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Electromagnetic field effects on the kinetics of solid-state reduction of iron oxides with gases

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Вплив електромагнітного поля на кінетику твердофазного відновлення оксидів заліза газами

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Анотація. Мета. *Метою роботи є дослідження фізико-хімічних закономірностей інтенсифікуючого впливу електромагнітного поля на процес твердофазного відновлення оксидів газами.* ***Методика.*** *Експерименти проводили у змінному магнітному полі з частотою змінного току в індукторі до 40 кГц. з використанням термогравіметричної методики. Процес непрямого відновлення досліджували із визначенням ступеню та швидкості відновлення оксиду у діапазоні температур 973-1373 К з використанням різних залізо- рудних матеріалів.* ***Результати.*** *Результати лабораторних експериментів свідчать про вплив високочастотного електромагнітного поля, що інтенсифікує, на процес твердофазного відновлення залізорудних матеріалів. Подано фізико-хімічну модель механізму інтенсифікуючого впливу ЕМП на процес відновлення. Високочастотне поле суттєво прискорює процеси дифузії, а також підвищує електронну та структурну дефектність кристалічних ґрат, що позитивно впливало на розвиток адсорбційно-хімічної ланки.* ***Наукова новизна.*** *Експериментально підтверджено вплив ЕМП на кінетику газового відновлення залізорудних матеріалів.* ***Практична значущість.*** *Інтенсифікація процесів твердофазного відновлення залізорудної сировини забезпечує інтенсифікацію процесу та підвищення продуктивності процесу.*

Ключові слова: *твердофазне відновлення, інтенсифікація, оксиди заліза, змінне електромагнітне поле.*

Abstract. Objective*. The aim of the work is to study the physicochemical correlations of the intensifying effect of an electromagnetic field on the process of solid-phase reduction of oxides by gases.* ***Methods****. The experiments were carried out in an alternating magnetic field with an alternating current frequency in the inductor up to 40 kHz using the thermogravimetric technique. The process of indirect reduction was studied to determine the degree and rate of oxide reduction in the temperature range of 973-1373 K using various iron ore materials.* ***Results****. The results of laboratory experiments indicate the influence of a high-frequency intensifying electromagnetic field on the process of solid-phase reduction of iron ore materials. A physicochemical model of the mechanism of the intensifying effect of EMF on the reduction process is presented. The high-frequency field significantly accelerates the diffusion processes and increases the electronic and structural defectiveness of the crystal lattice, which positively influenced the development of the adsorption-chemical link.* ***Scientific novelty****. The effect of EMF on the kinetics of gas reduction of iron ore materials has been experimentally confirmed.* ***Practical significance****. The intensification of solid-phase reduction of iron ore raw materials leads to the intensification of the process and increase of the process productivity.*

Keywords*: solid-phase reduction, intensification, iron oxides, alternating electromagnetic field.*

Introduction

One of the most important challenges facing the steel industry today is to reduce mineral and energy consumption, as well as to incorporate various man-made materials into the technological process. The most effective way to solve this problem is to further develop the physicochemical basis and technological aspects of solid-phase reduction of ore materials. The share of metals produced by this technology in the world is constantly increasing [1]. However, despite its significant advantages, the existing technological schemes of solid-phase reduction have a significant drawback - low productivity. Currently, various methods of intensification of reduction processes have been developed and successfully applied: physical, chemical-catalytic and energy impact on the reacting system. Common to these intensification methods is the impact on the diffusion and crystal-chemical links of the reduction process. However, the intensification of metal oxide reduction processes involving different types of energy impact remains insufficiently studied.

Various external energy (physical) influences as possible regulators of physical and chemical processes have long attracted the attention of researchers. Thanks to numerous studies, the most significant successes in this area have been achieved using electromagnetic and corpuscular radiation [2-6]. For example, under the influence of α-particles, the decomposition reactions of carbon monoxide, its oxidation, and many others are accelerated [6]. The observed effects are due to the excitation of gas molecules, their ionization, and the formation of atoms and radicals.

Radiation has a great impact on solids, including oxide semiconductors. High-frequency electromagnetic effects (visible light, γ-rays, etc.) cause the appearance of super-equilibrium free electrons and electron holes in crystals (possibly through an intermediate exciton state) [6,7]. Some of the free charge carriers can be localized on structural defects in the crystal lattice. All this leads to changes in the chemisorption and catalytic properties of the semiconductor surface. [2,6,7].

