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Assessment of the flux composition effect on the removal efficiency of non-metallic inclusions in high-chromium cast iron

Abstract: The problem to refine high-chromium cast iron melts obtained with the use of a large proportion of scraped metal and scrap is considered herein. Since fluxes containing calcium fluoride are currently considered to be environmentally polluting, it is required to reduce the use of fluorides, or completely replace them. It was shown that a decrease in the melting point of the flux can be achieved with the use of a mixture of calcium and magnesium fluorides or by the introduction of boron oxide into the composition of the fluxes. However, the efficiency of these fluxes, as well as silicocalcium additives and vacuum remelting in the high-chromium cast iron melting when a high proportion of scrap in the charge is used, has not been previously considered. In this regard, the effect of these refining methods on the removal of non-metallic inclusions in high-chromium cast iron of Grade 340X18HML was experimentally assessed. Thermodynamic calculations were performed for the interaction of magnesium and calcium fluorides with non-metallic oxide inclusions typical of high-chromium cast irons and with oxides used for neutral lining of induction furnaces. It has been shown that fluxes based on boron oxide, magnesium and calcium fluorides and their mixtures effectively remove oxide and sulfide non-metallic inclusions; however, they have a destructive effect on the lining of furnaces, significantly reducing its service life. The addition of silicocalcium reduces the content of sulfides but does not affect the content of non-metallic inclusions in the form of oxides and nitrides. Vacuum remelting effectively reduces the number of nitride inclusions.

Keywords: high chromium cast iron, HCCI, flux, calcium and magnesium fluorides, boron oxide, non-metallic inclusions

Introduction

White high-chromium cast irons are important industrial alloys used in the manufacture of parts subject to abrasive wear. Their use in the manufacture of parts for mills, crushers, pumps intended to pump pulp and other equipment determines their high demand in the mining, metallurgical and energy industries. Due to the high cost of such cast irons during production of castings from them, it is economically justified to use a high proportion of scraped metal and scrap in their melting. It results in an increase in non-metallic inclusions in castings. In industrial conditions, it leads to an increase in defects caused by cracking of castings both during cooling and during subsequent heat treatment, as well as a decrease in their wear resistance. Economic considerations, as well as the desire to increase the wear resistance of castings, justify the increasingly widespread use of high-chromium cast irons of hypereutectic composition. If non-metallic

inclusions are introduced during melting the proportion of defects increases even more since these cast irons are prone to the formation of cracks by their nature.

Studies for castings made of hypereutectic high-chromium cast irons produced under industrial conditions have shown that cracks propagate both through primary carbides $(Cr, Fe)_7C_3$ and through oxide non-metallic inclusions and pores (Panichkin et al., 2021). At the same time, castings obtained without use of a large proportion of scrap during cast iron melting have predominantly MnS and TiN inclusions. It indicates the need for effective refining of these cast iron melts. It is known (Kudryavikh, 2012) that many non-metallic inclusions are contained in the form of extremely thin formations of colloidal size in alloyed cast irons and in cast irons containing impurities of non-ferrous metals (1 cm^3 of ordinary gray cast iron can contain up to 5 million stable oxide inclusions, including about 70 % inclusions ranging in size from 0.2 to 1 micron; about 43 million sulfides; about 5 million carbonitrides). These endogenous inclusions, existing in liquid cast iron and formed during crystallization, can have a noticeable, and often decisive, effect on the physical properties of the metal, the processes of structure formation of cast iron and its properties in castings, and in the greater extent, the higher these properties are.

Treatment of cast iron with limestone reduces the contamination of alloys with non-metallic inclusions, especially sulfide ones, reduces their size, globularization and transfers them deep into grains, as well as incurs chopping and smoothing of the structure (Kolokoltsev, Shevchenko, 2011). A slag mixture is usually used in the acid melting process. This mixture consists of fireclay and glass breakage, freshly burnt ground lime and fluorspar. The authors of (Ri et al., 2015) recommend to treat chromium cast iron with slag-forming mixtures containing CaF_2, $CaCO_3$, SiO_2, C coke, CaC_2 to improve its quality. These mixtures are proposed in a crushed state (2-3 mm or less) to be poured onto the surface of the melt immediately after addition of ferroalloys. The main components of fluxes used in melting high-alloy cast irons in furnaces with a main lining are lime (CaO) and CaF. It can prevent contamination of the melt with non-metallic inclusions containing silicon oxide in contrast with melting in an acid-lined furnace.

