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Metallurgy



Study of the isothermal kinetics of reduction of sinter from mill scale

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ABSTRACT

Studies of the isothermal kinetics of the reduction of sinter from mill scale were carried out at Zh. Abisheva Chemical and Metallurgical Institute (Karaganda). The influence of temperature and heating time on the degree of reduction of iron and other elements was studied in the Tamman furnace by the thermogravimetric method. The amount of reducing agent was taken in the experiments based on the stoichiometrically necessary amount of carbon for the complete reduction of iron oxides by more than 60%, as well as for the carburization of the resulting metal to 2.0% C. To obtain the kinetic characteristics of the reduction of the studied materials, depending on the temperature and duration of the process, processing of the received data array according to the models developed especially for these cases. The results of experimental data processing according to the Ginstling-Brownstein model are presented. As a general assessment, this model with a sufficiently high correlation (R not less than 0.9) describes the studied process, which confirms its diffusion character. Analyzing the calculated data of the activation energy, one can assume the diffusion nature of the inhibition of the reduction reaction of all the materials under study. The value of the activation energy of the process shows that the agglomerate with dolomite (fluxed agglomerate) has the highest reducibility. During the frontal reduction of oxides of iron and other elements with carbon, a slag shell is formed around the grains of materials, which consists mainly of acidic oxides of barren rock, non-metallic inclusions of lightweight scrap and reducing agent ash and has a high viscosity. Its thickness depends on the composition of the material, temperature, time and degree of reduction.

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Introduction

Rolling scale is an oxidized product that is formed in the process of metal reduction in rolling mills in large quantities. From the total steel production, scale formation is 2% [1]. According to [2], about 13 million tons of mill scale are formed annually around the world. In world practice, much attention is paid to the recycling and use of industrial waste in the metallurgical process [3], [4], [5], [6]]. Rolling scale and other wastes generated at metallurgical plants are involved in production with

their use as additional iron-containing material [[7], [8], [9], [10]] in the form of briquettes and sinter [[11], [12], [13]].

Technical and economic indicators of the technology of smelting iron and ferroalloys [14] depend on the recovery rate of the leading element (Fe, Si, Mn, Cr and other elements) and the nature of the slag formation process. These factors are affected by the process temperature, the chemical composition of the materials used and their softening point, the electrical resistivity of the charge, the reducibility of the materials, and the

Table 1 – Chemical composition of materials

| Material | Chemical composition, % | | | | | | | | |
|---------------------------|-------------------------|------|--------------------------------|------|------|------------------|------|---|-------|
| | Fe _{gen} | FeO | Al ₂ O ₃ | CaO | MgO | SiO ₂ | S | C | P |
| Agglomerate | 38.0 | 24.9 | 15.8 | 4.96 | 4.86 | 11.3 | 0.05 | - | 0.005 |
| Agglomerate with dolomite | 55.0 | 12.1 | 1.46 | 7.06 | 6.80 | 5.10 | 0.07 | - | 0.001 |

types of reducing agents used. In [15], the kinetic reduction briquettes obtained from mill scale is carried out with hydrogen. As a result, it is shown that the recovery process is controlled by the process diffusion, and the activation energy ranges from 61.5 to 72.25 kJ/mol. In other studies [16], the kinetics and mechanism of reduction of agglomerates are studied using traditional types of reducing agents.

In this research, the authors obtained agglomerates from mill scale mixed with various metallurgical wastes, and studied the isothermal kinetics of agglomerate reduction. The obtained experimental data will help to better understand the mechanisms of carbothermal reduction of the sinter and the improvement of metallurgical processes.

Experimental part

When conducting research, lightweight steel scrap was used as a charge in a mixture with agglomerated ordinary iron ore and sinter with dolomite additives (up to 10% dolomite in sinter), produced in large-scale laboratory conditions of the Zh. Abisheva Chemical and Metallurgical Institute (Karaganda).

