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Petrographic research of metallurgical processes influence on durability of induction furnaces lining

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Abstract. One of the causes of corrosion and destruction of refractory lining in pyrometallurgical processes is the occurrence of melts in the most intensively altered zones of refractory material. This phenomenon has a direct analogy with the process of interaction of magma with crystalline rocks of various composition and the contact changes manifested in high-temperature metamorphism. Silicate, oxide (wustite and magnetite) and metallic melts which are produced resulting from mutual components diffusion in metallurgical processes are analogous with natural magmas. The formed minerals with complex isomorphism such as olivine, spinel, perovskite-brownmillerite have the analogues in natural objects as well. The petrographic studies applied in technical petrography (the petrography section) are used to investigate the artificial stone materials: concrete, cement, building bricks, ceramics, crystalline glass and glasses, slags, refractories, abrasives, ore agglomerates, etc. They allow to establish the structural and phase transformations in the lining during its operation and to draw a conclusion about its durability increase or decrease. This enables the creation of new lining materials that would allow to do alloy smelting in induction crucible furnace at mains frequency at higher temperatures.

1. Introduction

In the modern economy it is required to obtain simultaneously inexpensive and high-quality billets with a low level of rejection to improve the competitiveness of finished products. The cost of castings is influenced both by the chosen melting unit and by the materials used for its lining.

The induction crucible furnace of industrial frequency (ICHT) *at mains frequency* belongs to the basic production assets of the foundry and impacts directly on the level of production costs and labour productivity. Consequently, it affects the profit and profitability, and thus it is the main factor determining the content of development strategies of the enterprise as a whole.

Therefore, increasing the melting unit reliability is one of the major tasks that ensure the necessary degree of fixed production assets reproduction to comply with the following parameters of the specified equipment: versatility in alloy smelting, productivity, high lining stability (resistance), the usage of domestic manufacturers' materials, energy efficiency and maintenance costs.

The melting furnaces make it possible to obtain iron castings of higher quality in comparison with the cupola furnaces as they do not require the coke use which adds sulfur and phosphorus in the melt. Their utilization is particularly effective for synthetic cast iron production as it allows using up to 30% of steel scrap in the metal stock, which reduces the production cost.



For lining these furnaces, the acid lining masses based on quartzite are used as cheapest, which ensures its resistance to 300-350 smelts under the melting temperature conditions not exceeding 1450 °C.

2. Relevance.

Currently in the territory of the Russian Federation and the neighboring countries there is mainly the accumulation of light weight steel scrap. Cast iron scrap practically disappeared while the production of cast and pig iron, on the one hand, decreased, but on the other hand - sharply increased in price due to transportation.

For this reason, at many foundries which employ a normal-frequency induction crucible furnace for the synthetic cast iron smelting as a melting unit, the proportion of steel scrap in the metal stock made 90-100%. Thus, the melting temperature regime increased to 1550-1600 ° C and the resistance of the acid lining started declining significantly.

The main Russian material for acid lining which is used by the majority of the foundries (as the cheapest of all quartzites offered on the market) is the Pervouralsk quartzite and the lining resistance on its basis is 200-250 melts.

This results in downtime increase associated with the furnaces relining, the reduction in the castings output and the increase in the cost of materials, electric power and wages which ultimately has a significant impact on the production cost. Using the basic or neutral linings only worsens the situation as they are 6-8 times more expensive and their durability does not exceed 100-150 melts. During the furnace operation the quartzite undergoes various phase transformations which affect the lining resistance in different ways. The previous studies regarding these transformations have led to the conclusion that the composition of the traditional lining mass (quartzite and boric acid) does allow one to produce the lining that can withstand the melting point of 1550-1600°C for a long time [1]. This was the reason for searching the options to use acid lining based on Pervouralsk quartzite by increasing its durability (resistance) due to the introduction of various additives. The solution to this problem allows mastering the technology of the synthetic cast iron smelting in the normal-frequency induction crucible furnaces (ICHT) at a higher temperature using only the steel scrap in the charge [2]. This reduces production costs and increases the loading of the existing melting equipment [3].