High-energy corpuscular radiation (fluxes of α and β particles, protons, neutrons, etc.) causes the appearance of additional vacancies and inter-node ions in the lattice of crystals [2,5,6,8]. The generated structural defects, in turn, affect the concentration of electronic defects in the semiconductor, its chemisorption and catalytic activity. Similar shifts occur during the development of nuclear reactions that lead to the appearance of foreign atoms in the lattice.

Radiation not only affects the reaction of gases with each other on the surface of solids (heterogeneous catalysis) but also changes the rate of interaction of the latter with gases. It has been established that irradiation can accelerate the oxidation of metals and their reduction from oxides [9]. Author in study [10] has shown a significant intensification of the reduction of iron oxides by H_2 and CO under the influence of ionizing and gamma irradiation (during the reaction or preliminary); at the same time, the temperature of the beginning of the process decreased at a noticeable rate. It should be noted that the positive effect of γ-rays was also observed when they were used simultaneously with catalysts. The observed kinetic shifts are usually associated with favorable changes in the conditions of gas chemisorption, weakening of metal-oxygen bonds, acceleration of ion diffusion through crystal lattices, and facilitation of the formation of new phases.

Acoustic effects of ultrasonic frequencies have a wide range of effects on the course of physical and chemical processes. They accelerate the processes of dissolution and diffusion in solid phases, and some chemical reactions [11]. It is shown in study [11] a significant increase in the rate of iron oxidation by air, its reduction by hydrogen and CO from oxides. These effects are associated with the intensification of external diffusion mass transfer due to mechanical perturbations of the gas medium; with the acceleration of solid-phase diffusion and the facilitation of crystal lattice rearrangement due to their loosening by ultrasonic vibrations, dissipation of wave energy at the gas/solid interface.

There is information on the effect of external electric fields on a wide range of processes [3,12]. Their imposition on semiconductor materials, changing the surface concentration of free charges, causes an electro adsorption effect that allows regulating the donor-acceptor chemisorption of gases. By influencing the position of the Fermi level, external electric fields create an electrocatalytic effect and affect the rate of chemical reactions. In strong fields, semiconductors are enriched with additional charge carriers (mainly due to thermoelectric and impact ionization), which also affects the surface properties and reactivity of the solid. It is worth noting the possibility of superimposing electron transfer on the diffusion flow in ionic crystals and metals [4,12].

There is connection between the magnetic properties of solids and their catalytic and adsorption properties, and reactivity with gases and also influence of magnetic fields on the development of some physicochemical processes. In recent years, a limited number of works have been published on the kinetics of the reduction of iron oxides by gases under magnetic effects (see [13-15]). The authors noted an increase in the rate of reduction of iron oxides by hydrogen under the application of a magnetic field and a decrease in the temperature of the beginning of the process; in the flow of CO and CH_4, there was no positive effect (in a constant and alternating field). The established regularities were unambiguously explained. For example, in [13], the acceleration of the process was associated with the attraction of hydrogen orthohydrogen molecules by ferromagnetic solid phases and the resulting increase in the pressure of the reducing agent near the reaction surface. In work [14], the intensification effect was interpreted in thermodynamic terms as the introduction of an additional amount of energy due to the magnetic field.

The kinetic regularities and the mechanism of reduction of iron oxides by gases under the conditions of application of electromagnetic fields of different reacting frequencies (up to $5 \cdot 10^4$ Hz) to the system were studied.

Experimental procedure

The reduction of iron ore samples streamlined by gases was carried out at the installation, the scheme of which is shown in Fig. 1. To generate electromagnetic effects, it was additionally equipped with a water-cooled inductor located coaxially with the reactor and heating element.

In the case of generating low-frequency magnetic fields, the inductor was a multi-turn copper wire solenoid. It was powered from the power grid through an autotransformer, which allowed changing the field strength H. A multivibrator was used to adjust the frequency of the latter ($f < 50$ Hz). In experiments with a constant magnetic field, the solenoid was powered through a rectifier.

