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Thermodynamic modeling of interphase distribution of chromium and boron in slags of AOD reduction period

A. A. Babenko, V. I. Zhuchkov, N. I. Kel'✉,
A. G. Upolovnikova, R. R. Shartdinov

Institute of Metallurgy of the Ural Branch of the Russian Academy of Sciences (101 Amundsen Str., Yekaterinburg 620016, Russian Federation)

✉ dunnington@mail.ru

Abstract. The paper presents the results of a thermodynamic modeling of the chromium and boron reduction from slags of reduction period of argon-oxygen decarburization (AOD) by a complex reducing agent containing silicon and aluminum. Using the simplex lattice method, an experiment planning matrix is constructed containing 16 compositions of the oxide system $CaO–SiO_2$ – (3 – 6 %) B_2O_3 – 12 % Cr_2O_3 – 3 % Al_2O_3 – 8 % MgO of variable basicity 1.0 – 2.5. The results of thermodynamic modeling are graphically presented in form of dependence of equilibrium distribution of chromium and boron on the slag composition at temperatures of 1600 and 1700 °C. The constructed diagrams make it possible to quantify the influence of the temperature, basicity and B_2O_3 in the slag on equilibrium interphase distribution of chromium and boron. It is established that increasing the slag basicity from 1.0 to 2.5 improves the process of chromium reduction, but restores the boron stability. With an increase in B_2O_3 content in the slag, a slight deterioration of chromium reduction process occurs, while the boron content in the metal increases. With a simultaneous increase in basicity up to 2.5 and a decrease in boron oxide in the slag from 5 to 3 %, the interphase distribution coefficient of chromium is reduced to $1.5 \cdot 10^{-3}$. Changing the process temperature from 1600 to 1700 °C does not have a negative effect on the process of chromium reduction, but worsens the boron reduction conditions. Based on analysis of the formed slag phases and thermodynamics of the reactions of their formation, it is established that chromium is mainly reduced by aliminum with only partial development of silicothermy. The residual silicon content reduces boron, thereby limiting its concentration in the metal. The results of high-temperature experiments showed high correspondence with the results of thermodynamic studies.

Keywords: stainless steel, argon-oxygen decarburization (AOD), reduction period, thermodynamic modeling, chromium, boron, interphase distribution

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Термодинамическое моделирование межфазного распределения хрома и бора в шлаках восстановительного периода АКР-процесса

А. А. Бабенко, В. И. Жучков, И. Н. Кель✉,
А. Г. Уполовникова, Р. Р. Шартдинов

Институт металлургии Уральского отделения РАН (Россия, 620016, Екатеринбург, ул. Амудсена, 101)

✉ dunnington@mail.ru

Аннотация. В работе приведены результаты термодинамического моделирования процесса восстановления хрома и бора из шлаков восстановительного периода аргонокислородного рафинирования комплексным восстановителем, содержащим кремний и алюминий. При помощи симплекс решетчатого метода построена матрица планирования эксперимента, содержащая 16 составов оксидной системы $CaO–SiO_2$ – (3 – 6 %) B_2O_3 – 12 % Cr_2O_3 – 3 % Al_2O_3 – 8 % MgO переменной основности 1,0 – 2,5. Результаты термодинамического моделирования представлены графически в виде диаграмм зависимости равновесного распределения хрома и бора от состава шлака при температурах 1600 и 1700 °C. Построенные диаграммы позволили количественно оценить влияние температуры, основности и содержания

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B_2O_3 на равновесное межфазное распределение хрома и бора. Установлено, что повышение основности шлака с 1,0 до 2,5 улучшает процесс восстановления хрома, но ухудшает восстановление бора. При увеличении содержания B_2O_3 в шлаке происходит незначительное ухудшение процесса восстановления хрома, при этом увеличивается содержание бора в металле. При одновременном повышении основности до 2,5 и снижении содержания оксида бора в шлаке с 5 до 3 % коэффициент межфазного распределения хрома снижается до $1{,}5 \cdot 10^{-3}$. Изменение температуры процесса с 1600 до 1700 °С не оказывает существенного влияния на процесс восстановления хрома, однако ухудшает условия восстановления бора. На основе анализа фаз формируемого шлака и термодинамики реакций их образования установлено, что восстановление хрома протекает в основном за счет алюминия с частичным развитием силикотермических реакций. Остаточное содержание кремния восстанавливает бор, чем объясняется его низкая концентрация в металле. Результаты проведенных высокотемпературных экспериментов показали высокую согласованность с результатом термодинамического моделирования.

