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Studies of the Resistance of High-alumina Refractories to Fluoride-containing Refractory Materials to Curtain-containing Slags and Gaseous Atmosphere

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Abstract. *The modern production of ferrous metals requires a significant expansion of the complex of refractory materials that would make it possible to create porous products with sufficient strength and heat resistance. The high porosity of heat-insulating refractories causes their low bulk density and low thermal conductivity. In modern technological processes of metallurgy, the role of high-temperature linings of various thermal units made of refractory materials is extremely important. As a result of the study of the mechanisms of destruction of aluminosilicate refractories after operation in a coke gas cupola using fluorine-containing carbon wastes from aluminum production as a reducing agent, it was found that fluorine-containing impurities (cryolite* Na_3AlF_6*, fluorite* CaF_2*, villiomite NaF) decompose and sublimate at temperatures above 550-600°C and act on the refractory in the gas phase (in the form of vapors). In this case, the effect of fluorine-containing melts and vapors on the structure of the refractory leads to a decrease in the amount of glass phase, and to an increase in the degree of mullitization (the amount of secondary mullite formed from the glass phase). Thus, in almost the entire range of operating temperatures in industrial thermal units, fluorine from fluorine-containing carbon waste will be in the gas phase and affect the refractory lining over the entire working surface and the surface of the pore space of open pores. Given that fluorine-containing carbonaceous materials, when heated, release fluorine into the gas phase already at relatively low temperatures, then in the working space of thermal units that process fluorine-containing waste, it will be in the gas phase, as well as alkali metals present in the same waste. Therefore, to increase the resistance of the lining to the corrosive effects of the furnace atmosphere, it is necessary to use refractories with maximum density (low porosity) and maximum mullite content in the matrix (finely ground part of the charge). Such a set of properties can be obtained only in refractory products obtained by any molding method that provides a high density of grain packing and fired to obtain a ceramic mullite binder. Thus, for the lining of thermal units using fluorine-containing carbon waste as fuel or reducing agent, it is proposed to use mullite-silica or mullite refractory pressed fired products.*

Keywords: *heat-insulating and high-alumina refractories, linin, fluorine-containing carbonaceous wastes, cupola slag, mullite bond, clay, clay bond, alumina, sintering, mullite products, mullite-corundum products, cupola melting, study of refractories, slag resistance.*

Introduction

In the production of high-alumina products, ground highly plastic clay is used. The properties of the original clay and the fineness of the grinding determine the properties of the finished products.

With a semi-dry method for the production of products, when clay is introduced in a limited amount and must be evenly distributed over the surface of fireclay grains, the upper limit of its fineness does not exceed 0.2-0.5 mm.

High requirements for high alumina products also apply to bonding clay. The content of undesirable impurities (oxides of iron, calcium, magnesium, etc.) should be as low as possible.

High-alumina products are manufactured using multichamotte technology with a clay binder content in the charge in the amount of 10-20%. Depending on the type of feedstock used for the manufacture of products, various schemes for the preparation of high-alumina chamotte are used. The use of technical alumina makes it possible to obtain all types of high-alumina products.

The ratio and clay of technical alumina is determined by the calculation for finished products, and the content of Al_2O_3 in fireclay is taken 5-10% more than in finished products. This is necessary, since clay with a lower Al_2O_3 content goes into the bundle.

Technical alumina, having the form of spherulites, does not sinter well, therefore it is subjected to fine grinding in vibratory mills together with the dry part of the binder clay. Grinding bodies are steel balls with a diameter of 10-15 mm. The fineness of grinding is controlled by the residue on the sieve of 10,000 holes/cm^2 and should be no more than 2%.

Other high-alumina materials are also used in the refractory industry, for example, high-alumina slags formed in the production of chromium, titanium, and some other ferroalloys, and spent alumina-based catalysts.

Based on the analysis of the existing raw material base for the production of mullite-containing refractories and the range of high-alumina products, refractory kaolins, bauxites or andalusites mixed with commercial alumina for the production of high-alumina (mullite-silica or mullite) chamotte and a mixture of refractory clay and technical alumina as binder and matrix system of refractory products.