To create electromagnetic effects of high frequencies ($f = 50$ kHz), an inductor made of a copper water-cooling tube was used. It was powered and the field parameters were controlled using a power generator UZG 5-1.6 and a master generator GZ-33. The power supplied to the inductor was set and maintained by an indicator with a scale range from 0 to 100 relative units (W). The voltage (U) was measured with a tube voltmeter.

Various iron ore materials were used in the research: chemically pure iron oxides and industrial concentrates. The following were subjected to reduction: Fe_2O_3 of AG qualification, crystalline (particle size 0.5-2 mm); iron ores in pieces and grains of various sizes -

Lysakovskaya (Fe_{total} = 41.6-43.7%), Kryvyi Rih hematite (Fe_{total} = 54.7%), magnetite (Fe_{total} = 56.7%), rich martite ore; raw and subjected to oxidative firing at 1523 K; fluxed agglomerate with basicity of 1.1 and 1.33 with oxygen content of 22.5 and 22.2%, respectively.

Results and discussion

At the first stage of the work, the effect of alternating magnetic fields of industrial frequency (f = 50 Hz) on the rate of reduction ($υ_r$) of crystalline iron oxide (Fe) by hydrogen was studied. The study revealed an intensifying effect of external influences, which increases with increasing W_{H2} (Fig. 2).

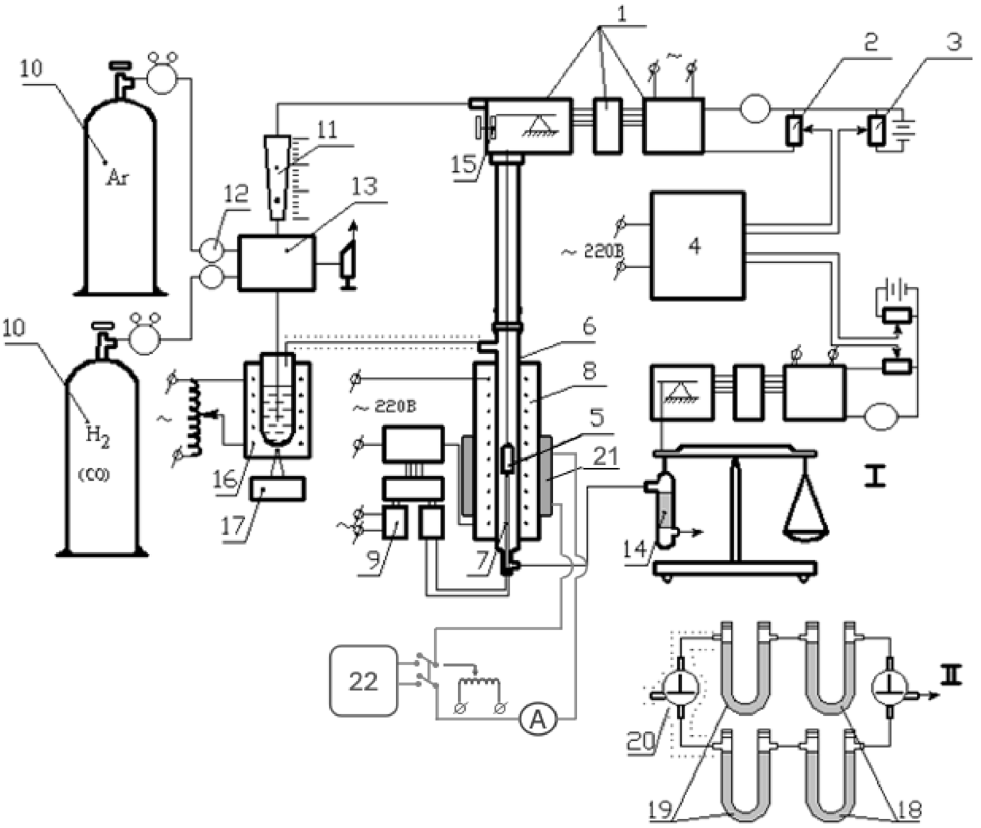

Fig. 1. The scheme of the experimental thermogravimetric setup *1-mechanoelectrical transducer; 2-scale divider; 3-counter-EMF; 4-automatic potentiometer KSP-4; 5-basket with a sample of the material under study; 6-reactor; 7-thermocouple PR 30/6; 8-resistance furnace; 9-temperature regulator VRT-3; 10-gas cylinders; 11-rotameter; 12-flow regulator; 13-valve box; 14-CO_2 absorber; 15-hoist; 16-saturator; 17-XA-thermocouple with PP-63; 18-CO_2 absorbers; 19-H_2O absorbers; 20-three-way valve, 21-water-cooled inductor, 22-frequency generator.*

