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SOLID-PHASE REDUCTION OF METALS FROM RICH OXIDES OF CHROMIUM ORES

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Annotation

The process of carbothermic solid-phase reduction of solid chrome ore in the crystal chrome spinelide lattice was experimentally investigated. It is established that the recovery is developed by an electrochemical mechanism. The release of metals occurs both on the surface and in the volume of ore grains. The transfer of electrons to the cations from the reductant is carried out by anion vacancies formed during the interaction of the reductant with the oxide. The primary product of reduction is a metallic alloy of iron and chromium, which are reduction at the same time, but at a different and variable rate, depending on their concentration in the local oxide volume. The formation of carbides on the surface of the ore grain as a result of the interaction of reduction metals with carbon causes the outflow of metal atoms from the grain volume to its surface, the appearance of cationic vacancies in the oxide, the dissolution of the metal phase and the reduction of the reduction process. With the destruction of carbides by silicon with the formation of silicocarbides, the process of extracting oxygen anions from the oxide lattice is restored, which generally contributes to the resumption of the reduction process.

Key words: chrome ore, ferrochrome, solid-phase reduction, crystal lattice of oxides, anion vacancy.

Introduction

Introduction solid-phase carbothermic reduction of iron and chromium plays an important role in the technology of production of carbon ferrochrome. It proceeds in the upper horizons of the ore-thermal furnaces, is accompanied by the formation of carbides of iron and chromium and leads to the formation of a high-carbon alloy, which later goes through decarburization in the so-called ore layer in the lower horizons of the furnaces. A large number of works, including experimental ones, are devoted to the study of these processes, but their results are interpreted ambiguously by different authors. The consequence of this is the presence of a wide range of existing ideas about the mechanism of metal reduction of metals by solid carbon, a relatively complete overview, which is given, for example, in [1].

In chromic ores, iron and chromium are found in chemical compounds like oxide spinels $(Me^{2+})[Me^{3+}]_2O_4$ type, along with cations of the more difficultly reduction metals like magnesium and aluminum [2, 3]. Due to the equality of charges and proximity of the ionic radius, Fe^{2+} cations can be mixed indefinitely with Mg^{2+} cations, and Cr^{3+} cations with Fe^{3+} and Al^{3+} cations, as a result of which the composition of spinel in chromium ruds in general form corresponds to the formula $(Mg^{2+}, Fe^{2+})[Fe^{3+}, Al^{3+}, Cr^{3+}]O_4$.

In case of solid-phase reduction in such ores, unlike traditional monometallic iron ore raw materials, direct contact of the reduction cations with the reduction agent can play only a minor role, since their basic mass is separated from the reductant by compounds of difficult reduction metals. In this case the most important link in the solid-phase reduction mechanism in this case is the processes in the crystal lattice of the complex oxide, which determine its transformation into the metal crystal lattice. In our opinion, this is precisely the essence of the recovery process, without which it is impossible to imagine a recovery mechanism as a whole.

Earlier, we experimentally investigated the reduction processes of metals in pieces of disseminated chrome ores. The results of these studies have led to the conclusion that the restoration is developed by an electrochemical mechanism, and the channels for the rapid propagation of the recovery process into the volume of ore pieces are layers of non-metallic silicates [4-6].

The purpose of this work is an experimental study of the process of solid-phase carbothermic reduction of iron and chromium in the chromespinelide lattice.

Experimental

The object of the research was the rich Kempirsai chrome ores. The experiments were carried out in a sealed resistance furnace with a graphite heater according to a previously developed method for the recovery of metals from poor and impregnated chrome ores [4,5]. Due to the much slower progress of the reduction process into the volume of a piece of rich ore, the ore was crushed to the size of 1...3 mm, that is, almost to the size of the ore grain. As a reductant, thin energetic coals were used, ground to size 0...0.63 mm. The mixture of ore and coal was poured into a graphite crucible and placed in the working zone of the furnace, the furnace was sealed to create a reducing atmosphere, heated and kept at a temperature of 1400°C or 1500°C for 60 to 180 minutes. The temperature inside the reaction mixture was controlled by a tungsten-rhenium thermocouple BP5/20. Crucibles with the reaction mixture were cooled to room temperature with the furnace. Ore grains were separated by sieving from the remainder of the reductant and poured into epoxy resin for the making of microsections. The sections were studied on optical and electron scanning microscopes JSM-6560LV and JSM-6460LV with wave and energy dispersive analyzers. In parallel, the original ore and reduction products were subjected to X-ray phase diffraction analysis on a DRON-4 diffractometer.

