarily due to the close contact between the iron oxide and the reducing agent, which increases the reduction rate and reduces fuel

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consumption [4-5]. Self-reducing materials allow the use of ultrafine iron oxide and steelmaking by-products such as sludge and powders with high iron content, further enhancing the sustainability of this industrial sector [6]. Furthermore, the use of highly reactive carbonaceous materials leads to a decrease in the temperature of the thermal reserve zone, thereby potentially lowering the FeO-Fe equilibrium temperature and reducing overall fuel consumption [7 - 9].

Numerous studies [10–21] have addressed the development and performance characteristics of self-reducing agglomerates, pellets, and developed models to predict the reduction kinetics using different carbon sources in agglomerates, pellets, and briquettes [22–28]. For use in blast furnaces, the self-reducing agglomerate must meet stringent quality requirements in terms of reactivity (or reducibility) and mechanical properties, both of which depend on the carbon source and its amount in the mixture [29, 30]. Furthermore, the volatiles of the reducing agent also have a reducing potential, as shown in previous studies [31–36], which may influence the formation of the agglomerate microstructure.

In recent years, to optimize the reducing process in the blast furnace a great attention is paid to self reducing pellets (SRP). These materials are used already in the operating blast furnace 1-5).The consumption of SRP from 60 to 80 kg/t_{NM} was used in the commercial blast furnace melt. It provides the coke rate lowering by 10-15 kg/t_{HM}· and the degree of direct reduction falls by 2% 2). The consumption of SRP could be come to 200 kg/t_{HM} in the blast furnace charge 3,4).

Results of the blast furnace operation indicated that the SRP charged into the blast furnace does not reduce the gas permeability of the charge and does not disturb the smooth run of the blast furnace. Reduction of the iron oxides contained in the SRP starts at low temperature zone and reduction to come to an end earlier as the sinter and pellets. Thus there is a tendency to a significant reduction in coke consumption.5)

The mechanism and kinetics of self reduction pellets has already been modelled. These models take into consideration not only the kinetics of gasification of carbon and reduction reactions but also the mass and heat transfer phenomena 6,7).

The simultaneous reaction between reduction of the iron oxides and gasification of carbon was examined. The obtained results are as follows: coupling phenomen between reduction and gasification existed. The starting temperature of reduction was 250 ˚C in the hematite graphite facing pair while the temperature was 420 ˚C in the single hematite.8)

It was calculated reduction degree at different temperatures during thermal analysis of SRP samples. Iron oxide reduction seems to start in relatively low temperature range between 500-600 ˚C. A possible explanation is that some carbon gasification catalyzed by H_2O from dihydroxylation of hydrates 9,10). The reduction mechanism of pellets with reducing gas can be transferred to the SRP 11,12).

The contribution ratios of direct reduction by solid carbon and indirect reduction and carbon gasification were estimated through reduction experiment of the composite under inert atmosphere. The reduction from Fe_2O_3 to Fe_3O_4 proceeds at low temperature ravel very small. During this period direct reduction proceeds because new contact points between Fe_2O_3 and graphite are formed. The contribution ratio of the direct reduction as approximately 45% during the reduction from Fe_2O_3 to Fe_3O_4–13). It carbonaceous material and iron bearing oxides could be adjoin, the starting temperature of the reaction could be lowered. With the increase in the degree of contact in such mixture the starting temperature is lowered. It is an effective method to increase the rate of direct reduction 14).

The blast furnace process differ from the processes production of iron by direct reduction. In the SRP as a part of the blast furnace burden simultaneously reactions occur: inside of the SRP –direct reduction by carbon (solid); gasification of carbon and indirect reduction by mono oxide of carbon and outside of SRP – indirect reduction by reducing gas coming from hearth of the blast furnace.

A significant number of research of a reduction processes carried out in the carbon composite agglomerates in an inert atmosphere (nitrogen, argon). From the point of a view of the use in the blast furnace process SRP it is necessary to take into account indirect reduction of iron bearing oxides by reducing gas coming from the hearth of the through the column of charge materials of the blast furnace.

The purpose of this paper is to estimate the ratio of indirect and direct reduction SRP depending on the temperature in the range of 500-1100 ˚C.

Experimental Procedure.

Sample

SRP sample was selected from industrial parties. The chemical composition and physical characteristics of the sample are given below: SRP were produced from the mixture of a blast furnace and converter sludge in proportion (3: 2), with the addition 10 % of the portland cement.