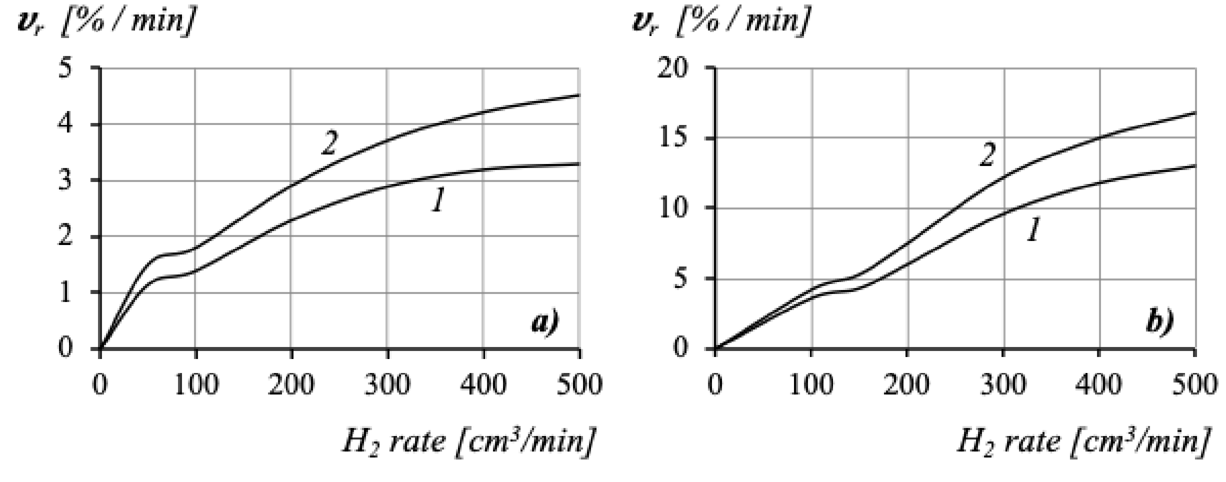

Fig. 2. Effect of an alternating field on the reduction of Fe_2O_3 by hydrogen at 773 K (a) and 973 K (b): *1-outside the field, 2-in the field H = 24 kA/m*

Temperature variations in the range of 773-1073 K showed that the application of a magnetic field accelerates the process most at 873-973 K. The character of the kinetic curves remains the same (Fig. 3a). Up to 973 K, the reduction (ω) developed stepwise; exceeding this temperature led to a zonal flow of the process. In the experiments at 773 K, the appearance of metastable wustite was observed.

The effect of an alternating magnetic field on the duration of the complete recovery of hematite $\tau_{\omega=100}$ in the temperature range 773-1073 K is illustrated in Table 1.

Similar patterns were observed in experiments with powdered materials - chemically pure Fe_2O_3 and Fe_3O_4. The magnetic field of industrial frequency significantly accelerated the oxygen removal almost throughout the entire process (Fig. 3b).

Table 1.
Time of complete reduction of crystalline Fe_2O_3 by hydrogen under normal conditions and under the application of an alternating magnetic field (H = 24 kA/m)

T, K		773	873	973	1073
$\tau_{\omega=100}$	Outside the field	34.5	26	21.7	17.2
	In the field	32	22.5	17.7	16