Ключевые слова: нержавеющая сталь, аргонокислородное рафинирование, восстановительный период, термодинамическое моделирование, хром, бор, межфазное распределение

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Introduction

Stainless steel is an absolutely essential part of a modern economy – the annually growing volumes of its consumption and a wide range of applications from medical products [1] to structural materials prove this statement[1]. Stainless steel is so popular because it is resistant to corrosion in various aggressive environments as an oxide layer, with a high concentration of chromium (12 wt. % and higher), forms on the metal surface, which prevents the steel from contacting air oxygen [2 – 4]. Despite the obvious advantages of stainless steel, the domestic production volumes are modest and the demand for this steel is covered by imports [5].

Currently, the main method of producing low-carbon stainless steel is the duplex process with smelting carbonaceous semi-product (1.5 – 2.0 wt. % C) in an arc furnace followed by treatment in the argon-oxygen decarburization (AOD) unit [6; 7]. The AOD process includes two periods: oxidation and reduction. During the oxidation period, the carbonaceous semi-product of stainless steel is decarbonized by blowing a mixture of oxygen and argon through it. When the carbon concentration in the metal drops to 0.03 wt. % or less, the reduction period of melting begins, during which the bath of the unit is purged with nothing but argon, and lime, ferroalloys (ferrosilicon, ferrosilicochrome) and calcium fluoride are added [8].

As a result of chromium oxidation by oxygen, the concentration of Cr_2O_3 in the slag increases, which negatively affects the technological processes occurring in the reduction period of melting, the intensity of their development being limited by the viscosity of the formed oxide system. According to [9], Cr_2O_3 usually has low solubility (5 %) in $CaO–SiO_2–Al_2O_3–MgO$ based slags, which increases their melting point and, consequently, viscosity. Therefore, calcium fluoride (CaF_2) is used to reduce the viscosity of slags in the reduction period of melting. The use of this flux has the following downsides: it is environmentally unfriendly as volatile carcinogenic fluorine compounds are formed, physical properties of the formed slags are inconsistent and the effect of silicate decomposition of solid slags persists during storage. Therefore, it is reasonable to explore other liquefying additives to replace calcium fluoride, for example, pegmatite [10], Al_2O_3 [11] or B_2O_3 [12]. Although the use of Al_2O_3 prevents the formation of volatile fluoride compounds, according to [11], the refining ability of the slag deteriorates. As such, its use is limited. Therefore, the boron-containing material is a reasonable choice as it is an inexpensive, available and environmentally friendly fluxing material.

Although B_2O_3 is an acidic oxide and facilitates polymerization [13], it helps to reduce slag viscosity by changing the structural components of the melt mesh. Adding B_2O_3 to the slag helps to improve kinetics of chromium reduction and metal desulfurization [14; 15]. Hardening characteristics of low-carbon steel are to improve and the aging effect is to reduce [17] due to expected partial reduction of boron by silicon and aluminum dissolved in the steel, followed by its transfer to the metal, in addition to the slag liquefying with boron oxide [17].

Practically no domestic or foreign researchers have explored the effectiveness of the interphase distribution of chromium and boron during their reduction by a complex ferroalloy containing aluminum and silicon.

The paper presents the results of thermodynamic modeling of equilibrium interphase distribution of chromium and boron reduced by silicon and aluminum from the $CaO–SiO_2–B_2O_3–Al_2O_3–Cr_2O_3–MgO$ oxide system by aluminum ferrosilicon, a complex ferroalloy.