Another source for the formation of non-metallic inclusions is the process of deoxidation of cast iron melts. Thus, deoxidation of the melt with ferrotitanium reults in contamination of cast iron with titanium carbonitrides and carbosulfonitrides. During deoxidization with aluminum or ferrotitanium containing up to 10% Al and 7% Si, dispersed compounds $nAl_2O_3mSiO_2$(aluminosilicates) that do not have time to float are formed in the structure of the castings and crumble during impact-abrasive wear (Goldstein & Mizin, 1986; Kolokoltsev et al., 2007; Taran, 1967; Orekhova. 2010;). The authors of (Ri et al., 2015) consider the use of ferrosilicon for deoxidation of cast iron unacceptable due to the formation of silicates. The authors of (Rozhkova, et al., 2005) believe that the most effective is the deoxidation of chromium cast iron with the alloy FCM-6 containing, %: $40 - 50$ Ce; $5 - 7$ Mg;$18 - 25$ La; $10 - 12$ Nd; $5 - 7$ Pr; not more than 10 of Fe. The optimal amount of FCM-6 additive was $0.2 - 0.3\%$ of the liquid metal mass. The introduction of a large amount of master alloy causes contamination, since REM and Mg, which are part of the master alloy, form complex compounds with oxygen and sulfur located in the form of a continuous front along the boundaries of eutectic chromium carbides, and form intercrystalline layers of non-metallic inclusions of REM and Mg oxides.

The study of the effect of silicocalcium on non-metallic inclusions in high-chromium cast irons is of great interest. Thus, it is known that calcium is slightly soluble in iron-based melts and does not form solid solutions with iron; this feature allows it to be used as a deoxidizing element. The formation of calcium oxides in the melt volume during deoxidation can lead to their direct interaction with dispersed silicate inclusions and their removal. At the same time, the process of interaction of silicocalcium with the melt will promote the dissolution of silicon in the melt volume, which can also interact with oxygen in the melt volume, forming silicates.

Being a part of fluxes, calcium fluoride significantly reduces the viscosity of highly basic slags Al_2O_3 - CaO-MgO-SiO_2 ($-CaF_2$) (Wu et al., 2011). However, CaF_2 has a negative effect on the refractory lining, and its effect on the physical and chemical properties of slags is short-lived due to the high volatility of environmentally harmful fluorides.

One of the ways to increase the efficiency of fluxes and reduce the release of harmful fluorides is to lower the melting point of slags by adjusting the composition of the fluxes. As follows from the information presented in Table 1, boron oxide and mixtures of calcium and magnesium fluorides have a significantly lower melting point compared to fluorspar. It allows us to predict the possibility of reducing the amount of fluorides in the composition of fluxes or completely replacing them with boron oxide.

Table 1 - Melting and boiling points of compounds considered as components of fluxes for melting wear-resistant chromium cast irons

Characteristic name	Compound				
	CaF_2	MgF_2	$CaF_2+ MgF_2$ (2/1 by wt.)	B_2O_3	CaF_2+ 14.5 wt.% CaO
Melting point,°C	1418	1263	980	480	1368
Boiling point, °C	2533	2239	-	1680	-