The studies were carried out on a charge, the ore part of which is represented by a mixture of lumpy iron ore (70%) and fine iron ore or agglomerates from mill scale (30%). PRC metallurgical coke (11.2% Ac, 4.20% Vg, 3.01% W) was used as a reducing agent. The composition of raw materials and coke ash is shown in Table 1.

The effect of temperature and heating time on the degree of reduction of iron and other elements was studied in the Tamman furnace by the thermogravimetric method. The method is fairly accurate and simple, does not require complex devices, and is acceptable for processes occurring at high temperatures. It is based on the assumption that during the reduction of charge oxides with carbon, only carbon monoxide is released in any period of time. This is only an assumption, because the charge material (ore) always contains moisture and carbon dioxide, which, at high temperatures, are added to the released gas. In the chosen

method, these precipitates are also taken into account by the weighing device, and we get a general picture of the apparent activation energy. To eliminate the influence of the particle size distribution of materials and the reducing agent on the results of the study of reducibility, all charge materials were pre-crushed to a fraction of 0–1 mm.

The amount of reducing agent in the experiments was taken based on the stoichiometrically necessary amount of carbon for the complete reduction of iron oxides by more than 60%, as well as for the carburization of the resulting metal to 2.0% C. For each experiment, samples were taken consisting of 75 g of the ore mixture and the corresponding amount coke. The weighed mixture was placed in a graphite crucible and placed in a furnace heated to a given temperature, suspended from an analytical balance with a molybdenum wire. The melting unit in the installation was a Tamman furnace with a graphite heater. The experiments were carried out in isothermal mode at temperatures of 1100; 1200; 1300; 1400; 1500 °C for 1 hour. The temperature was measured with a VR-5/20 thermocouple. The input power was regulated smoothly, so that the temperature could be maintained in the desired range. The temperature deviation from the set value did not exceed ±5 degrees. The crucible was cooled in air. The degree of recovery was judged by the weight loss of the sample. From the practice of studying the reducibility of iron ore materials, it is known [17] that the degree of reduction is determined by the ratio of the mass of oxygen taken away during the reduction to the mass of the initial oxygen bound in the material sample with the reduced iron. The degree of recovery was determined by the formula:

$$\alpha = \frac{O_2^B}{O_2^H}, \quad (1)$$

where O_2^H and O_2^B - the mass of oxygen in the original and reduced (exposed to the reducing agent for a certain period of time) samples of iron ore material.

For calculation α according to the experimental data, we used the following formula:

$$\alpha = \frac{O_2^B}{O_2^H} = \frac{\Delta m \cdot 16/28}{O_2^H}, \quad (2)$$

where Δm - weight loss, gr.

Due to the fact that iron-bound oxygen is not determined by direct chemical analysis, the value O_2^H It is usually calculated using calculation formulas in which, instead of oxygen, there are quantities that are more convenient for direct experimental determination, for example, those obtained by chemical analysis of the content of iron oxides and other elements. Assuming that in the original ore material under study and in the coke ash, oxygen is bound to iron in ratios determined by the stoichiometry of pure oxides, it is possible to

calculate the mass of oxygen in the iron ore material and coke.