Materials and methods

We performed thermodynamic modeling of the equilibrium interphase distribution of chromium and

[1] Stainless Steel in Figures 2019. URL: http://www.worldstainless.org/Files/issf/non-image-files/PDF/ISSF_Stainless_Steel_in_Figures_2019_English_public_version.pdf (accessed on 27.03.2023)

boron reduced by silicon and aluminum of the complex reducing agent (aluminum ferrosilicon) from the $CaO-SiO_2-B_2O_3-Al_2O_3-Cr_2O_3-MgO$ oxide system using the HSC Chemistry 6.12 software package. This software is based on the calculating equilibrium compositions and the amount of resulting products, using the Gibbs energy minimization algorithm.

Thermodynamic modeling was performed for the temperatures of 1600 and 1700 °C. The mass of the working medium was 115 kg (100 kg of metal and 15 kg of slag) with the gas phase (N_2) volume of 2.24 m^3 and the system pressure of 0.098 MPa. The amount of the reducing agent is 0.89 kg. The interphase distribution coefficients of chromium and boron were obtained by their concentration ratios in the slag and metal ($L_B = (B_2O_3)/[B]$ and $L_{Cr} = (Cr_2O_3)/[Cr]$).

The oxide system composition corresponds to 16 points of the local simplex plan and is presented in Table 1. In addition to calcium, silicon and boron oxides, all slags include chromium, magnesium and aluminum oxides in the amount of 12, 8 and 3 %, respectively. The metal part is represented by stainless steel containing, %: 16.0 Cr; 0.03 C; 0.28 Si; 0.010 S; 1.46 Mn; 6.98 Ni; 0.01 Al; the rest is Fe and aluminum ferrosilicon (AFS), a complex alloy containing, %: 55.8 Si; 18.8 Al; 25.4 Fe.

The results of thermodynamic modeling are presented by approximating mathematical models in the form of a reduced polynomial of the third degree, which describe the influence of the slag composition of the studied oxide system on the interphase distribution coefficients of chromium and boron at temperatures of 1600 and 1700 °C [18]. The adequacy of the constructed mathematical models was tested by three control points, not included in the experiment planning matrix, using *t*-criterion at the significance level of 0.01.

Figs. 1 and 2 graphically present the results of mathematical modeling in the form of composition – property diagrams. The solid lines show isolines of the equilibrium interphase distribution of chromium and boron, while thin lines reflect the basicity of the slag (CaO/SiO_2) with its value indicated.

Along with thermodynamic modeling of the equilibrium interphase distribution of chromium and boron, we conducted high-temperature experimental studies using an electric resistance furnace in magnesia crucibles in an argon current at 1600 °C. The low-carbon stainless steel was held for 30 min under slag in the points used for local simplex. The temperature was measured using a BP5/20 tungsten thermocouple. Metal samples were prepared from chips of AISI 304 stainless steel and steel ST3SP, as well as the slag from two base points Y_1 and Y_3 (Table 1). We took the test charge of ground metal and slag in the quantities of 75 and 50 g to achieve the maximum phase contact surface and to exclude the influence

Table 1. **Composition of slag in 16 points of the local simplex plan**

Таблица 1. **Состав шлака 16 точек плана локального симплекса**

Slag index	Slag composition						
	in pseudocomponent coordinates, unit fractions				in coordinates of initial components, wt. %		
	X_1	X_2	X_3	X_4	CaO	SiO_2	B_2O_3
Y_1	1.00	0	0	0	37.00	37.00	3
Y_2	0	1.00	0	0	52.86	21.14	3
Y_3	0	0	1.00	0	50.71	20.29	6
Y_4	0	0	0	1.00	35.50	35.50	6
Y_{12}	0.67	0.33	0	0	42.29	31.71	3
Y_{13}	0.33	0.67	0	0	47.57	26.43	3
Y_{21}	0	0.67	0.33	0	52.14	20.86	4
Y_{22}	0	0.33	0.67	0	51.43	20.57	5
Y_{31}	0	0	0.67	0.33	45.64	25.36	6
Y_{32}	0	0	0.33	0.67	40.57	30.43	6
Y_{41}	0.33	0	0	0.67	36.00	36.00	5
Y_{42}	0.67	0	0	0.33	36.50	36.50	4
Y_{121}	0.67	0	0.33	0	41.57	31.43	4
Y_{122}	0.33	0	0.33	0.33	41.07	30.93	5
Y_{131}	0.33	0.33	0.33	0	46.86	26.14	4
Y_{132}	0.33	0	0.67	0	46.14	25.86	5