It is known (Babenko et al., 2020; Babenko et al., 2020) that replacing CaF_2 with boron oxide allows not only to improve the physical properties of the slag but also the environmental situation. Work (Babenko, 2020) presents the results of experimental studies of the effect of chemical composition and temperature on the viscosity of slags of the $CaO-SiO_2-Cr_2O_3$ system containing 8 % MgO, 3 % Al_2O_3 and 6 % B_2O_3. It has been established that slag containing no chromium oxide with a basicity of 1.0 has a fairly low viscosity of 0.2–0.6 Pa·s in a wide temperature range of 1200–1350 °C, due to the high concentration of low-melting phases, reaching 22% ($CaO·B_2O_3$, $2CaO·B_2O_3$ and $CaO·MgO·2SiO_2$), and only 11% refractory ($2CaO·SiO_2$, $CaO·Cr_2O_3$, $MgO·Cr_2O_3$, CaO, Cr_2O_3 and MgO). Slag with 18% Cr_2O_3 with the same basicity retains a low viscosity of 0.1–1.0 Pa·s but at a much higher temperature of 1450–1570 °C due to an increase in the content of refractory compounds to 27% and an almost unchanged amount of fusible phases (21%). It is obvious that the use of a high proportion of scraped metal and scrap in the high-chromium cast iron melting sharply increases the risk of contamination with a large amount of chromium oxide, and it requires to take measures to reduce the melt viscosity.

The purpose of this work was to assess the efficiency of the use of fluxes from calcium and magnesium fluorides and their mixtures, boron oxide and its mixture with calcium fluoride, silicocalcium additives and vacuum remelting to remove non-metallic inclusions from a melt of high-chromium cast iron with Grade 340X18HML during melting in an induction furnace with a neutral lining.

Experimental part

With the purpose to assess the possibility to use fluxes CaF_2, MgF_2, a mixture of 56 wt.% CaF_2 + 44 wt.% MgF_2 of eutectic composition, B_2O_3 and mixture 50 wt.% B_2O_3+50 wt.% CaF_2, silicocalcium with Grade SK20, a series of laboratory experiments were performed to melt wear-resistant chromium cast iron with Grade 340X18HML (Table 2). The results were compared with castings obtained from cast iron smelted without the use of flux during the melting process. The mass of the cast iron sample melted during each experiment was 1300 g, the total proportion of scrap in the charge was ~70%. The flux sample was 50 g. The silicocalcium sample was 10.3 g. The melting was carried out in a normal atmosphere, in a corundum crucible with a neutral lining of the Insetag-86XT brand (84.7 wt% Al_2O_3, 14.6 wt% MgO). The charge used was Grade 3 steel (Grade C), ferrochrome FeCr60C70LSLP, cast iron shavings, AISI210 steel scrap (manganese steel), and H-2 nickel. When the steel scrap and cast iron shavings were melted, ferrochrome was added to the melt, then nickel and AISI210 steel scrap. Next, the alloy was deliberately kept at 1500-1550 °C without protective flux in a normal atmosphere for 10 minutes to oxidize it. After that, flux was added, and the melt was kept for another 15 minutes at the specified temperatures. Melting was performed in an induction furnace UIPV-0.001. Casting was carried out in a ∅ 40 mm graphite mold. Before casting, a layer of slag was mechanically removed from the surface of the melt.

Vacuum remelting was carried out using cast iron previously melted in a normal atmosphere. Remelting was performed in a vacuum induction furnace UIPV-0.001 at residual pressure (50-100 Pa). Heating was also carried out to 1450-1500°C and it was held for 0.5 hours. The melt was cast into a ∅40 mm graphite mold.

Table 2 - Composition of casting samples made of wear-resistant chromium cast iron after refining

Alloy	Chemical composition, wt.%											
	Fe	Cr	C	Mo	Ni	Mn	Si	P	S	Ti	Ca	Mg
340X18HML	base	17-20	3.3-3.6	≤1	≤1	0.6-1.2	0.6-1	≤0.1	≤0.06	-	-	-

The size and quantity of non-metallic inclusions were studied by metallographic analysis using a Leica DM IRM inverted optical microscope and the VideoTest-Metal software package. The surface of the sections was examined without etching. The content of sulfur and carbon in cast iron after refining was determined with the use of a Bruker G4 ICARUS TF analyzer. The phosphorus content was assessed by X-ray fluorescence analysis with an Axios wave-dispersive combined spectrometer. The number and proportion of non-metallic inclusions were determined under GOST 1778-80 by method P1. The composition of nonmetallic inclusions was studied with the use of a JEOL JXA-8230 microprobe analyzer.