High-alumina refractories were tested as refractories: mullite dense products of the MLP brand, obtained according to the traditional fireclay scheme; mullite products of the MKV-72 brand, obtained from burnt bauxite; mullite and mullite-corundum products of the MKV-72A and MKV-80A grades obtained on andalusite refractory aggregates; mullite-corundum dense products of the MKTP-85 brand, obtained from corundum and quartz powders.

Slags from an iron foundry coke-oven cupola for the production of cast iron with spheroidal graphite (table 1) and fluorine-containing carbonaceous wastes from aluminum production (table 2) were used as corrosives in ratios corresponding to their ratio under production conditions.

Research methods

The study of the processes of fluorine release from carbonaceous wastes of aluminum production was carried out on a STA 449 F3 Jupiter synchronous thermal analysis device together with a QMS 403 Aeolos Quadro mass spectrometer (NETZSCH) in aluminum crucibles in the temperature range of 30÷600°C with a heating rate of 10°C/min. The measuring cell with the sample was purged with air at a rate of 50 ml/min. The obtained data were processed using the NETZSCH Proteus software (figure 1). As a result of the research, it was found that the removal of volatiles from waste occurs in two stages. At the first stage, at temperatures of 80-120°C, fluorine F (molecular weight 19) and partially CO_2 (molecular weight 44) are released, and at the second, above 450°C, the release of carbon dioxide CO_2 is activated.

Thus, in almost the entire range of operating temperatures in industrial thermal units, fluorine from fluorine-containing carbon waste will be in the gas phase and affect the refractory lining over the entire working surface and the surface of the pore space of open pores.

Therefore, the structure of refractories capable of withstanding the action of aggressive factors of metallurgical production resulting from the use of fluorine-containing waste as a fuel and/or reducing agent should have the highest possible density (low porosity) and contain the maximum possible amount of mullite resistant to fluorine melts and vapors [1-3].

The chemical composition of the studied materials was determined by the X-ray fluorescence method on an X-ray fluorescence energy-dispersive spectrometer ARL QUANT'X from Thermo Scientific, USA using the UniQUANT program (RhKα radiation; tube power – 50 W; voltage range – (4-50) kV with a step of 1 kV , current range – (0-1.98) mA with a step of 0.02 mA; Si(Li) detector, energy resolution – 150 eV).

The phase composition was determined by XRF on a Miniflrex 600 rotating anode diffractometer (CuKα radiation, $\lambda = 1.541862$ Å, survey interval – 3.00-60.00°, scanning step – 0.02°) «Rigaku – Carl Zeiss» (Japan) with control and data acquisition programs MiniFlex guidance and data processing package PDXL Basic. Diffraction peaks were identified using the JSPDS data bank. The semiquantitative assessment of the phase content was carried out using the corundum number RIR (Reference Intensity Ratio) according to the Chung method [1].

The apparent density, open porosity and water

Table 1 – Chemical composition of cupola melting slag

Components	Al_2O_3	SiO_2	Fe_2O_3	R_2O	CaO	MgO	TiO_2	S	MnO
Containing mass %	18,16	50,26	12,93	0,091	10,18	1,03	1,28	0,15	2,218

Table 2 – The chemical composition of the breakage of electrodes of aluminum production

Components	C	NaF	Al_2O_3	Na_3AlF_6	Na_2CO_3	CaF_2	SiO_2
Containing mass %	62,0	12,0	3,0	13,0	3,5	3,0	3,0

absorption (for kerosene) were determined according to GOST 2409-2014 «Refractories. Method for determining apparent density, open and total porosity, water absorption».

The slag resistance of products was determined according to DIN 51069-72 part 2 «Testing of ceramic raw materials and materials. Comparative tests of resistance of refractories to corrosion under the influence of solid and liquid substances at high temperatures».

The chemical and phase composition of the investigated high-alumina refractories are presented in tables 3 and 4.

The results of the study of the resistance of various mullite-containing refractories to fluorine-containing slags are shown in the figure 2 and in table 5.