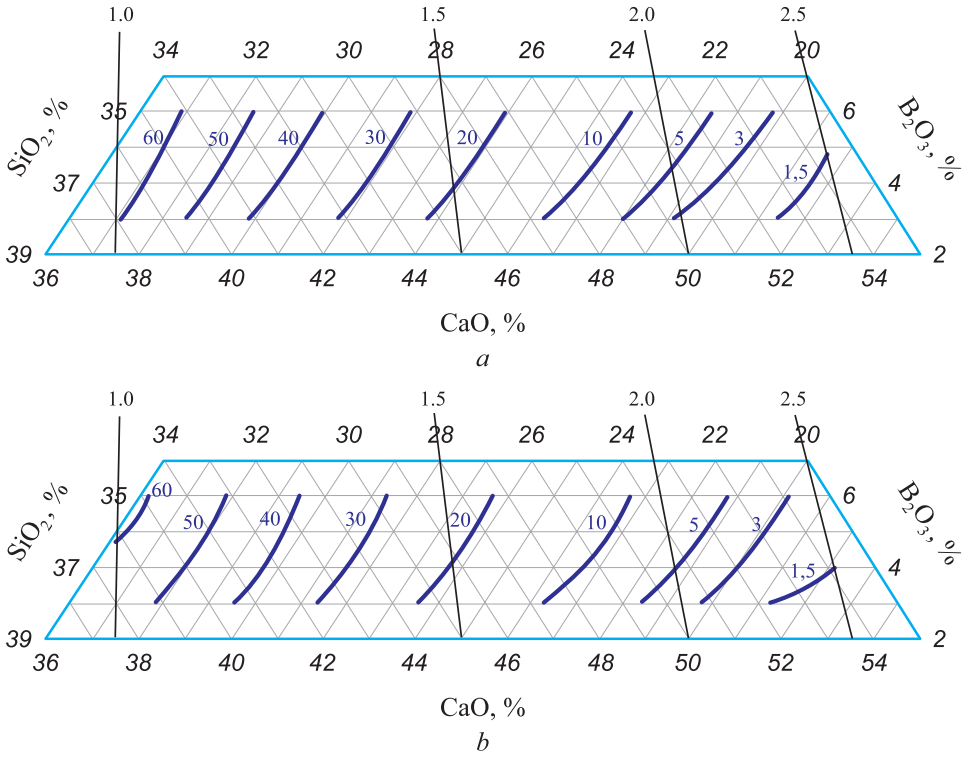

Fig. 1. Dependence of the coefficient of equilibrium interphase distribution of chromium on the slag chemical composition at 1600 (*a*) and 1700 °C (*b*)

Рис. 1. Зависимость коэффициента равновесного межфазного распределения хрома от химического состава шлака при 1600 (*a*) и 1700 °C (*b*)