Thermodynamic calculations of the interaction of magnesium fluorides, calcium and boron oxide with oxides characteristic of high-chromium cast iron, including the furnace lining, were performed using the HSC Chemistry 8.14 software package. The thermodynamic calculations made indicate that fluxes based on a mixture of magnesium and calcium fluorides will not enter into a chemical reaction with the lining of an induction furnace based on $MgO*Al_2O_3$ and with the oxides Cr_2O_3, SiO_2 located in the melt volume according to the reactions considered (Figures 1-3). The disadvantage of such fluxes will be the inability to use limestone or burnt lime to desulphurize the cast iron melt due to the development of reaction 11 with the formation of magnesium oxide that will significantly increase the viscosity of the melt. Based on this, it is possible to conclude that silicon and chromium oxides dissolve in fluoride-based fluxes, and the lining of the induction furnace will also be dissolved.
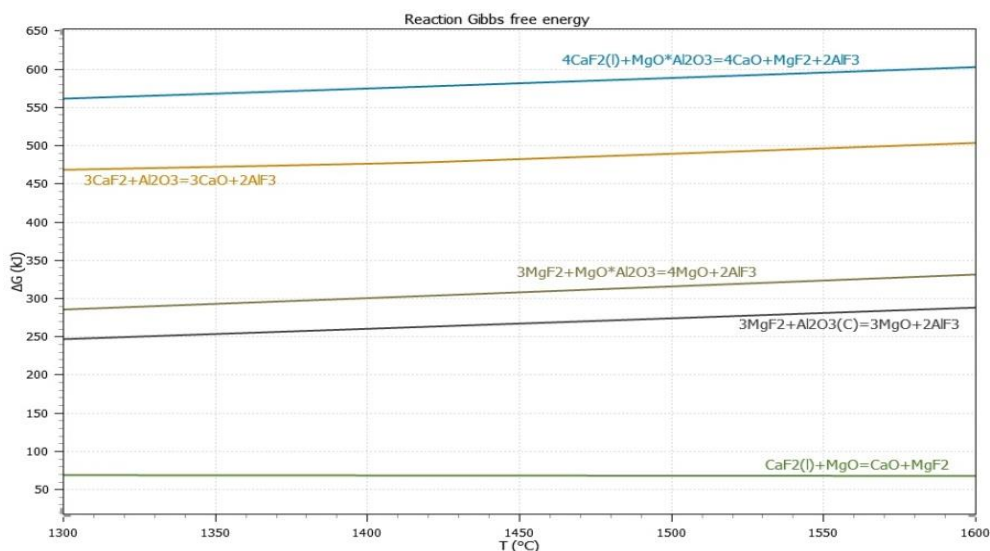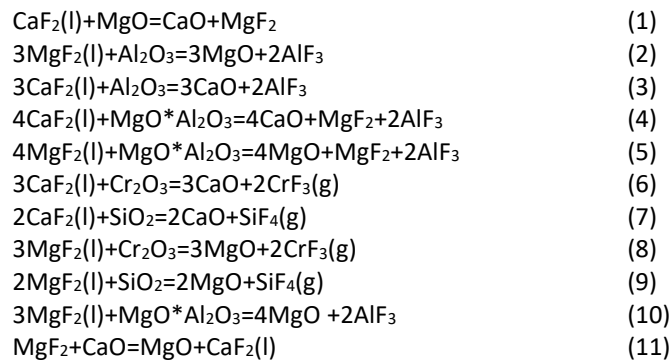
$$CaF_2(l)+MgO=CaO+MgF_2 \tag{1}$$
$$3MgF_2(l)+Al_2O_3=3MgO+2AlF_3 \tag{2}$$
$$3CaF_2(l)+Al_2O_3=3CaO+2AlF_3 \tag{3}$$
$$4CaF_2(l)+MgO*Al_2O_3=4CaO+MgF_2+2AlF_3 \tag{4}$$
$$4MgF_2(l)+MgO*Al_2O_3=4MgO+MgF_2+2AlF_3 \tag{5}$$
$$3CaF_2(l)+Cr_2O_3=3CaO+2CrF_3(g) \tag{6}$$
$$2CaF_2(l)+SiO_2=2CaO+SiF_4(g) \tag{7}$$
$$3MgF_2(l)+Cr_2O_3=3MgO+2CrF_3(g) \tag{8}$$
$$2MgF_2(l)+SiO_2=2MgO+SiF_4(g) \tag{9}$$
$$3MgF_2(l)+MgO*Al_2O_3=4MgO +2AlF_3 \tag{10}$$
$$MgF_2+CaO=MgO+CaF_2(l) \tag{11}$$

