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Corrosion testing of prospective chrome refractory materials in borosilicate glass melts

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Justification is provided of significant practical relevance of the issue of corrosion and erosion resistance of refractory materials in furnaces with direct electric heating that are used for vitrification of high-level waste. Main promising materials are listed, and an algorithm is given for assessing their resistance to attack of borosilicate glass melts under operating conditions of electric furnaces. Corrosion testing of chrome refractory materials of joint Chinese and Belgian origin has been carried out under static and dynamic conditions in a low-melting borosilicate glass melt and in a borosilicate glass melt containing simulated liquid high-level waste (HLW). A search has been conducted among chrome refractory materials of Chinese and Belgian origin for ones demonstrating the highest glass melt attack resistance with regard to the conditions of HLW solidification. The results of these tests will be considered in the design of removable and small-scale melters when selecting the material for melter lining.

keywords: refractory materials, corrosion, resistance to glass attack, chrome refractories, industrial vitrification furnaces, reprocessing of high-level waste (HLW), borosilicate glass, thermoviscosimetric characteristics, static tests, dynamic tests

1. Introduction

For several decades already, radiochemical production dedicated to the extraction reprocessing of spent nuclear fuel from VVER-440, BN-600, naval propulsion, and research reactors has been operating at Mayak PA. High-level solutions containing fuel fission and fuel activation products as well as structural materials are the most hazardous type of waste generated in the course of this reprocessing.

A new vitrification complex, at which the specified waste will be solidified in borosilicate glass, is scheduled to be put into operation approximately in 2027. The developed concept of the new vitrification complex envisages the use (as a part of the complex) of a removable small-scale melter with direct electric heating and a productivity of 20 L/h in terms of evaporated HLW solution [1]. The melter is designed for the melting of borosilicate glass, which has several advantages as compared to aluminophosphate glass: significantly higher retaining capacity in terms of fission products, resistance to chemical attack and radiation damage, and resistance to decrystallization [2]. At the same time, borosilicate glass has a higher melting temperature (ranging from 1100 °C to 1200 °C).

To create such a promising facility for HLW solidification that will fulfil the requirements of reliability and productivity, there is a need for the relevant structural materials that will come in direct contact with glass melts while meeting the pertinent criteria of resistance to high temperatures and corrosive components of glass melts for extended periods of time.

As a rule, lifetime of a furnace is limited not by a general poor condition of the entire refractory brickwork, but by failure (often as a result of some

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accident) of a limited number of structural elements of the melting chamber and plenum in the melting zone and zones of maximum temperatures [3]. For example, the refractory corrosion rate at the level of molten glass can be two to five times higher than that for the entire area of the refractory brickwork that is located below the melt surface [4]. In HLW vitrification furnaces, these structural elements include, first of all, the upper section of the melting chamber walls. A significant contribution is also made by interjoint corrosion caused by exceeding the normative gaps in the brickwork.

Despite a considerable amount of laboratory studies to date on the resistance of refractories to glass attack in general, the number of studies on the corrosion attack of glass melts containing HLW and, therefore, characterized by significant peculiarities is incomparably small.

Due to the difficult geopolitical situation and the imposition of sanctions on Russian organizations by many states, the range of available refractory materials has decreased. In most cases, this means that products of such global market leaders as SEFPRO (France) or RHI (Italy), in the product lines of which there are refractory materials with good resistance to glass attack, including chrome refractories (ZIRCHROM, SUPRAL, DIDURITAL, etc.), are no longer commercially available. A range of domestic producers of similar materials is limited to the Bakor Science and Technology Center (LLC). In this regard, Chinese-manufactured products available on the Russian market are of interest, chrome refractories produced by the Zhengzhou Yandong Refractories Co. (a joint Chinese-Belgian manufacturer), in particular.

The aim of this work was to search for refractories of Chinese origin that demonstrate the best resistance in borosilicate glass melts as applied to HLW solidification conditions. For this purpose, a number of modern materials with several glass-forming compositions and different content of corrosive components were tested, parameters of corrosion resistance (corrosion rate, behavior and degree of surface coverage) of the tested samples were compared, and refractories with the best characteristics suitable for the creation of new-generation melters were preliminary selected.