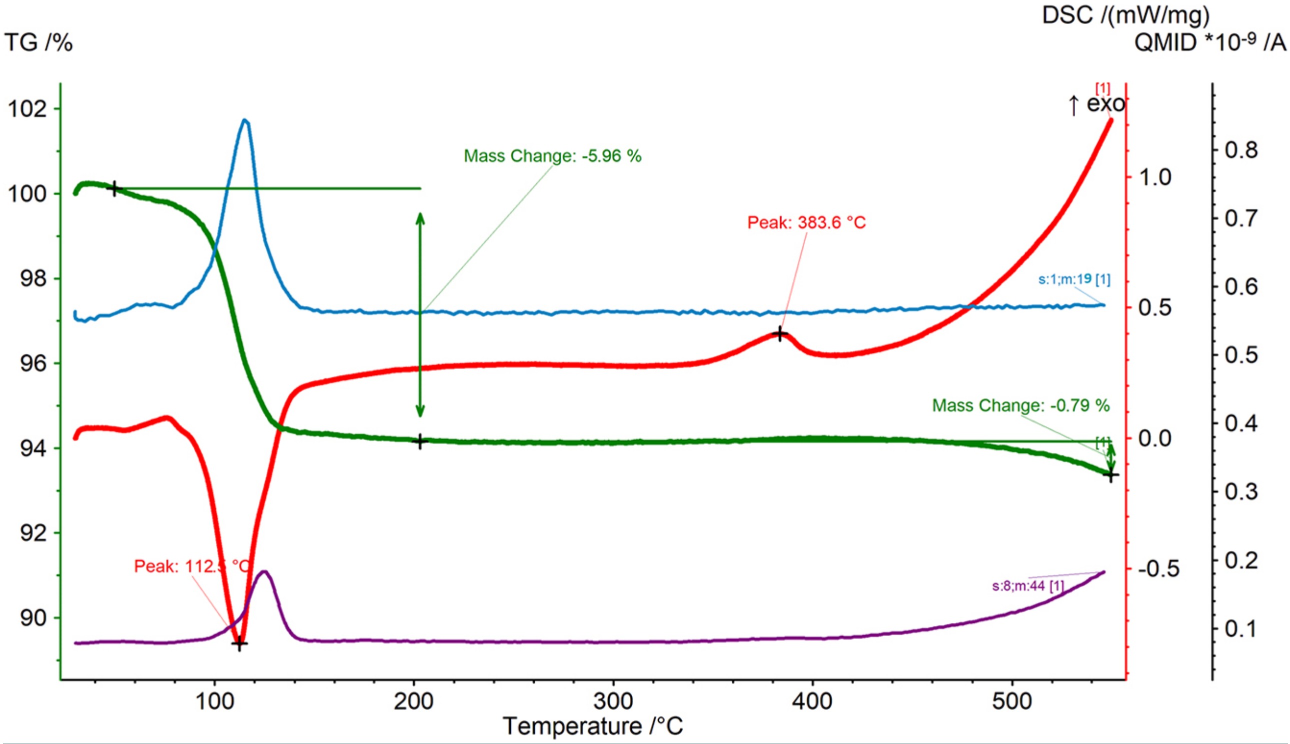

Figure 1 – Derivatogram of a sample of fluorinated carbon waste with mass spectrometry results

Table 3 – Chemical composition and properties of the studied refractories

Name	Chemical contents, mass, %				Features	
	SiO_2	Al_2O_3	Fe_2O_3	R_2O	Open Porosity %	Compressive strength, MPa
MLP	30,3	63,9	0,80	0,22	15	80
MKV-72	15,3	79,3	0,85	0,25	18	65
MKV-72A	16,0	77,9	1,2	0,25	19	70
MKV-80A	16,6	80,1	0,68	0,41	20	40
MCHD-85	10,5	87	1,3	0,35	20	60

Table 4 – Phase composition of the studied refractories

Name	Phase composition, mass fraction, %			
	Al_2O_3 SiO_2	$3Al_2O_3$ $2SiO_2$	SiO_2	Al_2O_3
MLP	-	78	4,0	7,0
MKV-72	impurities	61	-	30
MKV-72A	5,7	62	2,9	33
MKV-80A	11	49	1,0	50
MKTP-85	-	52	1,0	65