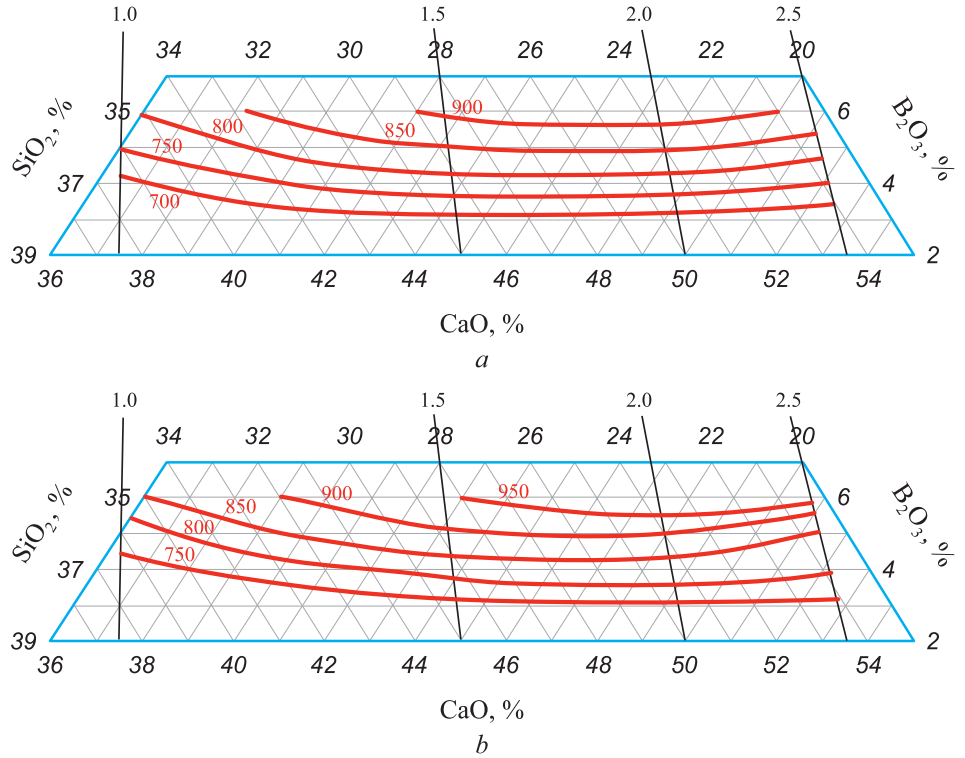

Fig. 2. Dependence of the coefficient of equilibrium interphase distribution of boron on the slag chemical composition at 1600 (*a*) and 1700 °C (*b*)

Рис. 2. Зависимость коэффициента равновесного межфазного распределения бора от химического состава шлака при 1600 (*a*) и 1700 °C (*b*)

of the mass of metal and slag phases on the interphase distribution coefficients of chromium and boron [19].

Results and discussion

Table 2 and Figs. 1, 2 present the results of thermodynamic modeling of the equilibrium interphase distribution of chromium and boron depending on the basicity of the slags of the studied oxide system and temperature.

With the slag basicity ranging from 1.0 to 1.5 and the boron oxide concentration varying from 3 to 6 wt. %, the interphase distribution coefficient of chromium dropped from $60 \cdot 10^{-3}$ to $20 \cdot 10^{-3}$ at 1600 °C (Fig. 1, *a*). The increase in the slags basicity to 2.5, with the boron oxide concentration changing from 5 to 3 %, results in drop of the interphase distribution coefficient of chromium to $1.5 \cdot 10^{-3}$, which is indicative of more effective chromium reduction due to growing basicity of formed slags. The increase in boron oxide concentration comes with a slight deterioration of the chromium reduction process. The rise in the B_2O_3 content in the slag from 3 to 6 % is accompanied (for example, at basicity of 2.0) by an increase in the equilibrium interphase distribution coefficient of chromium from $3 \cdot 10^{-3}$ to $5 \cdot 10^{-3}$ at a temperature of 1600 °C (Fig. 1, *a*). The temperature rise from 1600 to 1700 °C has a slight impact on the interphase distribution coefficient of chromium (Table 2). At 1700 °C in the considered range of basicity and boron oxide content, it remains at the level ranging from $60 \cdot 10^{-3}$ to $1.5 \cdot 10^{-3}$ (Fig. 1, *b*).

With the basicity of formed slags ranging from 1.0 to 2.5, as the boron oxide concentration rises from 3 to 6 %, the equilibrium interphase distribution coefficient of boron increases from 700 to 900 (Fig. 2, *a*). We can clearly observe the influence of boron oxide on the equilibrium interphase distribution coefficient, while the impact of basicity is weak. For example, in the basicity range of 1.5 – 2.5, the equilibrium interphase distribution coefficient of boron is 900, its oxide concentration ranging from 5.7 to 6.0 %. The decrease of boron oxide concentration to 5.0 – 5.3 % in the considered basicity range causes the drop of the equilibrium interphase distribution coefficient of boron to 850. The behavior of the equilibrium interphase distribution coefficient of boron follows a similar pattern when the boron oxide concentration reaches 3.0 – 3.4 %.