3. Experimental study.

The proposed lining composition for the study differs from the traditional as electrocorundum 0125 and 0315 is added there, considering that according to the Al₂O₃-SiO₂ state diagram while in operation under the action of high temperatures, hercynite-FeO * Al₂O₃ (FeAl₂O₄), mullite - 3Al₂O₃ * 2SiO₂ (Al₆Si₂O₁₅) and aluminum silicate Al₂O₃ * SiO₂ will be formed with a melting point of 2050, 1910 and 1860 ° C, respectively.

They should increase the chemical resistance of acid lining and reduce the penetration of oxide melts of the FeO-MnO-SiO₂ system into its working layer [4].

Belyankin D.S. was the first who reported on the processes occurring in acid lining. He provided the data that in 1939 A. T. Zhak and B. V. Ivanov investigated the spent, acid lining of the high-frequency induction crucible furnace. The quartzite charge (mixture) of different granulometry with the addition of boric acid was used for the lining manufacture. The investigated sample consisted of three zones:

1. unconverted (unreturned) (facing the coil) of black colour;
2. transition of chestnut-brown colour;
3. light green, facing the working space of the crucible.

In the unconverted zone the quartz grains to 6 mm in diameter were clearly distinguished. Its chemical composition was as follows (in %): SiO₂ - 72.48; Al₂O₃ - 9.07; Fe₂O₃ - 4.60; Cr₂O₃ - 0.58; MnO = 9.84; CaO = 0.69; MgO: 1.65. Under the microscope, the quartz grains as well as the cement from the brown glass were distinguished. The quartz grains were cracked. In the brown glass there were

the characteristic magnetite dendrites. Green ferruginous pyroxene was often crystallized at the boundary of the vitreous phase with magnetite.

The light green, hot zone directly entering the working space consisted mainly of isotropic, metastable cristobalite. Occasionally among the cristobalite sections, single quartzite grains were found. The microstructure of this zone is shown in Figure 1.

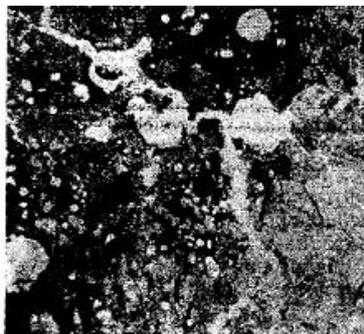


Figure 1. A microstructure of the quartz lining hot zone of a high-frequency induction furnace

Based on today's knowledge of quartz lining manufacturing and operation technology, we can make a conclusion that the study sample was taken from the lining which underwent the unacceptable changes in the unconverted (unreturned, unreduced) zone (the presence of the vitreous phase) that disrupts the experimental integrity.

The formation of 3 zones in the lining is the indispensable condition which characterizes its durability. Each zone consists of different phases formed under the influence of physical and chemical processes occurring during melting. Such processes can include: oxidation, alloying, desulfurization, deoxidation, modification, carburization, degassing and removal of nonmetallic inclusions [5]. The listed processes take place in a complex system when interacting with several phases of different physicochemical nature. Each phase characterizes the lining properties and gives the information about the correct (exact) application of new material in its composition.

The research was carried out at the enterprise operating the ICHT-10 melting furnaces. The following technological process of test work under production conditions was adopted (accepted) [6]:

- Drying of crude quartzite in the DLC furnace 15.15.10 / 12 for 20 hours.
- Stirring of all components in the grinding runners of the mod.12221 for 1 hour.
- Lining packing and its sintering according to the existing technology.
- Exposure at the temperature of 1550-1600 ° C for 2 hours.
- Draining of the first 5 melts with the volume not exceeding 0.3 volume of the crucible furnace into the molds.

The further work of the melting furnace was carried out in compliance with the existing technological process for producing castings from gray cast iron. The furnace was stopped for relining after 305 melting. The lining sample was taken when knocking out of it, on which the layers were clearly traced: a slag layer which was in direct contact with the liquid melt, sintered, semi-sintered, and free-flowing layer which preserved the initial state (Fig. 2).