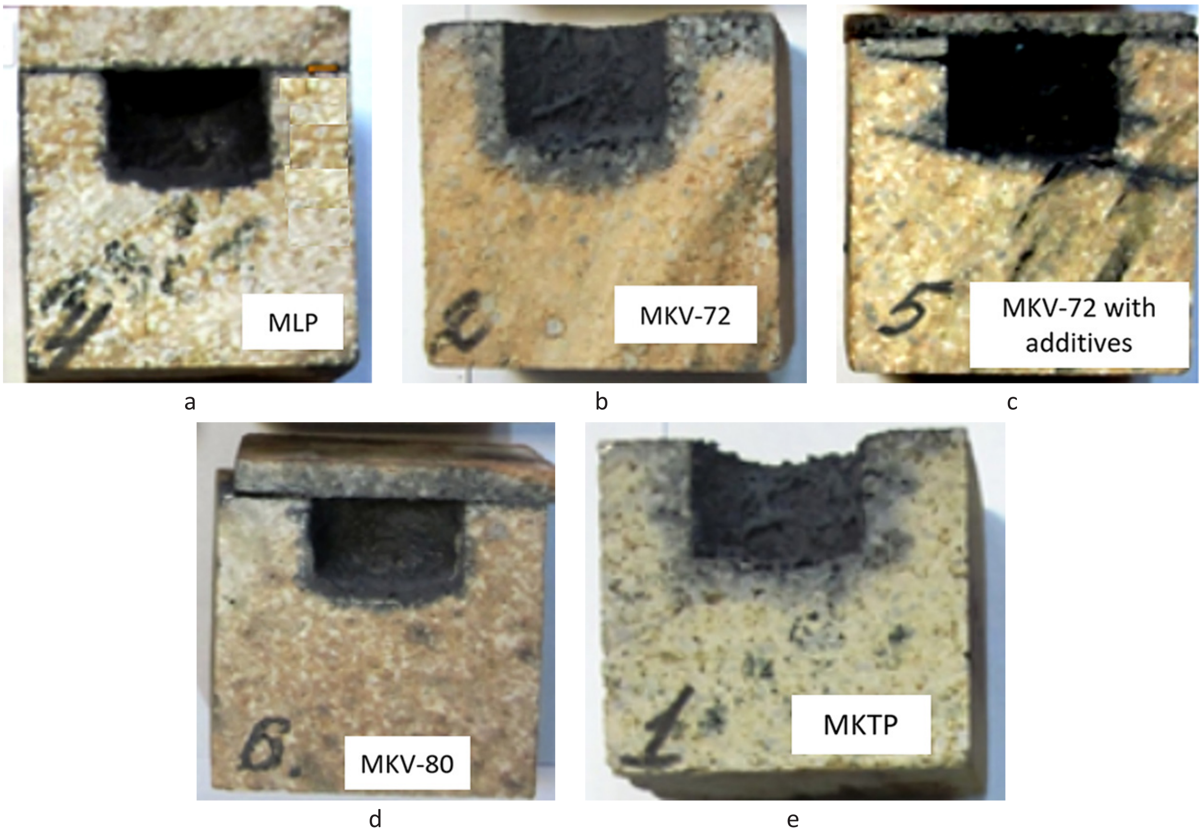

Figure 2 – Investigated refractories after slag resistance tests

Table 5 – Mineral composition of slag impregnated zones (%)

Name	Contents	MLP	MKV-72	MKV-72 with andalusite	MKV-80 with andalusite	MKTP-85
Mullit	$3Al_2O_3×2SiO_2$	20-25	10-15	10-15	35-40	35-40
Andalusite	$Al_2O_3×SiO_2$	-	-	3-5	10-15	-
Herzenite-magnetite	$Fe(Al,Fe)_2O_4$	10-15	10-15	10-15	10-15	15-20
Solid Solution [Al_2O_3-Fe_2O_3]	$(Al,Fe)_2O_3$	10-15	25-30	25-30	10-15	5-10
Gelenit	$(2CaO×Al_2O_3×2SiO_2)$	2-5	5-10	5-10	5-7	5-7
Fayalite	$2FeO×SiO_2$	5-7	10-15	10-15	7-10	10-15
Glass phase + cristobalite	$RO×Al_2O_3×Fe_2O_3×nSiO_2+SiO_2$	25-30	35-40	35-40	20-25	20-25

With the use of a binocular, the following maximum depth of slag impregnation of samples was established: MLP ~1.0%; MKV-72 ~3.0%; MKV-72 and MKV-80 (based on andalusite raw materials) ~2.0%; MKTP-85 ~2.0%.

Since cupola slag with fluorine and alkali additives has a low viscosity and complete wetting of all refractory phases of these products, the impregnation depth is determined mainly by the number and diameter of open pores and the chemical interaction of the refractory with slag [4].