The process temperature rising to 1700 °C leads to a slight increase in L_B by 50 units and deteriorates the process of boron reduction (Fig. 2, *b*).

The positive impact of basicity of formed slags in the studied range of the chemical composition on the chromium and boron reduction can be qualitatively explained in terms of the phase composition formation (Table 3) and thermodynamics of reactions of chromium and boron reduction by aluminum and silicon (Table 4).

Table 2. **Interfacial distribution coefficient of chromium and boron**

Таблица 2. **Коэффициент равновесного межфазного распределения хрома и бора**

Slag index	Basicity (CaO/SiO_2)	1600 °C		1700 °C	
		$L_{Cr} \cdot 10^{-3}$	L_B	$L_{Cr} \cdot 10^{-3}$	L_B
Y_1	1.0	60.28	619.114	56.26	642.657
Y_2	2.5	0.90	654.000	0.98	724.683
Y_3	2.5	2.16	887.343	2.31	983.439
Y_4	1.0	69.43	796.648	64.95	845.092
Y_{12}	1.3	27.53	677.126	25.63	710.389
Y_{13}	1.8	6.71	685.229	7.21	733.197
Y_{21}	2.5	1.19	745.779	1.48	802.996
Y_{22}	2.5	1.60	822.055	1.95	845.092
Y_{31}	1.8	12.89	922.397	12.85	976.023
Y_{32}	1.3	36.95	885.245	33.96	921.957
Y_{41}	1.0	66.68	751.965	60.48	777.277
Y_{42}	1.0	63.46	694.136	57.51	719.056
Y_{121}	1.3	31.45	762.105	29.07	797.220
Y_{122}	1.3	34.20	830.599	31.51	866.934
Y_{131}	1.8	8.73	780.574	9.09	831.897
Y_{132}	1.8	11.07	858.519	11.22	911.182

According to the results of thermodynamic modeling (Table 3), the composition of chromium-containing phases of low-base slag Y_1 is represented mainly by Cr_2O_3 and $CaO·Cr_2O_3$ phases, the number of which decreases in the process of chromium reduction from 8.1 and 5.4 % to 0.7 and 0.4 %, respectively. At the same time, the process of chromium reduction by silicon mainly occurs following the reactions (1) and (2) (Table 4), which is confirmed by the increase in content of SiO_2 and $CaSiO_3$ in the final slag from 4.8 and 18.8 % to 5.8 and 20.6 %, respectively (Table 3). The reduction of chromium by aluminum occurs following the reactions (6) and (7) (Table 4), which is confirmed by the increase in the content of the products of these reactions Al_2O_3 and $CaO·Al_2O_3$ from 0.8 and 0.3 % to 1.2 and 0.4 % (Table 3).

The reduction of chromium from low-base slag Y_4 (basicity of 1.0) occurs following the same reactions as for slag Y_1. Low-base slags are characterized by reduction of chromium mainly by aluminum as a result of reactions (6) and (7) with partial development of silicothermic reactions (1) and (2) (Table 4), which is attributed to the higher negative ΔG value of aluminothermic reactions compared to silicothermic ones.

In the highly basic slag Y_2 (basicity of 2.5) in the presence of a large amount of free CaO, the reduction of chromium by silicon is more active (reactions (2) – (4))

Table 3. **Slag phases involved in reduction of chromium and boron at 1600 °C**

Таблица 3. **Фазы шлака, участвующие в процессе восстановления хрома и бора при температуре 1600 °С**