Results

In the original ore, chromespinelide is represented by practically little-modified primary ferrichrompicatite $(Mg^{2+},Fe^{2+})[Fe^{3+},Al^{3+},Cr^{3+}]O_4$, which is typical of rich ores of the Kempirsay deposit [3]. Layers and blotches of “empty” rock are formed by aqueous silicates of the pyroxene $2(Mg,Fe)O{\cdot}SiO_2$ group (Fig. 1,a). X-ray phase analysis in the silicate phase also detected nickel in the composition of the complex silicate $(Mg_{1.02}Fe_{0.08}Ni_{0.09})SiO_4$.

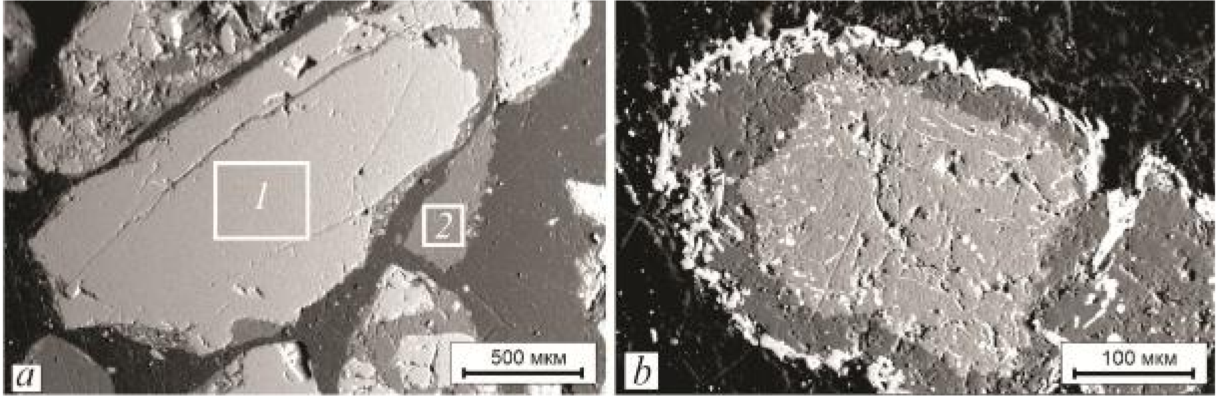


Figure 1 – The type of chromespinelide grains in the original ore (*a*) and after reduction at temperature of 1400°C for 1 hour (*b*).

The content of elements (at.%) in the grain of spinelide (1) and in the waste rock (2):

	O	Mg	Al	Si	Cr	Fe
1	56,76	9,06	3,98	–	24,89	5,31
2	67,92	19,38	–	10,52	–	2,18

After reduction exposure at a temperature of 1400°C, relatively large precipitates of metals were found in the form of a discontinuous shell on the surface of ore grains, and in a slightly altered internal volume of grains in the form of small metal formations forming separate lines. Between the outer metal shell and the inner little-modified part of the grain, an interlayer of the changed oxide phase is visible, which, with increasing exposure time, changes little in size, but becomes more pronounced (Fig. 1, *b*).

In the metal phase, chromium and iron are detected on the surface of the former grain of ferrichromicatite in proportion approximately to their content in the initial ore grain (analysis point 1; Fig.2, *a*). In the dark oxide phase adjacent to the metal shell, silicon, potassium, and calcium, previously not detectable in the ore grain, appear; silicon is found in relatively large amounts, the concentration of magnesium and aluminum increases, iron disappears, and a small amount of chromium remains (analysis point 2; Fig. 2 , *b*). In the metal phase released inside the less-altered grain behind the dark oxide phase, two phases of iron-chrome alloy are slightly different in color: iron-rich and chromium-depleted or vice versa, iron-depleted and chromium-rich (Fig.2, *b*; points 3 and 4)). In the oxide phase, adjacent to the metal precipitated inside the little-altered grains, small dark growths of magnesium aluminum silicate with an admixture of sodium, potassium and calcium cations appear (Fig.2, *b*; point 5). In the composition of the spinelid, the concentrations of iron and chromium decrease, the concentrations of magnesium and aluminum somewhat increase (Fig. 2, *b*; point 6).