A polished section was made from this sample (Fig. 3) in accordance with the requirements for conducting the petrographic analysis [7].



Figure 2. A sample of broken lining: 1 – a semi-sintered layer, 2 – a sintered layer, 3 – a slag layer

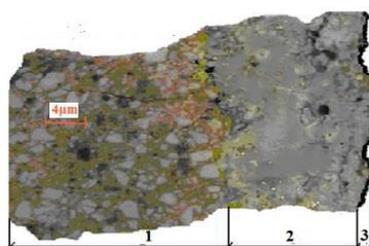


Figure 3. A polished section sample for investigation: 1 – a semi-sintered layer, 2 – a sintered layer, 3 – a slag layer

The analytical complex of the scanning electron microscope “VEGA II LMU” Tescan, Czech Republic combined with the energy-dispersive X-ray spectrometer of the model INCA Energy 350 (Fig. 4) was used for the study. The maximum size of the polished section is $20 \times 20 \times 5$ mm when tested in the TESCAN microscope. The minimum size is limited by the structural and texture inhomogeneities of the investigated surface. The study on the sample surface was carried out according to the technique which assumes the individual approach to setting the accelerating voltage, operating distance, probe current values and the selection of the diaphragm size that ensures the image optimization when describing the structural heterogeneities of rocks [8].



Figure 4. The analytical complex of the scanning electron microscope “VEGA II LMU” Tescan, Czech Republic combined with the energy-dispersive X-ray spectrometer of the model INCA Energy 350

The phase composition was investigated in a scanning electron microscope using a back scattered (reflected) electron detector (BSE detector). The obtained image enables one to distinguish mineral phases by atomic weight with the help of the BSE-detector. The heterogeneities of the composition in this image have clear boundaries while the lighter areas determine the presence of sites with heavy elements from which the electron beam is reflected better than from the mineral phases consisting of light elements.

The chemical analysis was carried out by the X-ray spectrometry method which is a set of spectra images with the chemical analysis data being reflected in the photos in the BSE mode of checkpoint data set.

For X-ray diffraction analysis a X'PERT PRO X-ray diffractometer manufactured by PANalytical was used for research and analytical control in the industry. A wide variety of possible configurations and additional devices ensure the versatility of this device when solving a wide range of problems of X-ray powder diffraction (diffractometry). The radiographs are taken with a step of about 0.02 in the interval of 9-81 degrees. 2θ with a rotation of 30 rpm and the delay of 0.1 seconds at a point, the working radius of 141 mm, a tube with a copper anode.

The study sites were accompanied by getting their photos on which the microprobe points were marked (the determination of the elemental phase composition). Figure 5 shows the photos of the sites 4.1 (zone 3) and 3.2 (zone 2).

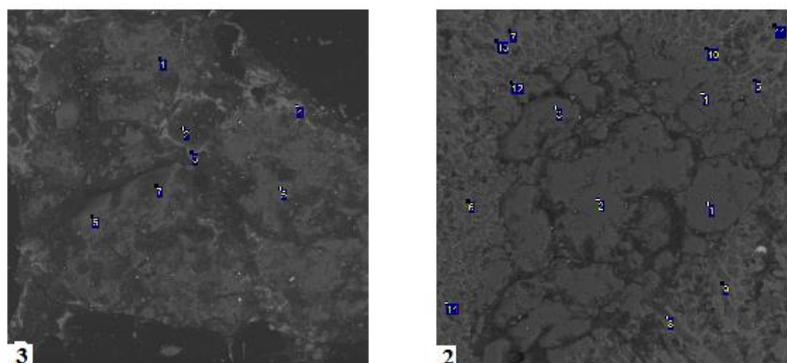


Figure 5. The fragments of zones 3 and 2 in sections 4.1 and 3.2, microprobe analysis of phases: zone 3 - points 1, 5-7 cristobalite; point 2 - epoxy resin; point 3 - Bustamite; zone 2 - points 1-8 - phases of SiO₂; points 9-14 – glass

As a result of electron-microscopic and X-ray phase studies in zone 3 apart from quartzite the synthetic phases have been found which correspond to such mineral formations as quartz, cristobalite, bustamite – (Mn,Ca)₃[Si₃O₉], melilite – (Ca,Na)₂(Al,Mg,Fe)[(Si,Al)₂O₇], anorthite, canoite – (Mn,Mg)₂[Si₂O₆], clinoferrosilite - Fe₂(Si₂O₆), olivine of tephroite-fayalite composition, vonsenite, magnesioferrit, magnetite, hematite, native iron and non-structured glassy phases.