Figure 1 - Temperature dependence of the Gibbs energy of the reactions of calcium and magnesium fluorides with the furnace lining
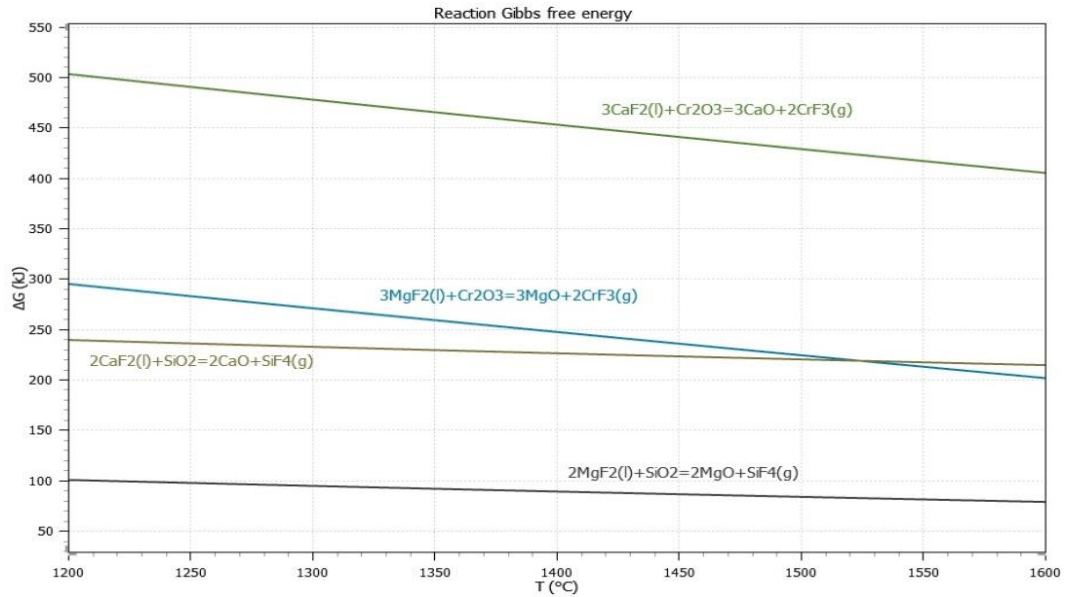
Figure 2 - Temperature dependence of the Gibbs energy of the reactions of calcium and magnesium fluorides with oxides in the melt structure
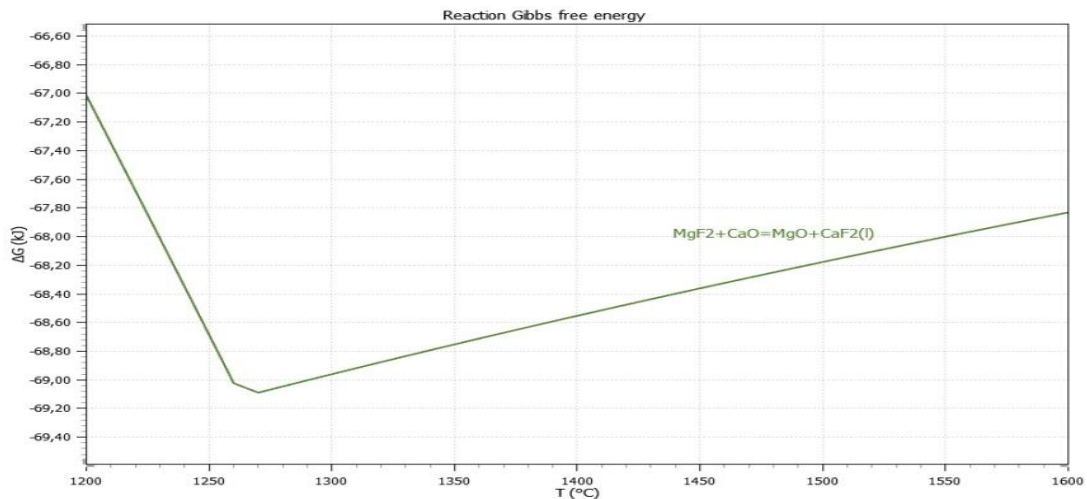