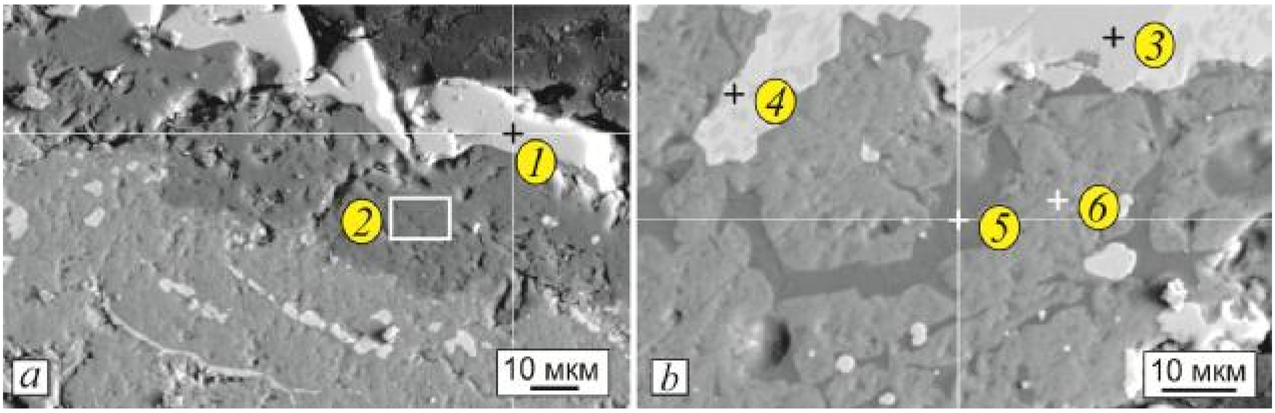


Figure 2 – The new growths in the chrome spinelide grain after reduction at the temperature of 1400°C for 1 hour

The content of elements (at.%) in the points of analysis:

	O	Na	Mg	Al	Si	K	Ca	Cr	Fe
1	-	-	-	-	-	-	-	83,07	16,93
2	63,10	-	13,99	6,70	13,70	0,51	0,34	1,65	-
3	-	-	-	-	-	-	-	76,28	23,72
4	-	-	-	-	-	-	-	23,88	76,12
5	52,89	1,11	13,14	2,73	16,17	0,66	2,32	2,10	-
6	56,86	-	11,95	4,84	-	-	-	25,94	0,41

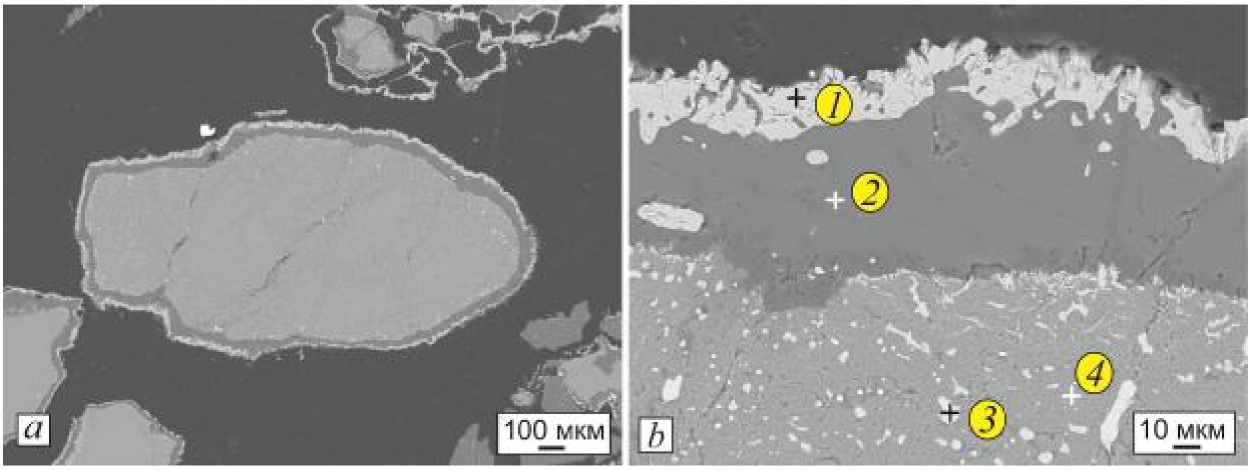


Figure 3 – New growths in chrome spinelide grain after reduction at 1400°C for 3 hours