2. Experimental and methodology

Four different grades of chrome refractory materials were chosen to investigate corrosion and erosion characteristics.

Refractory products used in the design of glass melting furnaces are classified as follows:

– by their chemical composition: aluminosilicate (chamotte, high-alumina, mullite), siliceous (dinas, quartz), corundum, baddeleyite-corundum (bakor), chrome-bearing (chrome), and magnesite (with additives of Al_2O_3, ZrO_2, and Cr_2O_3);

– by the method of manufacturing: ceramic (produced by sintering) and fused (produced by casting of melts).

The following types of refractories are used in components of domestic glass melting furnaces involved in the manufacturing of staple glass products:

– bakor: melting chamber, flow channel, and other crucial elements; bakor inlet ports, elements of the hearth walls;

– dinas: main crown, elements of the hearth walls;

– bakor and corundum: through channels, forehearths, gob-forming components;

– magnesite: regenerator checkers (upper rows) [5].

Ramming plastic masses and refractory concrete are also used for lining a furnace crown and walls that are not in contact with melts [6].

Melting chambers of glass making furnaces, including Mayak industrial vitrification furnaces of the EP type, are currently laid out mainly of baddeleyite-corundum (bakor) refractory bars. Bakor is chemically close to the eutectic region of the $Al_2O_3 – ZrO_2 – SiO_2$ ternary system [6].

At the same time, in some cases chrome refractories are used in the design of melting chambers of glass melting furnaces. For example, the melting chamber of the VEK small-scale melter (Karlsruhe, Germany), which was developed and designed for the vitrification of liquid radioactive waste, is made of a similar refractory material of the ER 2161 type (SEFPRO, France) with Al_2O_3, Cr_2O_3, and Zr_2O_3 being the major components of it [7].

In this work, chrome refractories of joint Chinese-Belgian origin were investigated as representing an alternative of currently available French analogues. Table 1 contains a list of samples and their chemical composition (by main components). The provided materials were studied in the form of square bars with a side of about 10 mm and a length of 50 to 120 mm.

Table 1 – Chemical composition of refractories (by main components).

Sample No.	Chemical composition, %				
	Al_2O_3	Cr_2O_3	ZrO_2	Na_2O	TiO_2
1	26.0	50.0	13.0	–	–
2	–	94.0	–	–	3.8
3	39.0	30.0	–	16.0	–
4	40.0	30.0	–	16.0	–

Performance of ceramic chrome refractories depends not only on their chemical composition, but also on several other characteristics determined by the manufacturing conditions, in particular, by their apparent porosity. It should be noted that apparent porosity of refractory material No. 1, 2, 4 is 18.0 %, whereas that of refractory material No. 3 is 20.0 %, that will potentially be manifested in the difference of their corrosion resistance.

Two different types of glass were selected as corrosive media for testing the refractory materials. Tables 2 and 3 present mass compositions of the selected glasses expressed as oxides.

Table 2 – Composition of glass No. 1 used in tests as a corrosive medium and proportions of reagents used for its preparation.

Oxides	Content, %
CaO	3.4
Al_2O_3	2.93
MgO	0.42
SiO_2	46.39
Na_2O	17.31
B_2O_3	15.28
Components of simulated HLW	
SrO	1.63
ZrO_2	2.27
MoO_3	2.04
Cs_2O	1.04
La_2O_3	0.61
CeO_2	2.62
Nd_2O_3	2.09
NiO	0.26
Cr_2O_3	0.38
Fe_2O_3	1.31
Total	100.0

Table 3 – Composition of glass No. 2 used in tests as a corrosive medium and proportions of reagents used for its preparation.

Oxides	Content, %
CaO	4.53
Al_2O_3	3.90
MgO	0.56
SiO_2	44.53
Na_2O	25.12
B_2O_3	21.36
Total	100

Viscosity is one of the major factors that determines melt corrosivity, which affects refractories. As a result, when corrosion resistance of materials is studied in glass melts with different characteristics of the viscosity-temperature dependence, erroneous conclusions can be drawn about the corrosivity of certain glass components, which, in fact, affect only melt viscosity and have merely some indirect impact on the corrosion rate in the examined melt. The difference in melt viscosities should be taken into account to avoid such errors.