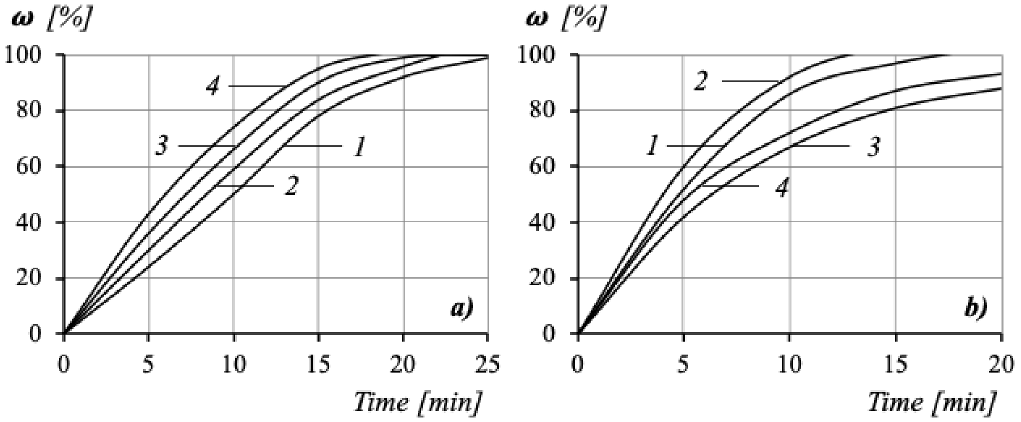

Fig. 3. Kinetics of reduction of iron oxides by hydrogen under industrial frequency EMF: a) - Fe_2O_3; *1,2-873 K, 3,4-973 K, 1,3-outside the field, 2,4-in the field;* ***b) - 973 K, powder;*** *1,2-Fe_2O_3, 3,4-Fe_3O_4; 1,3-outside the field, 2,4-in the field*

The next stage of the work was to study the effect of a high-frequency electromagnetic field on the kinetics of gas reduction of iron ore materials. The studies revealed significant opportunities to intensify the process using this method.

The reduction of chemically pure Fe_2O_3 with hydrogen (300 cm^3/min) showed that the application of an EMF (f = 25 kHz; W = 30, which corresponds to U = 80 V, H ~ 5 kA/m) strongly accelerates the removal of oxygen from the charge in the low temperature region: 573-673 K. Thus, ω, which was achieved in 20 min at 673 K, increased from ~ 40 to 70%, i.e., more than 1.7 times, and in the first 10 min of the experiment - 1.6 times. Increasing the temperature reduced the intensifying effect: at 873 K, it increased from 52 to ~ 65% or 1.25 times (Fig. 4).

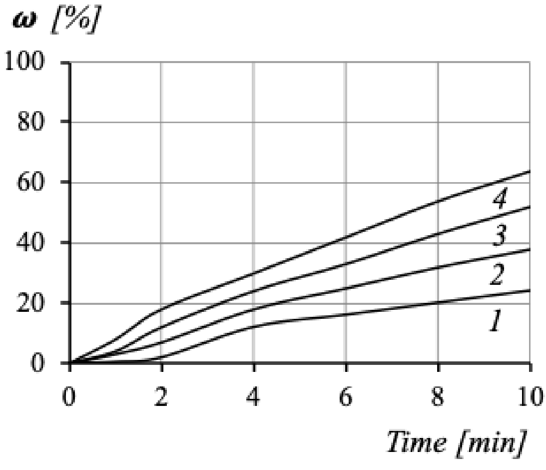

Fig. 4. Effect of a high-frequency electromagnetic field (f = 25 kHz, W = 30, H ~ 5kA/m) on the kinetics of Fe_2O_3 reduction by hydrogen (W_{H2} = 300 cm^3/min): *1,2-673 K; 3,4-873 K; 1,3-outside the field; 2,4-under the influence of the field, mode II*

The application of low-frequency magnetic fields (*f* ≤ 50 Hz) did not cause the charge to heat up. A thermocouple inserted inside the powdered Fe_2O_3, Fe_3O_4 and Fe charges did not record any temperature changes. Measurements during the reduction of granular Lysakivska ore with H_2 at 873 K led to similar results.

A different picture was observed under conditions of electromagnetic influences of high frequencies. A thermocouple located under the sample showed an increase in its temperature with a constant power consumption by the heating element. This additional heating of the charge decreased as the reduction temperature increased. The test showed, however, that the observed intensification of the process could not be reduced to a single heating (quantitative relationships are discussed below).

Given the above, experiments with the application of electromagnetic fields were carried out in two modes:

I - to fix the temperature rise caused by external energy effects without changing the power supply to the heater;

II - to stabilize the temperature in the reactor core by reducing the voltage supplied to the heating element.

The transition from the first mode to the second naturally reduced the accelerating effect of the field, but it remained very significant (Fig. 5a). Increasing the flow rate of H_2 contributed to the development of the process (Fig. 5b); however, the acceleration effect did not increase, as was the case with low-frequency magnetic effects.