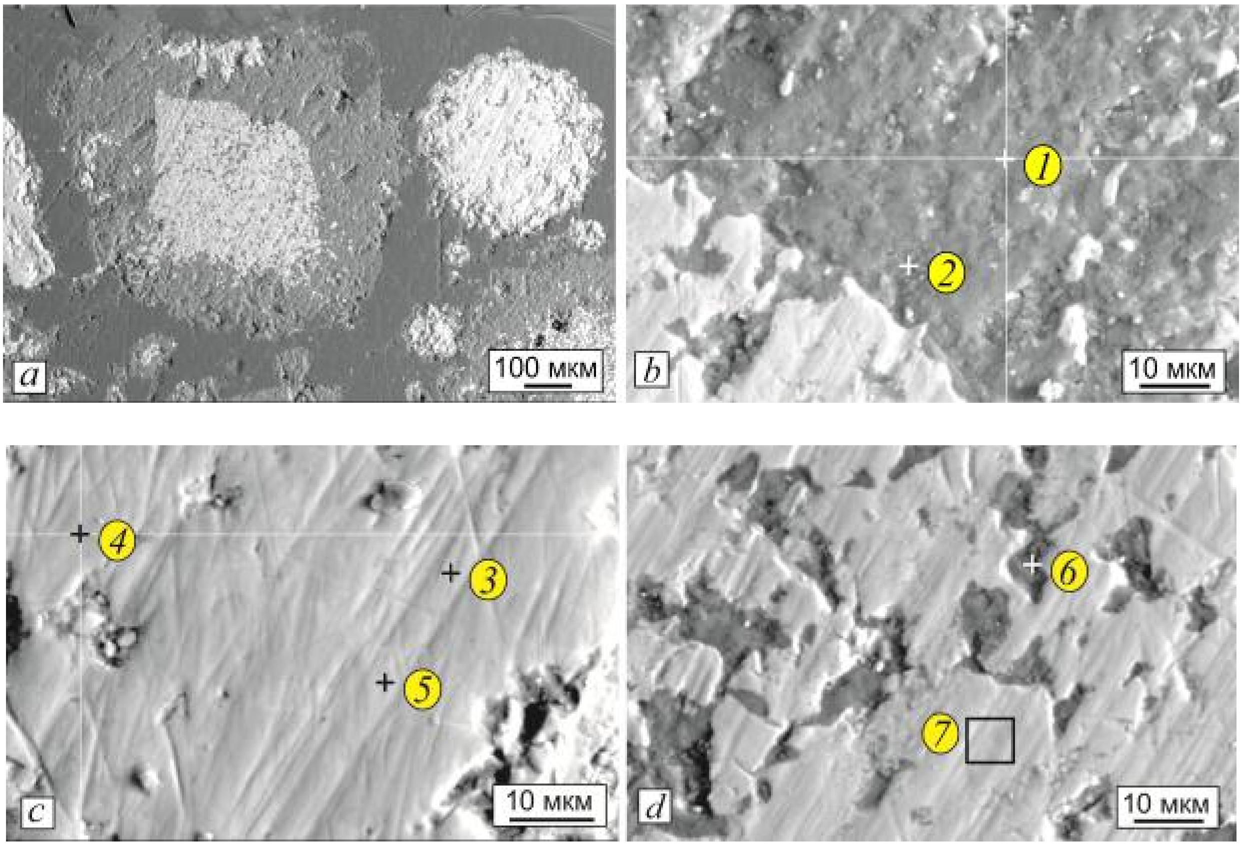
The content of elements (at. %) in the points of analysis:

	C	O	Na	Mg	Al	Si	S	K	Ca	Cr	Fe	Ni
1	44,17	-	-	-	-	-	-	-	-	46,17	9,66	-
2	-	61,63	0,16	9,08	7,91	16,91	0,27	0,10	3,31	0,55	0,08	-
3	-	-	-	-	-	-	-	-	-	45,69	53,83	0,48
4	-	60,48	-	10,7	4,50	0,07	-	-	0,03	20,51	4,14	-

The increase in the duration of exposure at a temperature of 1400°C to 3 hours of fundamental changes in the nature of the distribution of the products of reduction is not detected (Fig. 3, *a*). But at the same time, the zone distribution of chemical interaction products became clearer: with the same width of the buffer zone between the metal shell on the surface and the relatively little changed grain core, this zone was practically cleared of metallic secretions. In the metal shell, carbides are reliably detected on the surface of the grains and by micro X-ray spectral analysis (Fig. 3, point 1; Fig. 4). X-ray diffraction analysis revealed the compound $(Fe,Cr)_7C_3$ (Fig. 4). Magnesium aluminum silicate with an admixture of sodium, potassium, calcium cations, a small content of chromium and iron cations (Fig. 3, point 2) was formed in the buffer zone, which became a more plastic oxide phase, and the complex spinel was enriched with magnesium and aluminum inside the grains due to a decrease in chromium and iron cations . Small concentrations of previously undetectable silicon and calcium are also found in spinel (Fig. 3, point 4). In metallic particles in the grain volume, the ratio between the concentrations of iron and chromium is almost equal to unity (Fig. 3, point 3).

Significant changes in the nature of the distribution of the interaction products and in the composition of the phases caused an increase in the exposure temperature to 1500°C (Fig. 4). The outer shell of the grains, which previously consisted of chromium and iron carbides, almost disappeared; only its individual sections survived, and independent sphere-like formations formed from the material of these shells (Fig. 4, *a*). Instead of aluminum silicates, magnesia and aluminum-magnesium spinel appeared in the buffer oxide zone (Fig. 4, *b*; points 1 and 2), representing $MgAl_2O_4$ and MgO. The core of the grain is almost completely metalized, and the core metal, along with chromium and iron, contains a noticeable amount of silicon (Fig. 4, *c*; points 3-5). The oxide residue in the core of the former ore grain contains only aluminum-magnesium spinel (Fig. 4, *g*; point 6).

New sphere-like formations arising from carbides that previously existed on the grain surface are formed by an alloy of chromium, iron, and silicon (Fig. 4, *e* and *f*; points 8 and 10). Only in individual microscopic volumes of these conglomerates are carbides found (Fig. 4, *f*; point 10).



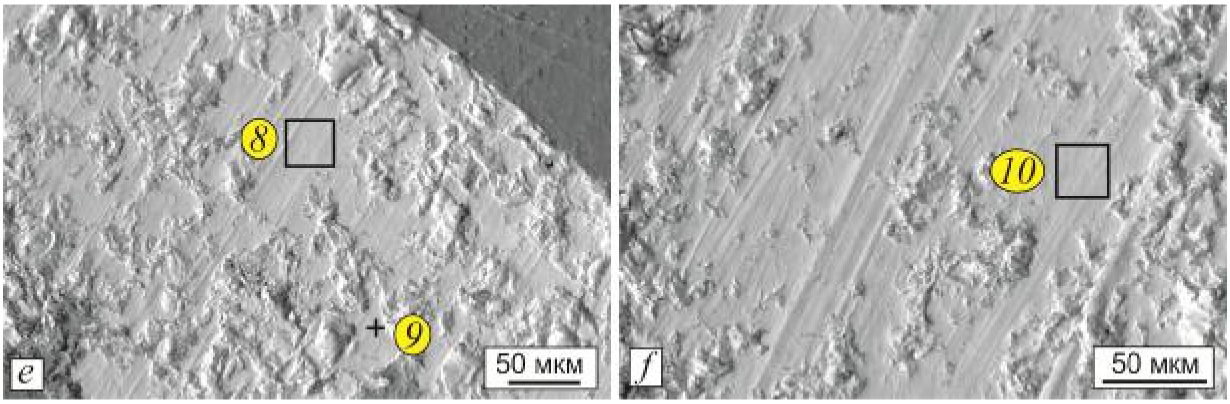


Figure 4 – X-ray products for chromium ore after reduction process at 1500 ºC for 3 hours