In order to exclude the influence of difference in viscosity of different glass compositions on corrosion rate in them, working test temperatures were selected for each of the compositions in this study, at which their viscosity was the same. Such a temperature selection became possible with the help of thermoviscosimetric characterization of melts of each glass type using the curve defined as follows:

$$\eta = f(T), \quad (1)$$

where η is the dynamic viscosity of the melt, dPa · s; T is the temperature of the melt, °C.

Glass viscosity was examined using a laboratory oscillatory viscometer VIS 403, TA Instruments, the principle of operation of which is based on the dependence of the amplitude of forced oscillations of the rod on the viscosity of the liquid, where these oscillations occur.

Characteristics obtained for the glasses used in the work are given in Figure 1 in the temperature-viscosity coordinates for the range of viscosity values of up to 500 dPa · s with an indication of the working temperatures determined for each glass composition. Tests in borosilicate glass melts were carried out under "stringent" conditions at a melt viscosity of 10 dPa · s.

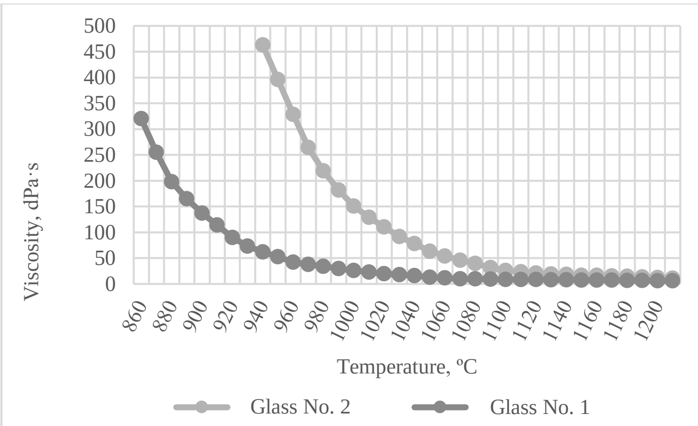

Figure 1 Thermoviscosimetric characteristics of glass melts with compositions No. 1 and No. 2 at a viscosity of up to 500 dPa · s.

This value of the viscosity of the melts was chosen based on the technological features of the process and is optimal for initiating the discharge of the glass melt from the melting agent intended for vitrification of HLW, since induction heating is used to drain the borosilicate glass melt.

To determine temperatures at which glass melts have the viscosity values indicated in Figure 1, straight lines were drawn parallel to the *x*-axis and, at the points of intersection of these lines with each of the graphs, working test temperatures were determined at the selected viscosity values for each of the compositions. Low-melting glass No. 1 reaches a viscosity value of 10 dPa · s at a temperature of 1100 °C. High-melting glass No. 2 attains the mentioned value at 1200 °C.

Therefore, samples were tested with melts of various compositions at a constant viscosity but at different temperatures, and this is what distinguishes the present investigation from other studies with similar topics, during which tests were carried out at the same temperature. Thus, viscosity, as well as temperature, is the main factor of degradation in such experimental tests. This is due to the fact that the viscosity of the glass depends on temperature and vice versa.

Table 4 summarizes working test temperatures determined for each of the used compositions.

Table 4 shows that the difference in test temperatures is significant and can appreciably affect the corrosion rate that is exponentially dependent on temperature as per the Arrhenius equation [4]:

$$k = A \cdot e^{-\frac{E_a}{R \cdot T}}, \quad (2)$$

where k is the reaction rate constant; e is the base of the natural logarithm; E_a is the activation energy, J/mol; R is the universal gas constant, 8.31 J/(mol · K); T is the temperature, K; A is the proportionality coefficient.

Tests were conducted in accordance with the technique developed by the National Glass Institute that consisted of determining of material mass or volume loss per unit of time, during which the refractory was in contact with the glass melt [8].

Table 4 – Conditions of corrosion tests with glass compositions No. 1 and No. 2.