Research results and discussions

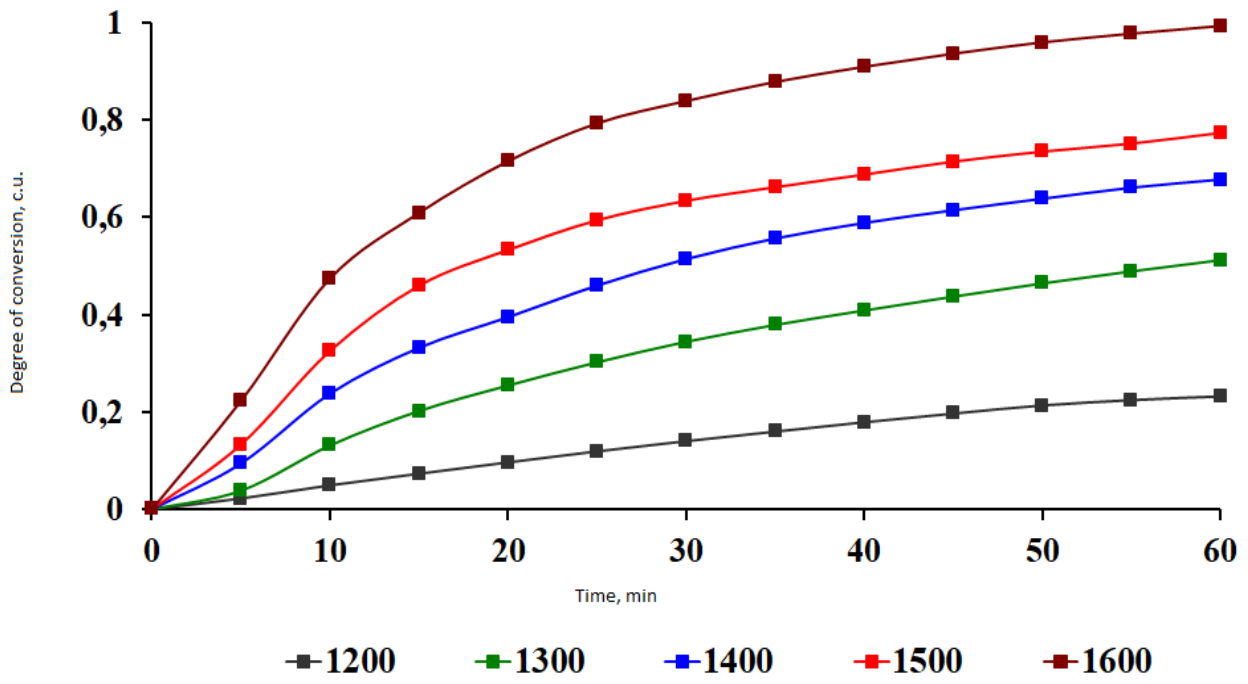
Table 2 gives the entire material of the experiment on the study of the reducibility of various materials under study by coke carbon.

To obtain the kinetic characteristics of the reduction of the studied materials, depending on the temperature and duration of the process, the obtained data array was processed according to specially developed models for these cases.

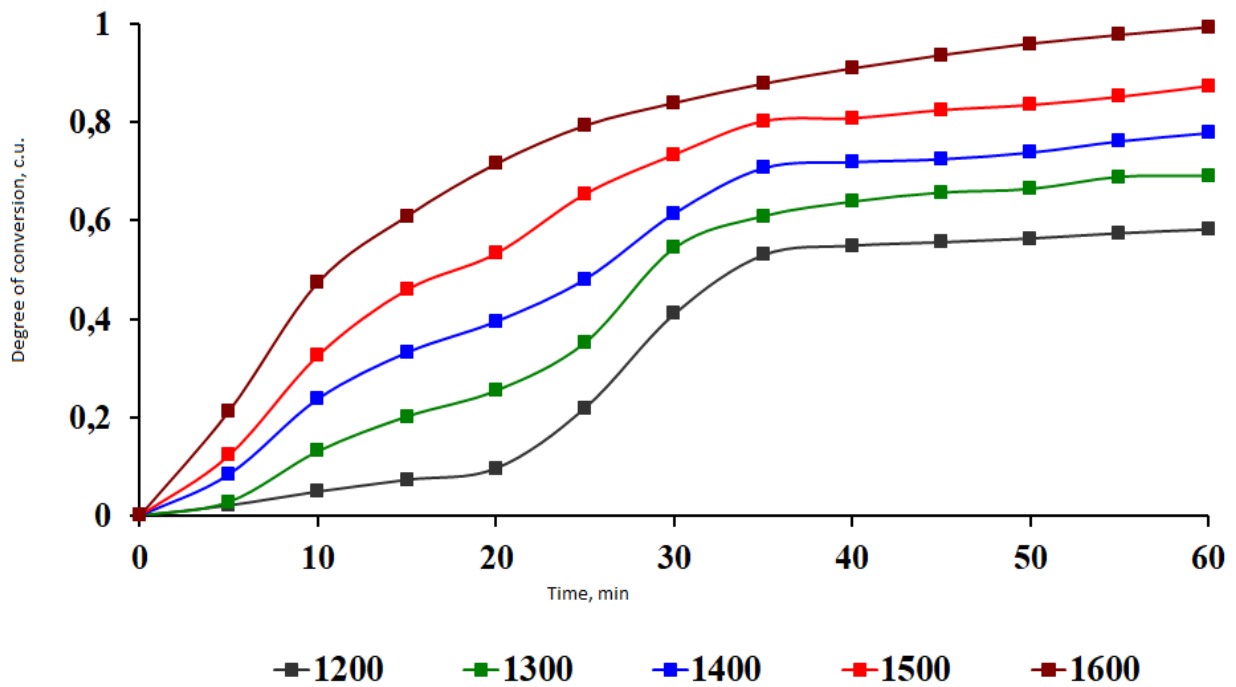
Figure 1 shows the kinetic curves for the reduction of various mixtures of light-weight scrap charge with conventional sinter and fluxed sinter under various temperature conditions.