An analysis of the microstructure of the impregnated zones of the investigated refractories indicates the reaction between all samples and the fluorine-alkali-containing slag melt according to the scheme of impregnation and partial dissolution, with the formation of ferruginous mullite and corundum, gehlenite, fayalite, and a large amount of glass phase.

The minimum impregnation and the highest slag resistance are shown by a sample of the product of the MLP brand, which is due to the high viscosity of the reaction melt (refractory-slag contact) and low channel porosity. A highly viscous (acidic) contact layer closes the pores (Tmelt – 1400°C). Samples of products of the MKV-72, andalusite MKV-72 and MKV-80, MKTP-85 brands will probably not last longer than the MLP brand, since it contains the minimum amount of SiO_2 consumed for the formation of a contact glass phase.

Mullite-containing products under service conditions (in a reducing fluorine- and alkali-containing environment) interact not with metallic iron, but with a gas atmosphere, slag and metal oxidation products FeO, Fe_2O_3, Fe_3O_4. Wear occurs due to melting due to the formation of a low-melting reaction layer at the contact of products with the melt of a thermal unit (eutectic in the system CaO-FeO-Fe_2O_3-SiO_2 ~1200°C). Under these conditions, the maximum resistance is provided by the maximum content of mullite in the refractory and its minimum open porosity.

Conclusions

1. Mullite dense refractory products can be obtained from natural fired (bauxite, andalusite) or synthetic (high-alumina chamotte) materials using the technology of multi-chamotte products by semi-dry pressing.

2. The resistance of products under operating conditions under the influence of a fluorine- and alkali-containing atmosphere under reducing conditions will depend on the density of the structure (open porosity) and on the amount of mullite in the original refractory.

3. The optimal high-alumina filler for the production of mullite products that are stable in thermal units using fluorine-containing carbon waste as a fuel or reducing agent will be high-alumina grog of mullite composition with a minimum content of glass phase.

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Жоғары глиноземді отқа төзімділердің құрамында фтор бар қождар және газды атмосфераға төзімділігін зерттеу

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Аңдатпа. *Қазіргі заманғы қара металл өндірісі отқа төзімді материалдар кешенін едәуір кеңейтуді талап етеді, бұл олардың беріктігі мен ыстыққа төзімділігі жеткілікті кеуекті өнімдер жасауға мүмкіндік береді. Жылу оқшаулағыш отқа төзімді заттардың жоғары кеуектілігі олардың төмен көлемдік тығыздығы мен төмен жылу өткізгіштігін анықтайды. Металлургияның қазіргі технологиялық процестерінде отқа төзімді материалдардан жасалған әртүрлі жылу агрегаттарының жоғары температуралы төсеніштерінің рөлі ерекше маңызды. Алюминий өндірісінің құрамында фторы бар көміртекті қалдықтарын тотықсызданды-*

рғыш ретінде пайдалана отырып, кокс-газ вагранкасында пайдаланғаннан кейін алюминосиликатты отқа төзімділердің бұзылу механизмдерін зерттеу нәтижесінде құрамында фторы бар қоспалар (Na_3AlF_6 криолиті, CaF_2 флюориті, NaF виллиомиті) 550-600°C жоғары температурада ыдырайтыны және сублимацияланатыны және газ фазасындағы (бу түрінде) отқа төзімділікке әсер ететіні анықталды. Бұл ретте құрамында фторы бар балқымалар мен булардың отқа төзімді құрылымға әсері шыны фаза санының азаюына және муллитизация дәрежесінің (шыны фазадан түзілетін қайталама муллит мөлшері) ұлғаюына әкеледі. Осылайша, өнеркәсіптік жылу қондырғыларындағы жұмыс температурасының барлық диапазонында фторы бар көміртегі қалдықтарынан фтор газ фазасында болады және бүкіл жұмыс беті мен ашық кеуектердің кеуек кеңістігінің бетінде отқа төзімді төсемге әсер етеді. Фторы бар көміртекті материалдар қызған кезде фторды газ фазасына салыстырмалы түрде төмен температурада шығаратындығын ескере отырып, фторды ұстайтын қалдықтарды өңдейтін жылу қондырғыларының жұмыс кеңістігінде ол газ фазасында, сондай-ақ сол қалдықтардағы сілтілі металдарда болады. Сондықтан, төсеніштің пеш атмосферасының коррозиялық әсеріне төзімділігін арттыру үшін максималды тығыздығы (төмен кеуектілігі) және матрицадағы муллиттің максималды мөлшері (зарядтың жұқа бөлігі) бар отқа төзімді заттарды қолдану қажет. Мұндай қасиеттер жиынтығын кез-келген қалыптау әдісімен алынған отқа төзімді бұйымдардан алуға болады, бұл астық қаптамасының жоғары тығыздығын қамтамасыз етеді және керамикалық муллит байламын алғанға дейін күйіп кетеді. Осылайша, құрамында фтор бар көміртекті қалдықтарды отын немесе тотықсыздандырғыш ретінде пайдаланатын жылу агрегаттарын футерлеу үшін муллит-кремнеземді немесе муллит отқа төзімді престелген күйдірілген бұйымдарды пайдалану ұсынылады.