Conditions	Glass composition No.	
	1	2
Temperature, °C	1100	1200
Viscosity, dPa · s	10	
Test time, h	100	

Resistance of refractory materials to glass attack can be characterized in detail only following a comprehensive analysis of this characteristic under static and dynamic conditions [9].

Under static conditions, samples of refractories are subject to maximum corrosion at the melt level. This fact determines the pattern of corrosion attack during the operation of refractories in industrial glass melting furnaces. Tests under static conditions were carried out in alundum crucibles by partial immersion of sample refractories in a glass melt to a depth of 10 mm, where they were kept for 100 h. In this work, all four types of refractory materials (see Table 1) with all glass melt compositions (see Tables 2 and 3) were tested under static conditions. The main component of the alundum crucibles is Al_2O_3. To assess its effect on the composition of the corrosive medium during the experiment and on the results in general, control studies were conducted that showed that the effect of Al_2O_3, which enters the glass melt as a result of crucible corrosion, is insignificant.

The dynamic method of testing simulates stringent operating conditions of refractories in the overflow area of a glass melting furnace, where refractory failure is caused both by chemical and erosion attack of the glass melt.

Tests under dynamic conditions were carried out in a crucible by rotating the sample refractories immerged in a glass melt to a depth of 20 mm around a longitudinal vertical axis for 100 h. A sample rotation rate of 60 rev/min corresponded to a velocity of 2.8 m/min, with which molten glass moves in the EP-500 furnace [10, 11].

The sample refractories were mechanically withdrawn from the glass melt when the glass was in a molten state. A layer of glaze formed on the samples after their cooling and hardening was removed with a help of nitric acid with a concentration of 4 mol/L. Using visual inspection, the extent of glass removal was evaluated.

Carrying out of these tests required an assembly of an experimental setup consisting of the following basic elements:

– operation-programmable shaft-type furnace;

– holders of sample refractories;

– two mixers (equipped with a hollow rotor shaft) with a feature of adjusting their rotation speed in the range from 40 to 2500 rev/min. The furnace is located in an exhaust hood, while the mixers are taken out of the exhaust hood, as the maximum allowable operating temperature of the mixers is + 35 °C.

Only two mixers could be placed above the furnace due to limited space. Therefore, only two samples were tested simultaneously under dynamic conditions, and two samples with the same composition were tested under static conditions.

Figure 2 demonstrates the setup layout.

During corrosion tests, linear erosion rate at the level of glass melt (mm/day) was determined. Sample cross-sections were also measured approximately at the immersion mid-depth.

The mass loss (Δm) was identified for all examined samples and the following parameters were calculated:

– initial area of the sample refractory contact with the glass melt;

– volume of the sample part immersed in the melt;

– value of this volume loss.

Using the obtained data, a value of mass loss of the samples per sample-melt contact area ($\Delta M/S$(mg/cm^2)) and a percentage value of relative volume loss were calculated. The mass of the samples was determined by weighing with the help of a technical balance (results were accurate to three decimal places). The contact area was calculated on the basis of the results of linear measurements (results were accurate to 0.1 mm).