The content of elements (at.%) In the points of analysis:

	C	O	Mg	Al	Si	Cr	Fe	Ti	Mn	Ni	Phase
1	-	50,16	48,70	0,76	-	0,39	-	-	-	-	Magnesium
2	-	56,07	31,38	12,08	-	0,30	-	0,16	-	-	Spinel
3	-	-	-	-	8,80	73,42	17,78	-	-	-	Silicide
4	-	-	-	-	2,15	83,53	13,14	-	0,18	-	Silicide
5	-	-	-	-	17,53	62,61	19,86	-	-	-	Silicide
6	-	56,56	22,53	18,74	0,34	1,84	-	-	-	-	Spinel
7	-	-	-	-	3,49	82,41	14,10	-	-	-	Silicide
8	-	-	-	-	15,21	46,86	37,19	-	-	0,74	Silicide
9	49,79		-	-	-	45,37	4,84	-	-	-	Carbide
10	-	-	-	-	8.80	73.42	17.78	-	-	-	Silicide

Discussion

The results of experiments with the solid-phase reduction of iron and chromium from the crystal lattice of almost unchanged natural ferrihrompicatite confirmed the electrochemical mechanism of this process. Earlier [7, 8] we showed that the essence of the reduction process is not the removal of oxygen from the ore by the reaction MeO + C = Me + CO, but the reaction $Me^{2+} + 2e = MeO$.

This implies the electrochemical nature of the process, the admissibility of the spatial separation of metal reduction reactions and the oxidation of the reductant, the possibility of closing the electrical circuit between the oxidizer and the reductant by the flow of electrons, the reduction of metals surrounded by oxygen anions, i.e. inside the oxide occur without the transfer of reagents. For the formation of a metal phase in the volume of the spinelide, it is not necessary to bring a reductant or cations of the metal reduction on the surface of the metal to the place of its separation, and also to withdraw the reaction product in the form of CO or CO_2. The only condition necessary for this is the appearance of “extra” electrons in this place, which can be localized by cations that located in this place, which will lead to the appearance of metal atoms.

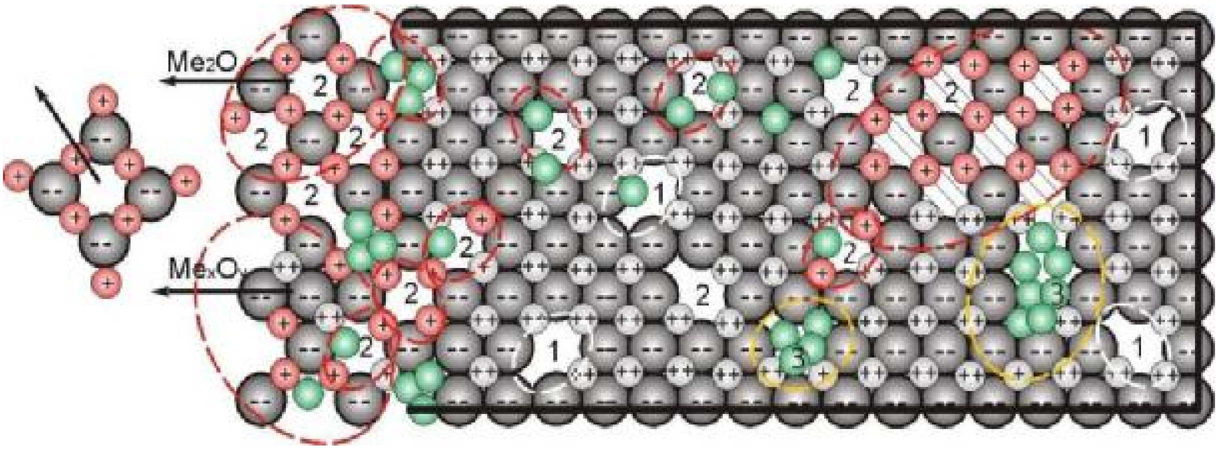


Figure 5 – Scheme of formation of the metal phase in the crystal lattice of oxides: 1 – thermal pair (anion + cation) vacancies, 2 – reduction (charged) anion vacancies, 3 – nucleus of the metallic phase