Phase, %	Slag							
	Y_1		Y_2		Y_3		Y_4	
	before	after	before	after	before	after	before	after
Cr_2O_3	8.1	0.7	1.0	0.002	1.6	0.007	8.7	0.9
$CaO·Cr_2O_3$	5.4	0.4	15.1	0.020	14.2	0.040	4.5	0.4
$Ca_2B_2O_5$	4.4	4.2	2.8	3.4	7.5	9.0	7.9	7.6
$Ca_3B_2O_6$	0.5	0.4	15.1	5.6	10.2	8.1	0.6	0.6
Al_2O_3	0.8	1.2	0.3	0.6	0.4	0.8	0.8	1.3
$CaO·Al_2O_3$	0.3	0.4	2.1	3.1	1.6	2.3	0.2	0.3
CaO	0.2	0.2	5.0	3.4	2.7	1.8	0.2	0.2
SiO_2	4.8	5.8	0	0.1	0.1	0.2	6.0	6.9
$CaSiO_3$	18.8	20.6	3.2	5.0	4.3	6.8	17.9	19.4
$2CaO·SiO_2$	8.9	9.0	33.7	37.2	26.4	27.9	6.5	6.6
$3CaO·2SiO_2$	10.0	10.8	6.1	11.0	6.9	11.9	6.7	7.2

Table 4. **Change in Gibbs energy during reduction of chromium and boron from oxides at 1600 °C**

Таблица 4. **Изменение энергии Гиббса реакций восстановления хрома и бора из оксидов при 1600 °С**

Number	Chemical reactions	ΔG_{1600}, kJ
Chromium reduction		
(1)	$Cr_2O_3 + 1.5Si = 2Cr + 1.5SiO_2$	–210.95
(2)	$Cr_2O_3 + 1.5Si + 1.5CaO = 1.5\ CaSiO_3 + 2Cr$	–347.82
(3)	$1.3Cr_2O_3 + 2Si + 3CaO = 3CaO·2SiO_2 + 2.7Cr$	–542.60
(4)	$Cr_2O_3 + 1.5Si + 3CaO = 1.5·2CaO·SiO_2 + 2Cr$	–441.64
(5)	$CaO·Cr_2O_3 + 1.5Si = 2Cr + CaO + 1.5SiO_2$	–141.83
(6)	$Cr_2O_3 + 2Al = Al_2O_3 + 2Cr$	–421.04
(7)	$CaO·Cr_2O_3 + 2Al = CaO·Al_2O_3 + 2Cr$	–408.11
Boron reduction		
(8)	$Ca_2B_2O_5 + 2Al = 2CaO·Al_2O_3 + 2B$	–50.148
(9)	$Ca_3B_2O_6 + 2Al = 3CaO·Al_2O_3 + 2B$	–78.91
(10)	$Ca_3B_2O_6 + 1{,}5Si = 1.5·2CaO·SiO_2 + 2B$	–12.79

(Table 4). Due to the slag high basicity, the content of $CaO \cdot Cr_2O_3$ in Y_2 is much higher than in low-base Y_1. Therefore, a considerable amount of this phase is reduced by aluminum following the reaction (7) (Table 4). It should be noted that after chromium reduction, the chromium-containing phases are present in negligible amounts, i.e., the content of Cr_2O_3 drops from 1.0 to 0.002 % and that of $CaO \cdot Cr_2O_3$ – from 15.1 to 0.02 % (Table 3). Similarly, chromium is reduced from the high-base slag Y_3.

Boron is insignificantly reduced from the slag as the change of Gibbs free energy of its reduction by aluminum and silicon from calcium borates is minimal, therefore, the transition of boron to metal for all studied slag compositions following the reactions (8) – (10) is insignificant (Table 4).

We conducted high-temperature experimental studies to verify if the results of thermodynamic modeling of the chromium and boron interphase distribution are adequate. The experiment showed that the Cr_2O_3 content in the slag at 1600 °C is 0.96 wt. %, which corresponds to the interphase distribution coefficient of $49.8 \cdot 10^{-3}$. The interphase distribution of boron reaches 648 with a residual B_2O_3 content in the slag of 3.89 wt. %. In general, the experimental results are close to the thermodynamic modeling, the kinetic factors accounting for the difference between them.

Conclusions

The thermodynamic modeling enabled us to obtain new data, based on which we constructed approximating mathematical models of the composition – property relation with graphical representation in the form of diagrams showing the equilibrium interphase distribution of chromium and boron depending on the process temperature, B_2O_3 content and basicity of the studied oxide system. Based on the plotted diagrams, we conducted quantitative evaluation of the impact that the above-mentioned factors had on the equilibrium interphase distribution of chromium and boron.