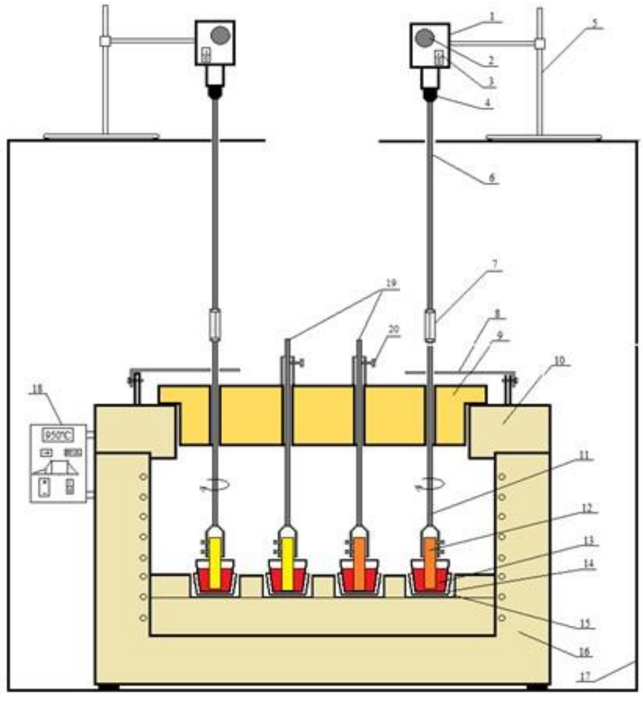

Figure 2 A layout of the setup for corrosion testing of refractories under static and dynamic conditions: 1 – mixer, 2 – rotation speed regulator, 3 – mixer switch, 4 – mixer chuck, 5 – rack, 6 – shaft, 7 – connecting nut with lock nuts, 8 – removable guard plate, 9 – removable apertured refractory insert, 10 – furnace refractory cover, 11 – test sample refractory holder (for dynamic conditions), 12 – test sample refractory, 13 – operating crucible with glass melt , 14 – guard crucible, 15 – recess in the furnace floor for crucibles, 16 – shaft-type furnace, 17 – exhaust hood, 18 – controller for programming the furnace operation conditions, 19 – test sample refractory holder (for static conditions), 20 – clamp.

Refractory corrosion rate at the level of the glass melt and at the level of the immersion mid-depth under static conditions was calculated by the formula:

$$v_c = (d_{av} - d'_{av}) \cdot 24/(2 \cdot \tau), \quad (3)$$

where d_{av} is the average thickness of the sample at the glass melt level before testing, mm; d'_{av} is the average thickness of the sample at the glass melt level after testing, mm; τ is the test duration, h; 2 is the coefficient taking into account the sample two-sided erosion.

Refractory volumetric corrosion (ΔV, vol. %) under dynamic conditions was found using a volume change of the sample part immersed in the melt over the testing period:

$$\Delta V = \frac{(V - V')}{V} \cdot 100, \quad (4)$$

where V is the volume of the immersed sample part before testing, cm^3; V' is the volume of the immersed sample part after testing, cm^3;

$$V = a \cdot b \cdot h, \quad (5)$$

where a, b are the sides of the refractory of rectangular cross-section before testing, cm; h is the depth of the sample refractory immersion in the glass melt, cm;

$$V' = V - \frac{M_0 - M}{\rho_0}, \quad (6)$$

where M_0 is the sample mass before testing, g; M is the sample refractory mass after testing, g; ρ_0 is the density of the refractory sample before testing, g/cm^3.

Additionally, a value of the specific mass loss of the samples per sample-melt contact area $\Delta M/S$(mg/cm^2) was calculated.

3. Results and discussion

3.1. Tests under static conditions

Figure 3 shows values of linear corrosion rate for materials with compositions No. 1 and No. 2 tested under static conditions.

Figure 4 demonstrates values of specific mass loss for the materials with compositions No. 1 and No. 2 tested under static conditions.

No visible traces of corrosion were found on samples No. 1 and No. 2, even along the glass melt boundary. Sample No. 2 demonstrated maximum corrosion resistance in both melts that is associated with the highest

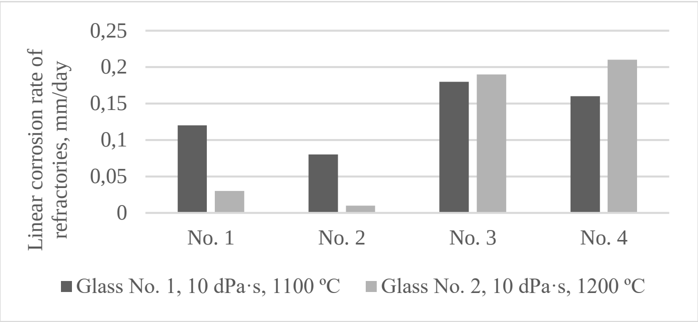

Figure 3 Linear corrosion rate of refractories, mm/day.