Chemical composition of SRP. %

Fe_{tot}	FeO	Fe_2O_3	SiO_2	CaO	MgO	C
43,10	8,0	52,68	7,50	14,0	8,0	9,8

Fractional size of SRP.

Size, mm	40-20	20-15	15-10	10-5	5-0
Yield, %	37,00	45,00	12,00	1,8	4,5

The SRP have been produced on the pelletizer 5,5 m. with productivity 25-30 ton per hour. After an probe exposure during 28 day the compressive strength of SRP was in average 90 kg/pellet.

The bulk weight g/cm^3-1,4.

Reaction behavior of the SRP involving with reduction of iron bearing oxide has been investigated Diameter of SRP ranked between 10-15 mm was set in reaction crucible and than in reaction tube.

Research facility

The experimental setup is shown in Fig. 1. It consists of a electrical heating furnace, which can be moved up and down. The quartz tube passes through the furnace. The reaction zone is in the middle of the furnace. Neutral argon atmosphere is created and for indirect reduction argon was changed on hydrogen. Gases of argon and hydrogen are introduced into the furnace separately. Wire of nickel alloy chromosome joins the scales test. A thermocouple is located in the tube.The crucible of wire chrome-nickel was permeable.

The reduced sample of SRP was to cool in Ar gas until room temperature was reached.

Experimental Procedure reduction of SRP by Hydrogen. Thermogravimetric method was used to measure the degree of a reduction depends on the temperature. The SRP fraction 10 - 15 mm was used. The furnace temperature was varied in the range 500-1100 ˚ C. The heating rate of the sample was varied in the range of 27.7 - 29.5 ˚ C/min.

In this investigation chemical analysis of reduced samples after thermal treatment was feasible. Calculation of reduction and metallization degree was done using results from chemical analysis and the change in mass due to oxygen removal from iron oxides of SRP separately due to direct reduction and indirect reduction. Reduction degree has been defined as follows.

$$RD\ (\%) = \left(\frac{\Delta m_{red}}{m_{initial}}\right) \times 100\%$$

Where RD – is reduction degree, %

Δm - is the change in mass due to oxygen removal from iron oxide;

$M_{initial}$ – initial mass of sample.

The SRP samples from heated to temperature: 1000 ˚C were studied using the methods of manual microscopy and petrography of ore using a notarizing microscopy the MIN – 9 microfotometric device PME – 1. An ore, slag and carbonaceous components were determined by reflectance, colour, polarization effect and internal reflex.

Results and discussion

Results of the SRP reduction process study shown in table 1 and figure 2. The experiments were performed continuously by from the start temperature (~200˚C). The sample was placed in an furnace and heated to the experimental temperature under argon, and then replacement by hydrogen reducing gas, at this temperature for 30 min. This cycle is to be repeated for the temperatures: 500 ˚C;700 ˚C; 900 ˚C; 1100 ˚C.

Fig 2 shows the dependence the reduction degree of the iron oxides of SRP on the temperature. The direct reduction degree of the SRP is 7,6%, at the temperature 500˚C.

Fig 2 shows the change in the indirect reduction degree of iron oxides of SRP by reducing gas-hydrogen on the outside surface of SRP. The degree of indirect reduction is 8,9 % at the temperature 500˚C.

Integrated degree of direct and indirect reduction is equal 16,5 %. Initial probe of SRP contains 43,1 % Fe_{tot}; and 8 % FeO that it is corresponded to content of 34, 88 % Fe_3O_4. Thus Fe_2O_3 could be reduced to Fe_3O_4. The following calculation let us to understand that integrated degree of reduction 16,5 % corresponds approximately to reduction of Fe_2O_3 to Fe_3O_4. It occurs due to reaction $3Fe_2O_3= 2\ Fe_3O_4+ □_2O_2$. The content of oxygen removed from Fe_2O_3 to transfer for Fe_3O_4 could be : $\frac{52,68\times144}{480} = 15,8\%$ that correspond approximately integrated degree of reduction (16,5 %).

Where: 52,68 – content of Fe_2O_3 in initial sample of SRP

144 – content of oxygen in 3 Fe_2O_3 according to reaction

480 – molecular mass of Fe_2O_3 according to reaction.