It was found that the increase in the basicity of the oxide system from 1.0 to 2.5, other conditions being equal, favorably affects the completeness of chromium reduction. At the same time, the increase in boron oxide concentration is accompanied by a slight decrease of the chromium reduction. The process temperature rise has no significant effect on the chromium reduction, but negatively affects the reduction of boron. It was determined that chromium is mostly reduced by aluminum with partial development of silicothermic reactions. The performed high-temperature experiment confirmed the results of thermodynamic modeling.

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Information about the Authors / Сведения об авторах

Anatoly A. Babenko, *Dr. Sci. (Eng.), Prof., Chief Researcher, Head of the Department of Ferrous Metallurgy,* Institute of Metallurgy, Ural Branch of the Russian Academy of Sciences
ORCID: 0000-0003-0734-6162
E-mail: babenko251@gmail.com

Vladimir I. Zhuchkov, *Dr. Sci. (Eng.), Prof., Chief Researcher of the Laboratory of Steel and Ferroalloys,* Institute of Metallurgy, Ural Branch of the Russian Academy of Science
E-mail: ntm2000@mail.ru

Il'ya N. Kel', *Cand. Sci. (Eng.), Senior Researcher of the Laboratory of Steel and Ferroalloys,* Institute of Metallurgy, Ural Branch of the Russian Academy of Sciences
ORCID: 0000-0001-6411-6932
E-mail: dunnington@mail.ru

Alena G. Upolovnikova, *Cand. Sci. (Eng.), Senior Researcher of the Laboratory of Steel and Ferroalloys,* Institute of Metallurgy, Ural Branch of the Russian Academy of Sciences
ORCID: 0000-0002-6698-5565
E-mail: upol.ru@mail.ru

Ruslan R. Shartdinov, *Junior Researcher of the Laboratory of Steel and Ferroalloys,* Institute of Metallurgy, Ural Branch of the Russian Academy of Sciences
ORCID: 0000-0003-0852-1161
E-mail: rr.shartdinov@gmail.com

Анатолий Алексеевич Бабенко, *д.т.н., профессор, главный научный сотрудник, руководитель отдела черной металлургии,* Институт металлургии Уральского отделения РАН
ORCID: 0000-0003-0734-6162
E-mail: babenko251@gmail.com

Владимир Иванович Жучков, *д.т.н., профессор, главный научный сотрудник лаборатории стали и ферросплавов,* Институт металлургии Уральского отделения РАН
E-mail: ntm2000@mail.ru

Илья Николаевич Кель, *к.т.н., старший научный сотрудник лаборатории стали и ферросплавов,* Институт металлургии Уральского отделения РАН
ORCID: 0000-0001-6411-6932
E-mail: dunnington@mail.ru

Алена Геннадьевна Уполовникова, *к.т.н., старший научный сотрудник лаборатории стали и ферросплавов,* Институт металлургии Уральского отделения РАН
ORCID: 0000-0002-6698-5565
E-mail: upol.ru@mail.ru

Руслан Рафикович Шартдинов, *младший научный сотрудник лаборатории стали и ферросплавов,* Институт металлургии Уральского отделения РАН
ORCID: 0000-0003-0852-1161
E-mail: rr.shartdinov@gmail.com

Contribution of the Authors

A. A. Babenko – development and proposal of an experiment planning matrix, discussion and analysis of results, editing the text.

V. I. Zhuchkov – discussion and analysis of results, editing the text.

I. N. Kel' – thermodynamic modeling, writing the text.

A. G. Upolovnikova – data visualization, editing the text.

R. R. Shartdinov – writing the text, preparing the samples for *X*-ray phase analysis.

Вклад авторов

А. А. Бабенко – разработка и предложение матрицы планирования эксперимента, обсуждение и анализ полученных данных, редактирование текста.

В. И. Жучков – обсуждение и анализ полученных данных, редактирование текста.

И. Н. Кель – проведение термодинамического моделирования, написание текста.

А. Г. Уполовникова – визуализация данных, редактирование текста.

Р. Р. Шартдинов – написание текста, подготовка проб для рентгенофазового анализа.

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