Table 2 – The degree of recovery of materials

| t, °C | Degree of conversion (α) at time (τ , min) | | | | | |
|-------------------------------------|---|-------|-------|-------|-------|-------|
| | 5 | 10 | 15 | 20 | 25 | 30 |
| Lightweight scrap + sinter | | | | | | |
| 1100 | 0.022 | 0.049 | 0.073 | 0.097 | 0.119 | 0.140 |
| 1200 | 0.038 | 0.131 | 0.202 | 0.254 | 0.302 | 0.344 |
| 1300 | 0.094 | 0.238 | 0.332 | 0.395 | 0.460 | 0.514 |
| 1400 | 0.133 | 0.326 | 0.460 | 0.533 | 0.594 | 0.634 |
| 1500 | 0.223 | 0.474 | 0.609 | 0.715 | 0.793 | 0.839 |
| Lightweight scrap and fluxed sinter | | | | | | |
| 1100 | 0.021 | 0.049 | 0.073 | 0.097 | 0.219 | 0.410 |
| 1200 | 0.028 | 0.131 | 0.202 | 0.254 | 0.352 | 0.544 |
| 1300 | 0.084 | 0.238 | 0.332 | 0.395 | 0.481 | 0.614 |
| 1400 | 0.123 | 0.326 | 0.460 | 0.533 | 0.654 | 0.734 |
| 1500 | 0.213 | 0.474 | 0.609 | 0.715 | 0.793 | 0.839 |
| t, °C | Degree of conversion (α) at time (τ , min) | | | | | |
| | 35 | 40 | 45 | 50 | 55 | 60 |
| Lightweight scrap + sinter | | | | | | |
| 1100 | 0.160 | 0.179 | 0.197 | 0.214 | 0.224 | 0.232 |
| 1200 | 0.379 | 0.409 | 0.437 | 0.464 | 0.489 | 0.512 |
| 1300 | 0.556 | 0.589 | 0.615 | 0.638 | 0.661 | 0.678 |
| 1400 | 0.662 | 0.688 | 0.714 | 0.735 | 0.752 | 0.773 |
| 1500 | 0.878 | 0.909 | 0.936 | 0.960 | 0.977 | 0.993 |
| Lightweight scrap and fluxed sinter | | | | | | |
| 1100 | 0.530 | 0.549 | 0.556 | 0.564 | 0.574 | 0.582 |
| 1200 | 0.609 | 0.639 | 0.657 | 0.664 | 0.689 | 0.691 |
| 1300 | 0.706 | 0.719 | 0.725 | 0.738 | 0.761 | 0.778 |
| 1400 | 0.802 | 0.808 | 0.824 | 0.835 | 0.852 | 0.873 |
| 1500 | 0.878 | 0.909 | 0.936 | 0.960 | 0.977 | 0.993 |