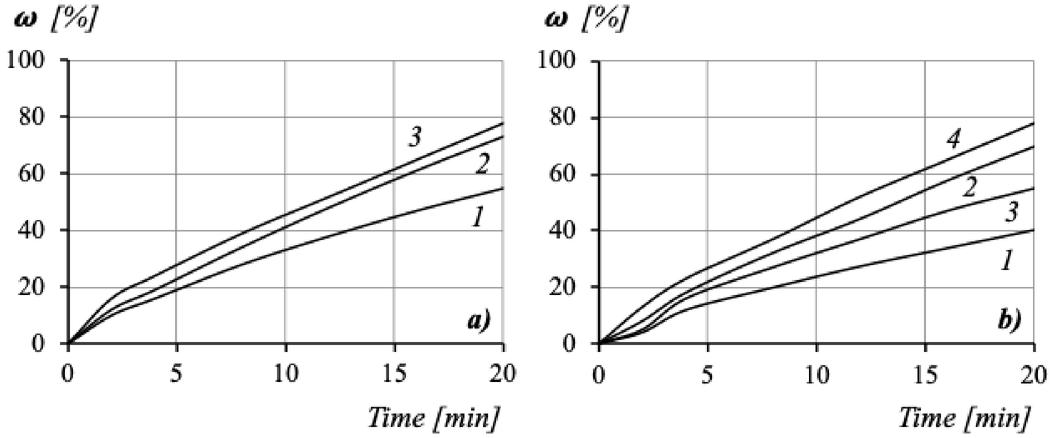

Fig. 5. Kinetics of Fe_2O_3 reduction by hydrogen under high-frequency electromagnetic action (f = 25 kHz, W = 30) at 673 K: a) - W_{H2} = 600 cm^3/min, *1-out of the field; 2,3-under the influence of the field (2-mode I, 3-mode II);* ***b) - mode II,*** *1,3-out of the field; 2,4-in the field; 1,2-W_{H2} = 300 cm^3/min; 3,4-W_{H2} = 600 cm^3/min.*

During the reduction of Fe_2O_3, a short incubation period was observed, followed by a self-acceleration of the process (Figs. 4 and 5). This may be due not only to the peculiarities of the development of the crystal-chemical link, but also to the heating of the sample after its transfer from the upper cold zone of the reactor to the working zone. Under normal conditions, the kinetic reduction curves were characterized by kinks, indicating a predominantly stepwise process. The application of high-frequency fields shortened the incubation period and smoothed out the kinks; a shift towards a zonal mode of process development occurred.

In general, similar patterns of the process and the same nature of the electromagnetic field effect were observed during the reduction of chemically pure Fe_3O_4 (Fig. 6).

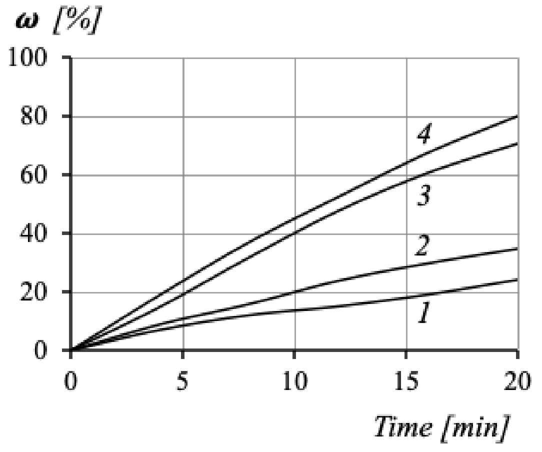

Fig. 6. The effect of high-frequency EMF (f = 40 kHz, W = 30) on the kinetics of Fe_3O_4 reduction by hydrogen with W_{H2} = 600 cm^3/min: *1,2-673 K; 3,4-973 K; 1,3-outside the field; 2,4-in the field; mode I*

Calculations show that at W_{H2} = 300 cm^3/min, the composition of the off gases at the first stage of Fe_2O_3 reduction is far from equilibrium. External energy influences do not significantly change this picture. At the subsequent stages of the process, under normal conditions, the H_2O concentration approaches equilibrium. In the case of superposition of fields, the water vapor content reaches equilibrium and even exceeds it. This should be attributed to some overlap of different degrees of recovery and inaccuracy of thermodynamic data, especially at low temperatures. At an increased H_2 flow rate of 600 cm^3/min, the exhaust gases contained a significant excess of reducing agent and gaseous products, apparently, did not significantly inhibit the accelerating effect of electromagnetic fields.