As to direct reduction of Fe_2O_3 to Fe_3O_4 by solid carbon which proceeds at temperature 200 – 500 ˚C, the gasification rate is very small. During this period direct reduction prossesd due to contact between Fe_2O_3 and particle of carbon of which are formed continuously due to expansion of iron oxides. The contribution part of the direct reduction is 46,08 % during the reduction from Fe_2O_3 to Fe_3O_4 {7,6 : 16,5}= 46,06 %. It is conform to result turn out earlier.[13)]

The degree of the iron oxides SRP reduction by hydrogen shows 8,9 % at temperature 500 ˚C. The reduction mechanism of burnt pellets by reducing gas could be transferred to the reduction of SRP. The degree of direct reduction by carbon and indirect reduction of iron oxides of SRP are equal 34,9 % and 62,2, % correspondingly at the temperature 700 ˚C. After reduction $Fe_2O_3 \rightarrow Fe_3O_4$ the total contents of magnetite could be 178,6 g/mol and oxygen (O_2) – 49,2 g/mol.

Table 1. Results of the integrated reduction of self-reducing pellets.

Temperature, oC	Summary duration of experiment, min	Summary loss of mass, g	Reduction by carbon in argon athmosphere Direct reduction			For time of reduction of hydrogen Indirect reduction			
			Duration, min	loss of mass	Degree of reduction	Duration, min	Degree of reduction loss mass	Degree of reduction	∑
500	48,0	0,76	18,0	0,35	7,6	30	0,41	8,9	16,5
700	55,0	3,44	25,0	1,61	34,9	30	2,87	62,2	97,1
900	61,5	4,93	31,5	2,40	48,7	30,1	2,53	51,3	100,0
1100	62,0	4,61	56,0	4,55	98,6	6	0,064	1,4	100,0

The integrated degree of the SRP reduction at the temperature is equal 97,1%. The contribution part of the direct reduction is 35,94 % at the temperature 700 °C and 48,7 at the temperature 900 °C and 98,6 at he temperature 1100 °C.

The quantity of oxygen could be removed according to indirect and direct reaction as follows:

$Fe_3O_4+H_2=3FeO+H_2O$

$Fe_3O_4+C=3FeO+CO$

The quantity of oxygen removed by hydrogen (49,2 * 62,2 %): 100= 30,60 g

And by carbon (49,2* 34,9):100 = 17,17g , where 62,2 % and 34,9 % degree of indirect and direct reduction of SRP at the temperature of 700 °C correspondingly.

By the same wat it is possible to calculate quantity of oxygen removed out of FeO to Fe.

$3FeO+H_2=3Fe+H_2O$

$3FeO+ C=3Fe+CO$

($\sum O_2$=35,46 g.). Degree of reduction is equal

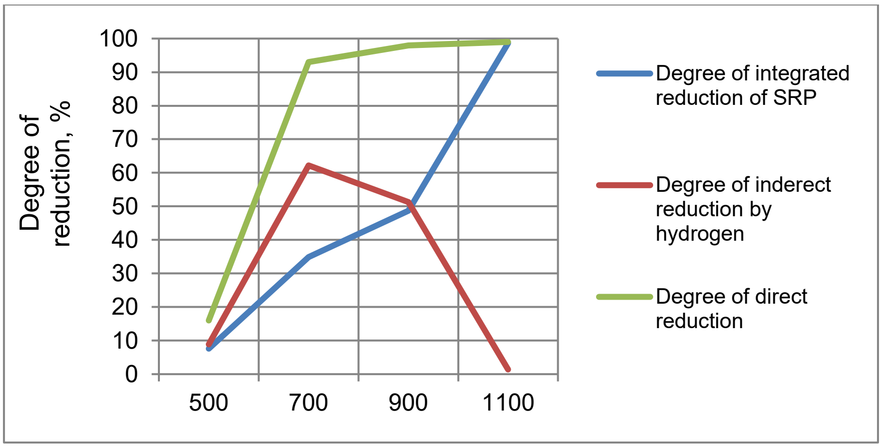

Fig.2 Depends of degrees reduction o on the temperature the SRP

Table 2. Chemical analysis of reduced SRP

Temperature, °C	Content of the component,%					Content of oxide oxygen,%	Degree of reduction, %	Degree of metalization, %
	Fe_{total}, %	FeO	Fe_2O_3	Fe_{metall}	Carbon-residual			
1100 °C	60,00	11,80	2,05	48,45	0,67	0,10	84,60	80,75
1000 °C	59,25	3,95	5,30	53,30	0,85	0,19	92,50	89,90
900 °C	62,20	7,85	4,70	51,85	0,63	0,35	85,70	83,30