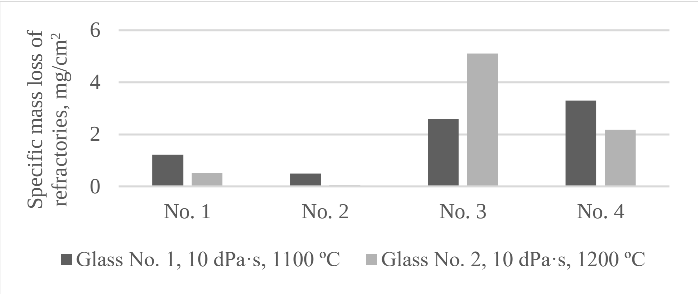

Figure 4 Specific mass loss of refractories, mg/cm².

content of chromium oxide in this sample.

Linear corrosion rates obtained for the refractories of Chinese origin were compared with linear corrosion rates of the Bk-41 refractory manufactured by Podolsk Refractories JSC (0.13 mm/day for glass composition No. 1 and 0.15 mm/day for glass composition No. 2) [12], which was tested in the same borosilicate glass compositions. The comparison showed that the linear corrosion rate of the Bk-41 refractory is comparable to the linear corrosion rate of refractories No. 3 and No. 4.

A similar conclusion can be drawn in relation to the specific mass loss of Chinese-made refractories No. 3 and No. 4: it is comparable to the specific mass loss of the Bk-41 refractory manufactured by Podolsk Refractories JSC (3.72 mg/cm² for glass composition No.1 and 2.96 mg/cm² for glass composition No. 2) [12].

Due to a wide variety of the examined refractories, the provided results do not allow gaining an unambiguous understanding of the impact of glass composition and glass test temperature on corrosion damage of materials. At the same time, the high corrosive activity of the glass melt with composition No. 1 can be explained by the presence in it of such corrosive components as iron, chrome, and nickel.

An increasing order of indices of relative (as compared to the Bk-41 refractory manufactured by Podolsk Refractories JSC) [12]) corrosion resistance of the tested materials calculated using average values of linear corrosion (a) and specific mass loss (b) is given in Figure 5.

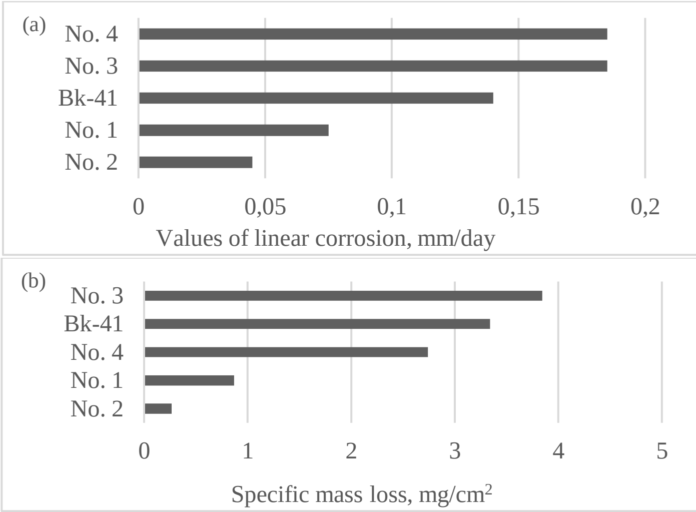

Figure 5 Indices of relative corrosion resistance of the tested materials calculated using: (a) – values of linear corrosion, mm/day; (b) – specific mass loss, mg/cm².

The displayed histograms show that refractory No. 2 demonstrated higher corrosion resistance compared to the Bk-41 refractory manufactured by Podolsk Refractories JSC [12]. Refractory No. 1 demonstrated somewhat lower resistance. Other materials showed significantly lower resistance compared with the Bk-41 refractory.

3.2. Tests under dynamic conditions

Figures 6 and 7 represent the results of corrosion tests carried out under dynamic conditions.

Volumetric corrosion of the examined Chinese-made materials ranges from 1.9 % (composition No. 2) to 34.9 % (composition No. 1). It should be noted that volumetric corrosion of the Bk-41 refractory manufactured by Podolsk Refractories JSC, when it is part of composition No. 2, is 35 % [12] that is comparable to the values obtained for refractories No. 3 and No. 4.

The linear corrosion rate under dynamic conditions for the examined materials ranges from 0.02 mm/day (sample No. 2) to 0.35 mm/day (sample No. 4).