a)



b)

a – light scrap + agglomerate; b – light weight scrap and fluxed sinter

Figure 1 – Kinetic recovery curves of various iron ore materials at 1200-1600 °C

For inside the diffusion region, Jander, based on the parabolic distribution of the growth law of the product layer, proposed the equation:

$$(1 - \sqrt[3]{1 - \alpha})^2 = k\tau, \tag{3}$$

where α - conversion rate;
 k - process rate constant;
 τ - time elapsed since the start of the experiment.

A more correct equation for describing the constant of the solid-state process in a spherical grain was obtained by Gistling and Brownstein:

$$1 - (2/3 \cdot \alpha) - (1 - \alpha)^{2/3} = k\tau, \tag{4}$$

It should be noted that the Jander equation (3) and Gistling-Brownstein equation (4) are based on the general assumption that the rate of the diffusion process is inversely proportional to the thickness of the reaction product layer. The difference between these equations, which model a solid-phase process, is based only on different geometric assumptions: Jander considers diffusion leading to the formation of a reaction product at a flat interface; Ginstling and Brownstein solve the problem of diffusion in a spherical grain. According to the Gistling-Brownstein equation, most often used in studies of the kinetics of reduction of various materials [[18], [19], [20]], the mathematical model of the dependence of the degree of transformation of manganese ore on time is expressed as a straight line equation:

$$\alpha = a + bx, \tag{5}$$

where (b) – slope, represents the rate constant of the process.

Table 3 shows the results of processing experimental data using the Gistling-Brownstein model. As a general assessment, it can be indicated that this model with a sufficiently high correlation (R not less than 0.9) describes the studied process, which once again confirms its diffusion character, since this equation is developed for such processes.

Analyzing the data in Table 3, we can conclude that at the same temperature, various manganese ore materials are arranged in the following order in order to increase the rate constant of their interaction with coke carbon: manganese ore → unfluxed sinter → sinter with dolomite. Thus, at 1100 °C, the reduction rate constant of sinter with dolomite is 3.0 times higher than that of light-weight scrap mixed with conventional sinter. A similar effect can be achieved by increasing the process temperature.

Using the data in Table 3, one can find the temperature dependence of the rate constant for various types of materials under study. To do this, we set the following formula for the dependence of the rate constant on temperature, called in physical chemistry the Arrhenius equation

$$k = A \cdot \exp\left(-\frac{E_{act}}{RT}\right), \tag{6}$$

where A - constant;
 E_{act} - process activation energy;
 R - universal gas constant (8.3144 J/mol·K);
 T - temperature, K.

Table 3 – Results of experimental data processing according to the Gistling-Brownstein model

| Material | t, °C | y = a + bx | | R | k·10 ⁻³ |
|---|-------|------------|-------|-------|--------------------|
| | | a | b | | |
| Lightweight scrap with agglomerate | 1100 | -3.995 | 0.779 | 0.962 | 14.343 |
| | 1200 | -3.559 | 0.756 | 0.989 | 21.533 |
| | 1300 | -3.008 | 0.716 | 0.988 | 35.379 |
| | 1400 | -2.257 | 0.589 | 0.978 | 61.649 |
| | 1500 | -2.081 | 0.670 | 0.983 | 83.594 |
| Lightweight scrap with fluxed agglomerate | 1100 | -2.720 | 0.653 | 0.976 | 43.004 |
| | 1200 | -2.224 | 0.587 | 0.974 | 63.542 |
| | 1300 | -1.795 | 0.535 | 0.982 | 88.951 |
| | 1400 | -1.493 | 0.522 | 0.987 | 117.226 |
| | 1500 | -1.420 | 0.590 | 0.996 | 142.725 |