In carbothermic reduction, a source of "extra" electrons is a chemical reaction of extracting oxygen on the surface of a piece or ore grain. As a result of the removal of oxygen, an oxygen vacancy charged with two electrons that is formed in the anion sublattice of the oxide, which is mandatory for storing the equality of positive and negative charges in the lattice as a whole and in the local volume. The oxide surface is loosened by anion vacancies, and the “extra” electrons in an anion vacancy under the action of the charge of cations are displaced and localized at the cations, reducing the charge of the cations to zero, i.e. to a metallic state. "Extra" electrons move with the anion vacancy and accumulate in the places of disappearance (runoff) of vacancies, which are usually associated with lattice defects. In places where vacancies flow, cations with a zero charge accumulate, which, with a certain amount of them, is accompanied by the formation of a metallic bond between them and the appearance of a metallic nucleus (Fig. 5).

The location of the release of the metal phase is determined by the rate of formation and dispersion of charged vacancies. With a high rate of formation and a low rate of dispersion, the metal phase is released on the surface. The low rate of formation and the relatively high rate of scattering of vacancies lead to the formation of complexes corresponding to lower oxides on the surface, their separation and sublimation, as well as to the displacement of electrons in the oxide volume and the release of the metallic phase inside the oxide one.

It is considered [1,2] that, due to a greater change in the Gibbs energy of the system, the primary product of the carbothermic reduction are carbides. It is noted that, in accordance with the thermodynamic parameters, iron is first reduction from FeO, and then chromium is reduction from the formed Cr_2O_3 oxide, whose carbides form a solution of iron and chromium carbides.

In our experiments, the primary product of reduction both on the surface and inside the grain is a carbon-free alloy of iron and chromium. The reduction of chromium and iron in the lattice of ferrihrompicatite enriches spinelide with elements that are not renewable under these conditions – magnesium and aluminum. Increased the concentration of previously undetectable in the spinel due to low concentrations of silicon, sodium, potassium, calcium and titanium leads to the appearance of complex silicates formed by these cations and spinel residues dissolved in silicates between the metal shell on the surface and the grain core of the buffer shell.

The formation of a silicate phase from the remnants of the ore grain contradicts the conclusion of the authors [2] about the impossibility of dissolving silicon cations in chromespinelides. These authors believe that, according to the geometric relationships, all cations with an ionic radius from 44 pm to 100 pm can be included in the spinel structure, and since r_{Si}^{4+} = 0.39 pm and r_{Ca}^{2+} = 106 pm, neither silicon nor calcium should not enter the spinel crystal lattice. The results of our experiments rather confirm the point of view of the authors [9–11] about the possibility of substitution like $2Cr^{3+} \rightarrow Mg^{2+} + Si^{4+}$ or $Cr^{3+} + Al^{3+} \rightarrow Mg^{2+} + Si^{4+}$ in the spinel structure and dissolving potassium and sodium cations in spinelide with the formation of anion vacancies. As a result of such substitution in the lattice of spinel is also possible the presence of the cation Ti^{4+}, which is detected in the spectrum of the point 2 in Fig. 5.

Formed on the surface of the metal alloy is gradually saturated with carbon, which leads with an increase in exposure at a temperature of 1400°C to the formation of carbides in the surface metal layer. The formation of carbides is accompanied by a further decrease in the Gibbs energy of the system; therefore, gradually, the entire alloy that formed on the surface turns into carbides. As a result of the lower chemical potential of chromium and iron in carbides and the contact of carbides with free carbon of the reductant, the transition into the carbide layer of iron and chromium cations from the silicate phase of the buffer zone begins later. This leads to disruption of the established equilibrium between the metal and oxide phases in the buffer zone and is accompanied by the dissolution of the metal formations which located here. The visible result is the release of the zone from metallic inclusions.

However, the extraction of cations from the oxide lattice and the formation of cationic vacancies in the silicate phase leads to an increase in the density of the close-packed anion sublattice [12, 13]. As a result, the mass transfer processes in the lattice slow down and practically stop. Therefore, an increase in the duration of exposure at a temperature of 1400°C to 3 hours does not lead either to an expansion of the buffer zone or to an increase in the amount of the metal phase in the center of the ore grain.