The intensification of the process also occurred in the case of reduction of chemically pure iron oxides with carbon monoxide (Fig. 7).

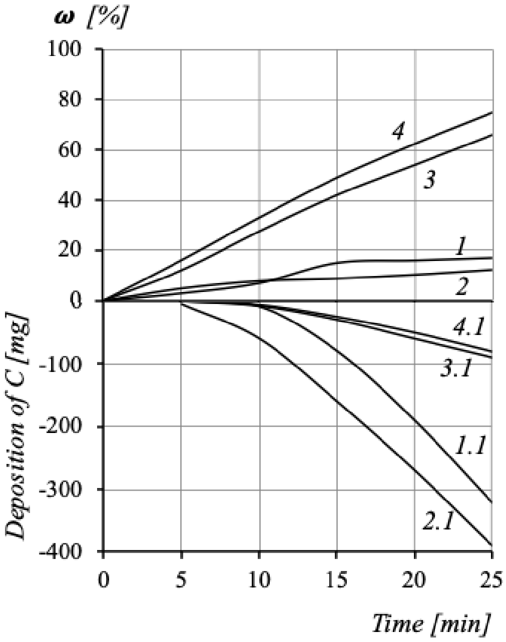

Fig. 7. Effect of a high-frequency electromagnetic field (f = 40 kHz, W = 30) on the kinetics of reduction of magnetite concentrate by carbon monoxide at (W_{CO} = 600 cm^3/min): *1,2-673 K; 3,4-973 K; 1,3 -outside the field; 2,4-under the influence of the field; mode I.*

At elevated temperatures, an increase in υ_r was observed throughout the experiment; the rate of the carbon gasification reaction, which proceeded slowly, was weakly dependent on the application of external effects. In the low-temperature region, electromagnetic fields accelerated the initial stage of oxygen removal and simultaneously promoted earlier decomposition. As a result, υ_r decreased, but, unlike the reduction under normal conditions, it remained significant despite the intensive course of the gasification reaction.

The analysis of experimental data showed that at W = 600 cm^3/min, the CO_2 concentration in the off gas at all stages of the recovery does not reach equilibrium values.

The degree of acceleration of the process by electromagnetic effects significantly depended on their parameters: frequency and power supplied to the inductor. In the tested frequency range, an increase in *f* to ~ 35 kHz generally accelerated the removal of oxygen (some deviations were observed around 15 kHz; later, the intensifying effect stabilized (Table 2).

Table 2.
Effect of the electromagnetic field frequency and power supplied to the inductor on the degree of reduction of Fe_2O_3 by hydrogen during 20 min at 673 K (W_{H2} = 600 cm^3/min; temperature regime II)

f, kHz with W = 30	Outside the field	2	10	20	30	40	50
$\omega_{T=20}$, %	54.5	64.5	68	66.5	74.5	78	78
W with *f* = 42 kHz	Outside the field	10	20	30	40		
$\omega_{T=20}$, %	54.5	58.5	67	78.5	92.5		

An increase in the power of external influences at different values of (*f*) proved to be very effective in terms of process acceleration. As for the reduction of Fe_2O_3 with hydrogen at 673 K, this is illustrated in Table 2. Similar results were obtained in the region of higher temperatures, as well as in the CO flow. For example, in the experiments on the reduction of Fe_3O_4 by carbon monoxide at 973 K, $\omega_{T=25}$ in the field with W = 30 and 50, the reduction rate increased from 67.5% to 76 and 82.5%, respectively.

Studies of the gas reduction of industrial iron ore materials have confirmed the significant potential of intensifying electromagnetic fields of high frequencies. The degree of acceleration of the process in experiments with magnetite concentrate (Fig. 8) was close to that of chemically pure iron oxides observed during the reduction.