Table 4 – Kinetic characteristics of recovery

| Material | Equation | R | A | E_{act} , kJ/mol |
|-------------------------------------|-------------------------------|-------|-------|--------------------|
| Lightweight scrap (LVS) and sinter | $\ln k = -12722.0/T + 3.814$ | 0.998 | 70.43 | 105.775 |
| Lightweight scrap and fluxed sinter | $\ln k = -8408.37/T + 1.0171$ | 0.997 | 8.34 | 69.9105 |

Taking the logarithm of this expression gives the dependence

$$\ln k = \ln A - \frac{E_{act}}{R} \cdot \frac{1}{T}, \quad (7)$$

which can be represented as an equation of a straight line

$$\alpha = a + bx \quad (8)$$

Table 2 Data Processing in Coordinates $\ln k - 1/T$ makes it possible to find the coefficient b in equation (4.9), numerically equal to $-E_{act}/R$, and coefficient a , equal $\ln A$. With a known R (8,3144) one can find the activation energy of the process and the value of the pre-exponential coefficient A . The results of the calculations are placed in Table 4, which lists the required equations, correlation coefficients (R) and values of the apparent activation energy (E_{act}).

Analyzing the calculated activation energy data in Table 4, we can assume the diffusion nature of the inhibition of the reduction reaction of all the materials under study. Judging by the values of the activation energy of the process, the sinter with dolomite has the highest reducibility.

During the frontal reduction of oxides of iron and other elements with carbon, a slag shell is formed around the grains of materials, which consists mainly of acidic oxides of barren rock, non-metallic inclusions of lightweight scrap and reducing agent ash and has a high viscosity. Its thickness depends on the composition of the material, temperature, time and degree of reduction [19]. This shell is a slag barrier for the development of recovery along the depth of the grains of the materials used. Calcium and magnesium oxides, contained in an increased amount in dolomite, dissolve in the process of reduction in acidic and viscous slag-forming oxides - products that appear during the reduction of the leading elements of the

agglomerate, and as a result, the formation of more basic and, accordingly, liquid slags. Such slags easily destroy the slag diffusion barrier and expose the surface of the sinter core for a new contact between iron oxides and carbon.

Thus, the magnesium contained in the agglomerate removes the frontally occurring reactions of ore reduction, a diffusion barrier and contributes to a more complete and intensive reduction of oxides of iron and other elements.

Conclusions

The reduction kinetics of manganese agglomerates has been experimentally studied in comparison with other manganese ore materials currently used in the practice of silicomanganese production in the temperature range of 1100-1500°C. Experimental data are processed according to the Khrushchev kinetic model, which establishes a relationship between the degree of transformation of a substance and the process time at a constant temperature.

According to the increase in the rate constant of their interaction with coke carbon, the studied materials are arranged in the following order: lightweight scrap → non-fluxed sinter → fluxed sinter.

The kinetic characteristics of the reduction of agglomerated mill scale with carbon have been determined. The activation energies of the reduction processes of the studied materials are calculated, analyzing which it is possible to draw a conclusion about the diffusion nature of the inhibition of the reduction reaction of all the studied materials. The development of the reduction process is limited by the slag shell, which consists mainly of acidic oxides of the waste rock of the ore and reducing agent ash, which have a high viscosity.

In dolomite-fluxed agglomerate, the negative effect of the slag barrier is reduced due to the increased content of basic calcium and magnesium oxides in its composition, which dissolve in viscous

slag-forming oxides during the reduction process, resulting in the formation of low-melting and liquid-moving slags on the contact surface. Such slags easily destroy the slag diffusion barrier and expose the surface of the core of minerals for a new contact between the oxides of iron, silicon and other

elements with carbon, contributing to their more complete and intensive recovery.

Conflict of interest. On behalf of all authors, the correspondent author declares that there is no conflict of interest.