At a temperature of 1500°C, silicon is reduction from the silicate phase of the buffer zone by chromium and iron carbides. Carbides are transformed into silicocarbides [14, 15] with different ratios of silicon and carbon concentrations and with a lower melting point, which form sphere-like conglomerates, freeing the surface of the ore grain for further interaction of the reductant with oxygen anions. The process of formation of charged anion vacancies accelerate again, the recovery process extends to the entire volume of ore grains. At the same time, in the central zone of the ore grain an alloy of iron, chromium and silicon is formed, but without the participation of carbon (points 3, 4, 5, 7 in Fig.5). A small amount of oxide residue from the original chromespinelide after extraction of iron, chromium and silicon is converted to magnesia spinel and magnesia. The two-phase metal particles with different ratios of chromium and iron found in the samples (see Fig. 2; points 3 and 4) are the result of the periodic nature of the reaction of alternate reduction of elements from the complex oxide as the concentration of cations of either one or the other decreases.

Conclusions

1. Solid-phase reduction of iron and chromium in the volume of the complex oxide is carried out selectively by an electrochemical process by the propagation in the crystal lattice of anion vacancies formed on the surface of the reaction with a reductant, and associated with the vacancies of "extra" electrons.

2. The primary product of the carbon-thermal reduction of metals from chromespinelides are metallic iron and chromium. The formation of carbides is a secondary process of interaction of metals released on the surface with carbon of the reductant.

3. The formation of carbides on the reduction surface inhibits the formation of anion vacancies in the lattice due to the extraction of carbide-forming metals from the lattice, the formation of cationic vacancies in it, and the dissolution of the already formed metal phase, which leads to a compaction of the anion sublattice and the reduction of the reduction process.

4. The restoration of silicon dissolved in the spinelide lattice, the replacement of carbon and the formation of chromium and iron silicocarbides contributes to the destruction of the carbide shell, the resumption of the formation of anion vacancies and the recovery of metals in the entire volume of spinelide.

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БАЙ ХРОМ КЕНДЕРІ ТОТЫҚТАРЫНАН МЕТАЛДАРДЫҢ ҚАТТЫ ФАЗАЛЫ ТОТЫҚСЫЗДАНУЫ

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Түйін

Жұмыстың нәтижесі бойынша темір мен хромның кешенді қорытпа көлеміндегі қатты фазалық тотықсыздануы электрохимиялық үрдіс арқылы селективті жүзеге асатыны анықталды.

Зерттеу барысында, бұл үдеріс электрохимиялық тотықсыздану механизмі бойынша жүретіні анықталды. Металдардың бөлінуі хромшпинелидтердің бетінде ғана емес, ішкі фазасында бөлінуі де жүреді. Электрондарды катиондарға тотықсыздандырғыш арқылы беріліп, оксидпен өзара әрекеттесуі кезінде пайда болатын аниондық вакансиялармен жүзеге асырылады. Тотықсыданудың бастапқы өнімі – бір мезгілде тотықсыздану нәтежесінде пайда болатын темір-хром қорытпасы ауыспалы тотықсызану жылдамдықтармен жүреді.

Кілттік сөздер: хром кені, феррохром, қатты фазалы тотықсыздану, тотықтардың кристалды торы, аниондық вакансия.

ТВЕРДОФАЗНОЕ ВОССТАНОВЛЕНИЕ МЕТАЛЛОВ ИЗ ОКСИДОВ БОГАТЫХ ХРОМОВЫХ РУД

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Резюме

По результатам работы определено, что твёрдофазное восстановление железа и хрома в объёме комплексного оксида осуществляется селективно электрохимическим процессом путём распространения в кристаллической решётке анионных вакансий, образовавшихся на поверхности реагирования с восстановителем, и связанных с вакансиями «лишних» электронов.

Установлено, что восстановление развивается по электрохимическому механизму. Выделение металлов происходит как на поверхности, так и в объеме рудного зерна. Передача электронов катионам от восстановителя осуществляется анионными вакансиями, образующимися при взаимодействии восстановителя с оксидом. Первичным продуктом восстановления является металлический сплав железа и хрома, которые восстанавливаются одновременно, но с разной и переменной скоростью, зависящей от их концентрации в локальном объеме оксида.

Ключевые слова: хромовая руда, феррохром, твёрдофазное восстановление, кристаллическая решётка оксидов, анионная вакансия.