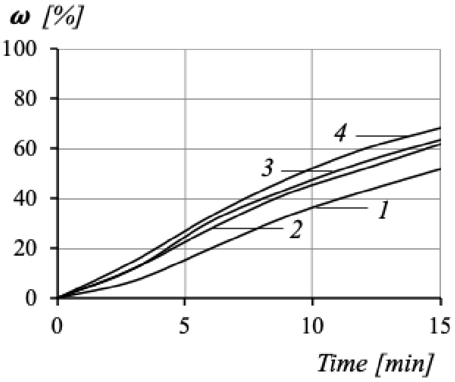

Fig. 8. Kinetics of reduction of magnetite concentrate by hydrogen (W_{H2} = 600 cm³/min) under high-frequency EMF (f = 25 kHz, W = 30): *1,2-773 K; 3,4-973 K; 1,3-outside the field; 2,4-under the influence of the field; mode I*

The general patterns of the field effect were preserved during the transition from powder to granular charge. Thus, in the experiments with Lysakivska ore, the application of the field (f = 40 kHz, W = 50, which corresponds to U = 120 V, H ~ 3.5 kA/m) increased the $υ_r$ at 773 K by 1.5 times. This result was obtained under conditions of stabilized temperature; in mode **I**, the average value of $υ_r$ almost doubled. In the region of elevated temperatures, the effectiveness of external influences decreased, but even at 1173 K, there was a significant reduction in the time of complete recovery (Fig. 9). No noticeable heating of the charge was observed.

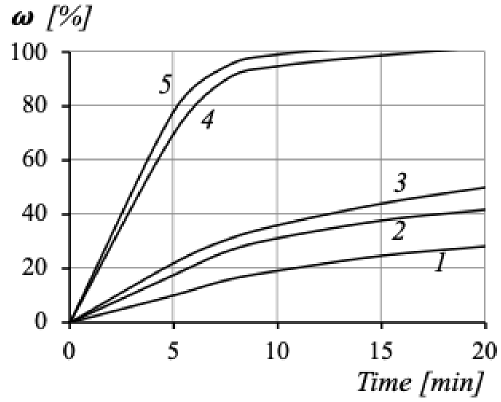

Figure 9. Kinetics of reduction of iron ore materials by hydrogen (W_{H2} = 600 cm³/min) under conditions of high-frequency EMF (f = 40 kHz, W = 50): iron ore in grains 0.5-1.0 mm; *1,2,3-773 K; 4,5-1173 K; 1,4-out-of-field; 2,3,5-under EMF conditions; 2,5-mode II; 3-mode I;*

The studies showed that the nature of the field frequency effect coincides with that discussed earlier. As before, the power of external influences was strongly felt. This can be illustrated by the results of the reduction of magnetite concentrate with hydrogen at 973 K: an electromagnetic field with a frequency of 25 kHz and W = 30 increased the reduction from 63 to ~ 69%, and in the case of W = 50, this value increased to 77%.

External influences significantly accelerated the removal of oxygen from pelleted ore materials. The positive effect of high-frequency fields (f = 40 kHz, W = 50) was established in experiments with oxidized magnetite pellets. Even at an elevated temperature of 1173 K, the oxygen recovery in the H_2 stream increased from 58.5 to 68%, and in the CO stream from 24 to ~ 30%.

Conclusions

1. The intensifying effect of weak electromagnetic fields with a frequency of $0.5\text{-}5{\cdot}10^4$ Hz during the reduction of iron by gases - H_2, CO and their mixtures - has been established.
2. High-frequency electromagnetic fields most strongly accelerated the reduction of iron in the temperature range of 573-773 K. With an increase in frequency (up to 35-40 kHz) and intensity, the effectiveness of their superposition increased.
3. In this temperature range, electromagnetic effects (*f* = 25-40 kHz; H = 3.5-5 kA/m) increased the rate of reduction of chemically pure iron oxides and Lysakivskiy ore by 1.5-2 times.
4. Studies have shown that the application of low-frequency magnetic fields passivates the product of metallization of iron ore raw materials. High-frequency effects have different effects on the oxidisability of the reduction product, but they do not lead to its pyrophoricity. Only the iron obtained by reduction in a variable cross-section reactor under pulsed effects on the reacting system had high oxidisability. However, it was eliminated by a ten-minute exposure of the metallized concentrate at 973 K in an inert atmosphere.

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