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Илемделген отқабыршақтан агломераттың тотықсыздануының изотермиялық кинетикасын зерттеу

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ТҮЙІНДЕМЕ

Илемделген отқабыршақтан агломератты қалпына келтірудің изотермиялық кинетикасын зерттеу Ж. Әбішев атындағы Химия-металлургиялық институтта жүргізілді. (Қарағанды қ.). Темірдің және басқа элементтердің тотықсыздану дәрежесіне температура мен қыздыру уақытының әсері Тамман пешінде термогравиметриялық әдіспен зерттелді. Тәжірибелерде тотықсыздандырығыштың мөлшері темір оксидтерін 60%-дан астам толық тотықсыздандыру үшін, сондай-ақ алынған металды 2,0% С-ға дейін көміртектендіру үшін көміртегінің стехиометриялық қажетті мөлшерін негізге ала отырып алынды. Температура мен процестің ұзақтығына байланысты зерттелетін материалдардың тотықсыздануының кинетикалық сипаттамаларын алу үшін алынған деректер ауқымы осы жағдайлар үшін арнайы әзірленген модельдер бойынша өңделді. Гистлинг-Браунштейн моделі бойынша мәліметтерді тәжірибелік өңдеу нәтижелері келтірілген. Жалпы бағалау ретінде жеткілікті жоғары корреляцияланғандыққа ие ($R > 0,9$ кем емес) бұл модель оның диффузиялық сипатын растайтын зерттелетін процесті сипаттайды. Белсендіру энергиясының есептелген мәліметтерін талдай отырып, барлық зерттелетін материалдардың тотықсыздану реакциясының тежелуінің диффузиялық сипатын болжауға болады. Процестің белсендіру энергиясының мәні агломераттың доломитпен (флюстелген агломерат) тотықсыздану қабілетінің жоғары екенін көрсетеді. Темірдің және басқа элементтердің оксидтерінің көміртегімен фронтальды тотықсыздануы кезінде материалдар түйіршіктерінің айналасында қож қабығы түзіледі, ол негізінен бос тау жыныстарының қышқылдық оксидтерінен, салмағы жеңіл сынықтардан, тотықсыздандырығыш күлдің металл емес қосындыларынан тұрады және жоғары тұтқырлыққа ие болады. Оның қалыңдығы материалдың құрамына, температураға, уақытқа және қалпына келу дәрежесіне байланысты.

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Исследование изотермической кинетики восстановления агломерата из прокатной окалины

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АННОТАЦИЯ

Исследования изотермической кинетики восстановления агломерата из прокатной окалины проводили в Химико-металлургическом институте им.Ж. Абишева (г. Караганды). Влияние температуры и времени нагрева на степень восстановления железа и других элементов исследовали в печи Таммана термогравиметрическим методом. Количество восстановителя в опытах принимали исходя из стехиометрически необходимого количества углерода для полного восстановления оксидов железа на более чем 60 %, а также на науглероживание полученного металла до 2,0 % С. Для получения кинетических характеристик восстановления исследуемых материалов в зависимости от температуры и продолжительности процесса проведена обработка полученного массива данных по специально для этих случаев разработанным моделям. Приведены результаты обработки экспериментальных данных по модели Гистлинга - Брунштейна. В качестве общей оценки данная модель с достаточно высокой коррелированностью (R не менее 0,9) описывает изученный процесс, что подтверждает его диффузионный характер. Анализируя расчетные данные энергии активации можно предположить диффузионный характер торможения реакции восстановления всех исследуемых материалов. Величина энергии активации процесса показывает, что наиболее высокой восстановимостью обладает агломерат с доломитом (офлюсованный агломерат). При фронтальном восстановлении оксидов железа и других элементов углеродом вокруг зерен материалов образуется шлаковая оболочка, состоящая преимущественно из кислых оксидов пустой породы, неметаллических включений легковесного лома и золы восстановителя и обладающая высокой вязкостью. Толщина ее зависит от состава материала, температуры, времени и степени восстановления.

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