Figure 3 - Temperature dependence of the Gibbs energy of the reactions of magnesium fluoride with calcium oxide

Results and Discussion

Analysis of the composition of the alloys after refining with all types of fluxes showed a decrease in sulfur content (Table 3) compared to the alloy not treated with flux. The introduction of silicocalcium also led to a significant reduction in the sulfur content in the alloy. Vacuum remelting had little effect on the content of this element in cast iron. None of the melt processing methods used caused a decrease in the phosphorus content in it.

The metallographic analysis of the resulting castings showed a decrease in sulfide inclusions in the structure of cast irons melted using fluorides and boron oxide as fluxes and with the addition of silicocalcium (Table 3). In this case, the maximum reduction in the amount of sulfides to 0.09 vol.% occurred during use of a flux of 50 wt.% $B_2O_3+CaF_2$ and in case of introduction of silicocalcium into the melt. A good result in melt desulfurization is also achieved during use of B_2O_3 flux.

The most effective way to reduce inclusions of titanium nitrides turned out to be vacuum remelting. It ensured a reduction in their amount by ~80%. The effect of fluxes on the volume fraction of inclusions of titanium nitrides and carbonitrides in the obtained samples is significantly less, however, a decrease in their

quantity is also noted by an amount ranging from 33% for CaF_2 flux to 45% for 50 wt.% flux $B_2O_3+CaF_2$. It is explained by the fact that the flux protects against the penetration of nitrogen into the melt from the atmosphere during the melting process but does not refine it from this element.

Table 3 - Effect of refining cast iron on the content of sulfur, phosphorus and non-metallic inclusions in their structure

Flux composition	Content, wt.%		Nitrides, vol.%	Sulfides, vol.%	Oxide inclusions, vol.%
	P	S			
Alloy without flux	0.02	0.052	0.37	0.298	0.63
CaF_2	0.03	0.028	0.249	0.175	0.26
MgF_2	0.03	0.027	0.234	0.187	0.34
56 wt.% CaF_2 + 44 wt.% MgF_2	0.03	0.028	0.245	0.174	0.32
B_2O_3	0.03	0.017	0.205	0.148	0.19
50 wt.% $B_2O_3+CaF_2$	0.02	0.017	0.203	0.07	0.22
Ferroalloy SK20	0.03	0.014	0.312	0.09	0.55
Remelting in vacuum (50-100 Pa)	0.019	0.033	0.074	0.267	-

All tested fluxes clean the wear-resistant high-chromium melt well from oxide inclusions, with the best results obtained with the use of B_2O_3 flux, which reduces the number of oxide inclusions by ~70%. This is due to the lowest melting point and, accordingly, the viscosity of slags formed during interaction with oxides of chromium, iron, and silicon. Analysis of the composition of slags formed on the surface of molten cast iron during its treatment with flux 56 wt.% CaF_2 + 44 wt.% MgF_2 shows (Table 4) that it contains large amounts of oxides of iron, chromium and silicon.

Meanwhile, it has been discovered that the use of all fluoride- and oxide-based fluxes causes accelerated destruction of the furnace lining. It is due to the fact that fluxes gradually penetrate into lining material through the pores and react due to their low viscosity and good wetting of its surface. It is confirmed with the results of analyzes for the composition of the slag taken from the surface of the lining after melting cast iron using a flux of 56 wt.% CaF_2 + 44 wt.% MgF_2 (Table 4). They indicate the dissolution of aluminum oxide by flux. The least influence was found during use of CaF_2 flux. It is due to the formation of more refractory eutectics with lining components. B_2O_3 has the most significant effect on the lining; its use reduces the service life of the lining up to 2-3 heats.