Кілт сөздер: *жылу оқшаулағыш және жоғары глиноземді отқа төзімді, футеровка, құрамында фтор бар көміртекті қалдықтар, муллит байламы, саз, саз байламы, сазбалшық, жентектеу, муллитті бұйымдар, муллиткорундты бұйымдар, вагранкалы балқыту, отқа төзімді заттарды зерттеу, қожға төзімділік.*

Исследования стойкости высокоглинозёмистых огнеупоров к фторсодержащим шлакам и газовой атмосфере

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Аннотация. *Современное производство черных металлов требует значительного расширения комплекса огнеупорных материалов, которые позволили бы создать пористые изделия при достаточной их прочности и термостойкости. Высокая пористость теплоизоляционных огнеупоров обусловливает их малую объемную плотность и низкую теплопроводность. В современных технологических процессах металлургии роль высокотемпературных футеровок различных тепловых агрегатов, выполненных из огнеупорных материалов, исключительна важна. В результате исследования механизмов разрушения алюмосиликатных огнеупоров после эксплуатации в коксогазовой вагранке с использованием в качестве восстановителя фторсодержащих углеродистых отходов алюминиевого производства установлено, что фторсодержащие примеси (криолит Na_3AlF_6, флюорит CaF_2, виллиомит NaF) разлагаются и возгоняются при температурах выше 550-600°C и воздействуют на огнеупор в газовой фазе (в виде паров). При этом воздействие фторсодержащих расплавов и паров на структуру огнеупора приводит к снижению количества стеклофазы и к увеличению степени муллитизации (количества вторичного муллита, образующегося из стеклофазы). Таким образом, практически во всём интервале рабочих температур в промышленных тепловых агрегатах фтор из фторсодержащих углеродистых отходов будет находиться в газовой фазе и воздействовать на огнеупорную футеровку по всей рабочей поверхности и поверхности порового пространства открытых пор. Учитывая, что фторсодержащие углеродистые материалы при нагревании выделяют фтор в газовую фазу уже при относительно низких температурах, то в рабочем пространстве тепловых агрегатов, перерабатывающих фторсодержащие отходы, он будет находиться в газовой фазе, так же как и щелочные металлы, присутствующие в этих же отходах. Следовательно, для повышения стойкости футеровки к корродирующему воздействию печной атмосферы нужно использовать огнеупоры с максимальной плотностью (низкой пористостью) и максимальным содержанием муллита в матрице (тонкомолотой части шихты). Такой набор свойств можно получить только в огнеупорных изделиях, получаемых любым методом формования, обеспечивающим высокую плотность упаковки зёрен и обожжённых до получения керамической муллитовой связки. Таким образом, для футеровки тепловых агрегатов, использующих в качестве топлива или восстановителя фторсодержащие углеродистые отходы, предлагается использовать муллитокремнезёмистые или муллитовые огнеупорные*

прессованные обожжённые изделия.

Ключевые слова: *теплоизоляционные и высокоглинозёмистые огнеупоры, футеровка, фторсодержащие углеродистые отходы, ваграночный шлак, муллитовая связка, глина, глиняная связка, глинозем, спекание, муллитовые изделия, муллитокорундовые изделия, ваграночная плавка, исследование огнеупоров, шлакоустойчивость.*

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