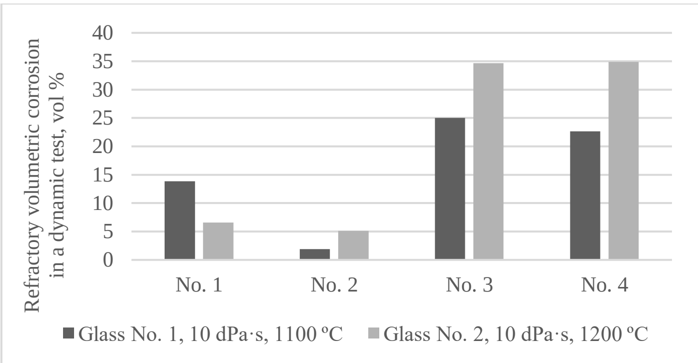

Figure 6 Refractory volumetric corrosion in a dynamic test, %.

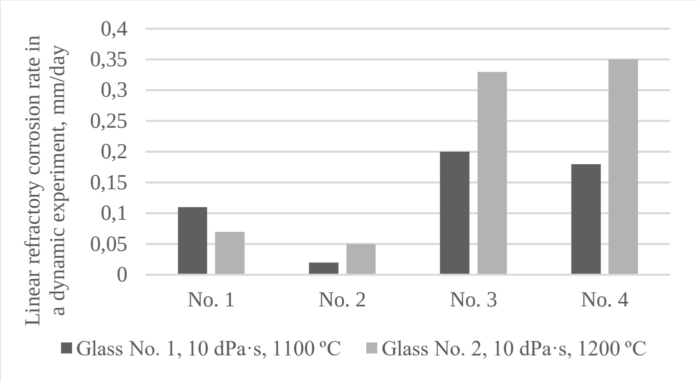

Figure 7 Linear refractory corrosion rate in a dynamic experiment, mm/day.

Linear corrosion rate of the Bk-41 refractory in the glass with composition No. 2 is 0.30 mm/day, and it is comparable to that of materials No. 3 and No. 4, as in the case of volumetric corrosion [12].

An increasing order of the indices of averaged volumetric (a) and linear (b) corrosion resistance of the materials tested under dynamic conditions (as compared to the Bk-41 refractory manufactured by Podolsk Refractories JSC [12]) is shown in Figure 8.

The displayed histograms demonstrate that, compared to the Bk-41 refractory manufactured by Podolsk Refractories JSC [11], all of the examined refractories show higher corrosion resistance in terms of averaged volumetric and linear corrosion (2 to 17.5 times lower averaged volumetric corrosion and 2.5 to 19.5 times lower averaged linear corrosion).

According to the experimental data obtained under dynamic conditions, there is an obvious superiority of refractories with a high content of chromium oxide over the rest of materials with regard to their resistance to glass melt corrosive attack. However, as opposed to static conditions, glass melt No. 2 demonstrates the highest corrosive activity in most experiments.

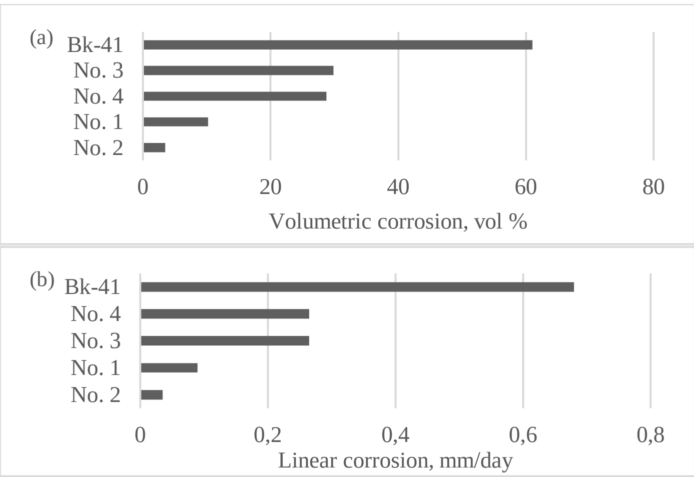

Figure 8 Indices of the averaged corrosion resistance of the tested materials calculated using: (a) – volumetric corrosion, %; (b) – linear corrosion, mm/day.