In zone 2 except quartz, crystallite, bustamite, melilite (akermanite), clinoferrosilite, magnetite and native iron noted in zone 3, the phases of the predominant tridymite in the second zone as well as the admixtures of sassolite, dumortierite, canoeite, mullite, glass, magnetite, native iron, relict rutile, anatase and corundum are met. Processing the X-ray phase analysis data of the zones' material and the microprobe analysis of the mineral phases composing the zones made it possible to calculate the total chemical composition of zones 3 and 2 separately and compare it with the initial quartzite (Table 1).

Table 1. Chemical composition of the polished section of zones 3 and 2 and initial quartzite of the Pervouralsk field

Lining zone	Chemical composition, in % by weight											
	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃ + FeO	Cr ₂ O ₃	B ₂ O ₃	MgO	MnO	CaO	K ₂ O	Na ₂ O	P ₂ O ₅
Zone-3 (slagged)	60,07	-	0,76	31,0	0,2	0,1	3,90	3,5	0,22	-	-	-
Zone-2 (sintered)	88,1	1,33	6,47	1,55	0,2	≤0,1	0,3	0,4	0,03	1,23	0,39	-
Pervouralsk quartzite	99,0- 99,4	0,01- 0,09	0,5- 0,86	0,15- 0,4	-	≤0,1	0,01- 0,02	-	0,01- 0,3	-	-	0,015- 0,025

4. Conclusion.

The analysis of the petrographic and chemical analysis results indicates the complex effect of various physical and chemical processes (chemical reactions, formation of solutions, wetting and impregnation) on the structural-phase transformation and wear of the lining. During the melting process, the charge components (Fe, Mn, Si, Cr, etc.) are oxidized and they actively interact with the silica of the quartzite lining to form silicates [9].

Iron and manganese silicates form an acidic slag system FeO - MnO - SiO₂, in which it dissolves to 48-50% SiO₂ at the temperature of 1500 – 16500° C. The micro impurities Al, Ca, Cr, Na and Mg enter the metal melt from the charge and oxidized switch to the oxide melt reducing the melting temperature and the viscosity of the liquid slag.

It is known that corundum, magnesium oxide and spinel-type compounds (RO•Al₂O₃) are poorly wetted with oxide melts if compared with the quartzite and are not wetted with iron-based metal melts.

For this reason, the formation of mullite (3Al₂O₃ • 2SiO₂) contributes to low volumetric expansion at high temperature as the silicon oxide being the part of the dry lining mass passes into a bound state after sintering. When melting, the metal comes into the contact only with the mullite layer; that is why the silicon penetration into the metal is impossible. The spinel-forming mass from granite, magnetite, clinoferrrosilite, magnesioferrite and hematite discovered as the analysis' result ensures a low coefficient of volumetric expansion resulting in a significant decrease in the formation of lining microcracks as it cools and increases the resistance to chemical attacks. In addition, these phases reduce the penetration of oxide melts of the FeO-MnO-SiO₂ system into the working, sintered lining layer.

Thus, with the help of the petrographic analysis it was revealed that the use of electrocorundum in the lining material composition gave a positive effect. The lining sustained a long-term operation at a temperature regime of 1550-1600 ° C providing resistance to 305 melts. It allows melting synthetic pig iron from a metal scrap consisting of 90-95% of steel scrap which significantly reduces the production cost. Apart from that, the possibility of such synthetic pig iron smelting allows one to ensure the loading capacities of the foundries equipped only with the smelting furnaces.

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