Fig 2 shows that indirect degree of the SRP reduction is higher than direct reduction up to 900˚C when indirect and direct reduction of SRP are equal approximately. When the temperature is higher then 900˚C the direct reduction degree of iron bearing oxides of SRP increase from 53,1 % to 100 % at the temperature 1100˚ it occurs due to reaction of gasification of carbon inside of the SRP ($C+CO_2=2CO$). Chemical analysis of reduced samples of SRP showed the higher degree of reduction and metallization in range of temperature from 900˚C to 1100˚C.

This paper presents the results of petrographic studies of the microstructure SRP obtained from a mixture of blast furnace and converter sludge production.

Three samples SRP studied: the initial and two reduced one. Sample obtained by heating in argon at 1000 ˚C undelayed and the second sample is also heated in argon to 1000 ˚C and kept at this temperature - 150 minutes. ˚C

The original sample was diagnosed with iron oxides: hematite (Fe_2O_3), magnetite (Fe_3O_4), coke, slag-forming components differ in shape, size and composition. The detrital material: hematite, magnetite, coke submicroscopic particles (Fig. 2) cemented cryptocrystalline material.

Recycled sample (undelayed) differs in composition from the starting components increased amount of the metal phase. Metallic phase forming the finest dendrites in the glassy mass of slag-forming components. The metal is also dispersed in the form of units in unit microns in size, which are concentrated on the surface of the coke fragments and slag-forming components (Figure 3).

The reduced sample (delayed) at 1000 ˚C over a third metallized. Dendrites large metal. The rest obschschee iron represented magnetite (Fe_3O_4) (Figure 4). In the structure of the pellet is not coke.

Marked transformation in SRP provides a significant increase in strength due to formation of a metal frame. The strength of the reduced pellets (degree of reduction 75 - 94,2 % and content of Fe_{met} 42,5 – 55,1%; compressive strength increases to 42,7 kg/s).

Conclusions

The blast furnace process differs from the processes production of iron by direct reduction. In the SRP as a part of the blast furnace burden occur simultaneously reactions: inside of the SRP – direct reduction by carbon (solid);gasification of carbon and indirect reduction by mono oxide of carbon and outside of SRP- indirect reduction by reducing gas coming from hearth of the blast furnace.

The integrated degree of SRP reduction namely there was direct reduction by solid carbon which contains in the SRP and indirect reduction by reducing gas – hydrogen outside of SRP was investigated at the temperatures 500; 700; 900 and 1100°C. SRP by solid carbon direct reduction and by reducing gas (outside surface of SRP) through experiment. There were estimated reduction degrees of SRP by solid carbon (inside sample) under argon atmosphere by hydrogen (outside surface of sample) through reduction experiment. The following results are obtained.

The reduction F_2O_3 to Fe_3O_4 proceeds by direct reduction (degree of direct reduction is 46, 06 %) and indirect reduction by hydrogen (degree of reduction 53,94 %) under temperature 500 °C. The content of oxygen in F_2O_3 to transfer for Fe_3O_4 is equal 15,8% that is corresponds to integrated degree of reduction – 16,5 %. The same way integrated degree of SRP is equal 97,1 % which includes 34,9 % by direct temperature 700 °C; and 100 % which includes 48,7 % by direct reduction and 51,3 % indirect reduction under temperatures 900 °C. The integrated degree of reduction is equal 100 %, which includes 98,6 % direct reduction by solid carbon under temperatures 1100 °C.

The chemical analysis of the reduced SRP showed the degree of integrated reduction change from 85,79 % (900 °C) to 92,50 % (1000 °C) and 84,6 % (1100 °C) and metallization 83,30 % (900 °C), 89,90 % (1000 °C), 80,75 % (1100 °C).These data correspond to results of degree of reduction SRP depends on temperature.

This paper presente the results of petrographic studies of SRP after heating of sample in argon under 1000 °C. Recycled sample contains metallic phase forming the finest dendrites in glassy mass of slog forming components. Markes transformation in SRP provides a significant increase in strength due to formation of metal frame.

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