It is worth noting that under static conditions, the corrosion rate of sample No. 2 is significantly higher in the composition of borosilicate glass No. 1, and under dynamic conditions, in the composition of borosilicate glass No. 2. This effect may be related, on one hand, to the experimental conditions (static versus dynamic conditions), on the other hand, to the fact that in the composition of sample No. 2 does not contain oxides such as Al_2O_3, and ZrO_2 or Na_2O, which apparently have an effect during static tests.

Ceramic refractories No. 1 and No. 2, which contain 50 % and 94 % of Cr_2O_3 respectively, are the most resistant materials among the examined ones to chemical corrosion in glass melts studied. At the same time, they are characterized by high electrical conductivity compared to other refractories, which increases with temperature as in the case of Class 2 conductors [13], so, to substantiate their use in electrothermal equipment, additional research on electrochemical corrosion as well as testing of the relevant model units of equipment are required. In the absence of electrochemical corrosion, these materials have given a good account of themselves, when used in crucial elements of melting chambers of gas furnaces for glass making.

Refractory materials No. 3 and No. 4, in turn, are inferior in corrosion resistance to refractory No. 2, but currently they can be considered as the most suitable out of this group of materials for use in melting chambers of electric furnaces under conditions characterized by the uncertainties mentioned above.

China has set up production of this refractory in the Zhengzhou Yandong Refractories Co. factory, and today it is at a rather high and stable level that is comparable to the production level of Russian-made refractory (Podolsk Refractories JSC) and of European refractories.

The Bk-41 molten-cast refractory, in turn, is inferior in corrosion resistance to materials No. 1 and No. 2, but under static conditions, it is comparable to materials No. 3 and No. 4 manufactured in China. At this, the Bk-41 refractory is the most reliable, examined and widely spread refractory in the world for use in melting chambers of both gas and electric furnaces. Manufacturing of this grade of refractory in Russia at Podolsk Refractories JSC is at a rather high and stable level that is comparable to the global level of refractory production. The Bk-41 refractory was used in elements of the melting chamber of the Mayak EP-500/5 electric furnace (the Bk-37 material with lower corrosion resistance was applied in previous models of vitrification furnaces) [14].

The undertaken studies showed that corrosion properties of the examined group of refractories are affected by all factors considered in the experiments, including glass melt composition, test temperature, significant differences in refractory compositions, and test conditions (static or dynamic). In this work, it is not possible to determine a factor, which, to a large extent, stipulates the corrosion properties of refractories.

4. Conclusions

The experimental data obtained during the tests will be used for selection of the promising material for lining of a new generation removable small-scale melter.

Taking into account the results of material corrosion tests and other properties of refractories as well as production and economic factors, Chinese-made refractories No. 3 and No. 4 can be regarded as suitable for use in the refractory brickwork of a prototype small-scale melter, as they have shown sufficiently good results comparable to characteristics of the traditionally used Bk-41 material manufactured by Podolsk Refractories JSC or in several cases even surpassing those.

In order to recommend refractories with high content of chrome for use in the design of industrial electric furnaces for HLW vitrification, there is a need to carry out additional investigation of their electric conductivity at high temperatures, since effects associated with intensive electrochemical corrosion of materials may appear. In addition, in the future it is necessary to carry out chemical analysis of glasses after corrosion tests and monitor changes in the content of corrosion products, which will make it possible to draw more unambiguous conclusions and assess the rate of corrosion by the content of corrosion products.

Supplementary materials

No supplementary materials are available.

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Author contributions

Sergey Shaydullin: Investigation; Conceptualization; Resources; Data curation; Formal Analysis; Writing – Original draft.

Pavel Kozlov: Methodology; Project administration; Supervision; Writing – Original draft.

Mikhail Remizov: Software; Validation; Visualization; Formal Analysis; Writing – Review & Editing.

Sergey Lukin: Project administration; Software; Validation; Formal Analysis; Writing – Review & Editing.

Conflict of interest

The authors declare no conflict of interest.

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