Table 4 - Composition of slag after melting the wear-resistant high-chromium melt under a layer of flux of 56 wt.% CaF_2 + 44 wt.% MgF_2

Slag sampling location	Chemical composition, wt.%										
	O	F	Na	Mg	Al	Si	P	S	K	Ca	Ti
From the lining surface	17.770	22.888	0.388	12.451	6.015	5.227	0.004	0.028	0.074	12.260	0.518
	Cr	Mn	Fe	Sr	Y	Zr	Ba	Pb	Co	Ni	Zn
	0.030	0.485	0.292	0.018	0.006	0.015	0.242	0.040	-	-	-
From the surface of the melt	O	F	Na	Mg	Al	Si	P	S	K	Ca	Ti
	10.088	25.859	0.109	14.958	0.168	0.782	0.013	0.012	0.022	15.110	0.055
	Cr	Mn	Fe	Sr	Y	Zr	Ba	Pb	Co	Ni	Zn
	6.953	0.154	13.292	-	0.004	-	0.302	0.029	0.026	0.052	0.007

Thus, it has been established that replacing CaF2 flux with fluxes B2O3 and 50 wt.% B2O3 + 50 wt.% CaF2 promotes deeper purification of the melt from sulfide (by 75%) and oxide (by 70%) non-metallic inclusions. The effect of melt desulfurization from the use of flux 50 wt.% B2O3 + 50 wt.% CaF2 is comparable to modifying the alloy with silicocalcium CK30 in an amount of 1%. Fluxes CaF2, MgF2, 56 wt.% CaF2 + 44 wt.% MgF2, B2O3 and 50 wt.% B2O3 + 50 wt.% CaF2 protect the melt from dissolving nitrogen in it, but do not refine it from nitrogen impurities. Meanwhile, the use of these fluxes reduces the service life of the furnace lining. The least effect on the lining was found when using CaF2 flux; the most significant effect is exerted by B2O3, which is associated with the interaction of this compound with the components of the lining with the formation of low-melting eutectics. Vacuum remelting is an effective way to clean cast iron

from nitrogen dissolved in its volume. Based on the results obtained, we can conclude that it is necessary to search for more effective refining fluxes that provide comprehensive cleaning of high-chromium cast irons from sulfide and oxide non-metallic inclusions and have minimal impact on the furnace lining.

Conclusions

- The thermodynamic calculations made indicate that fluxes based on a mixture of magnesium and calcium fluorides enter into a chemical reaction with the lining of an induction furnace based on $MgO*Al_2O_3$ and with the oxides Cr_2O_3, SiO_2 located in the melt volume. Analysis of phase diagrams indicates that these fluxes will form melts with these oxides. The disadvantage of these fluxes is the inability to use limestone or burnt lime to desulphurize the cast iron melt.

- Replacement of CaF_2 flux with fluxes - B_2O_3 and 50 wt.% B_2O_3+50 wt.% CaF_2 promotes deeper purification of the melt from sulfide (~75%) and oxide (~70%) non-metallic inclusions. The effect of melt desulfurization from the use of flux 50 wt.% B_2O_3+50 wt.% CaF_2 is comparable to alloying of the alloy with silicocalcium CK30 in an amount of 1%.

- Fluxes CaF_2, MgF_2, 56 wt.% CaF_2 + 44 wt.% MgF_2, B_2O_3 and 50 wt.% B_2O_3+50 wt.% CaF_2 protect the melt from dissolution of nitrogen in it but does not refine it from nitrogen impurities.

- The use of fluxes based on CaF_2, MgF_2 and B_2O_3 and their mixtures shortens the service life of the furnace lining due to active dissolution. The least effect on the lining was found during the use of CaF_2 flux; B_2O_3 had the most significant effect on the lining.

- Vacuum melting at 50-100 Pa for 30 minutes is effective to remove nitrogen from a melt of high-chromium cast iron but it does not have a significant effect on the sulfur content in the alloy.

Conflict of interests. The corresponding author declares that there is no conflict of interest on behalf of all authors.

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