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Technology of production of aluminosilicate refractories for units processing fluorinated waste

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Annotation
The aluminium production process through the electrolysis of cryolite-alumina melts involves a series of interconnected, sequential, and parallel technological operations, each defined by a specific level of engineering and technological advancement. The development of the modern aluminum industry is closely tied to the adoption of resource-efficient and environmentally friendly technologies, which focus on recycling secondary materials and industrial waste. Fluorinated carbon-based materials release fluorine into the gas phase at relatively low temperatures when heated, and in thermal units processing fluorinated waste, this fluorine, along with alkali metals, will remain in the gas phase. To enhance the durability of furnace linings against the corrosive atmosphere, refractories with the highest possible density (low porosity) and a high concentration of mullite in the matrix (the finely ground component of the batch) are required. These properties can only be achieved in refractory products produced by the semi-dry pressing method, which ensures high grain packing density and leads to the formation of a ceramic mullite bond after firing.

Keywords: fluorinated carbonaceous materials, crushing, mullite refractories, highly lamellar clay, grinding, mullite-silica products, binding, chamotte, drying, firing.

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Introduction

The production of aluminum by electrolysis of cryolite-alumina molten includes several interrelated and sequentially parallel technical processes, each characterized by a certain level of engineering and technology development. Despite the constant progressive development in terms of improving the means and methods of implementing technical processes, several technical problems remain unresolved. The existence of these problems is due to the lack of theoretically and economically informed technical solutions, which are a powerful lever for increasing production efficiency [1].

The development of the aluminum industry at the present stage is associated with developing and implementing resource-saving and environmental technologies for processing secondary resources and technical waste. Implementing technological solutions aimed at reducing material and labor costs in c production and involving illiquid waste and production products in efficient processing will increase competitiveness, economic attractiveness and environmental safety [2].

Since fluorinated carbonaceous materials release fluorine into the gas phase at relatively low temperatures, both fluorine and alkali metals from the same waste will be present in the gas phase within the working area of the fluorinated waste heat treatment plant. To enhance the durability of the furnace lining against the corrosive effects of this atmosphere, it is essential to use refractories with high density (low porosity) and a maximum concentration of mullite in the matrix (the finely ground portion of the material). These characteristics can only be achieved by producing refractories through semi-dry molding, which ensures a high packing density of grains and results in a ceramic-mullite bond after firing [[3], [4]].

For the lining of thermal units that utilize fluorinated carbon waste as fuel or a reducing agent, the use of mullite-silica or mullite refractory pressed and fired products is recommended.

In the production of mullite-silica refractories, two stages are conditionally distinguished: the production of high-alumina chamotte and the production of products. The basis of production is the production of densely baked high-alumina chamotte, which serves as a thinning material and binds when pressing products with high-plastic clay [5].

Production of clay in batches: In the manufacture of mullite-silicon products, finely ground high-plasticity clay is utilized. The characteristics of the raw clay and the degree of grinding directly influence the properties of the final products [[6], [7]].

In the semi-dry production method, where clay is added in limited quantities and needs to be evenly distributed across the surface of the chamotte grains, the maximum particle size is restricted to 0.2-0.5 mm. The process flow for preparing binder clay is illustrated in Fig. 1.1.

After rough crushing to pieces of no more than 50 mm, the clay enters the drying drum for drying, where it is dried in direct flow, i.e. the coolant and clay in the drying drum move in the same direction. In this case, the temperature of the coolant drops sharply at the beginning of the drum due to a large amount of evaporating water from the clay, thereby eliminating the possibility of drying the clay [8].

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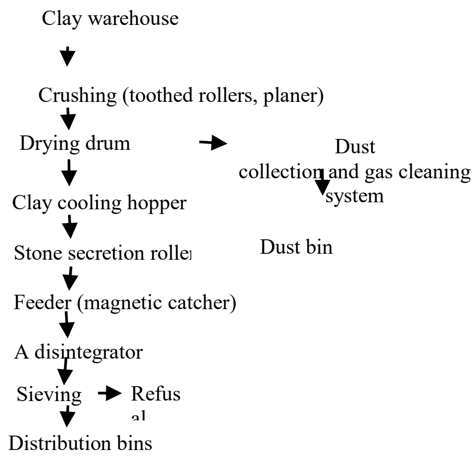

Figure 1.1. - Technological scheme of preparation of binder clay

Flue gases from natural gas combustion, waste heat from kilns, etc. are used as heat carriers. The temperature of the incoming gases into the drying drum is no more than 900 ° C, at the outlet – not less than 100 ° C. The moisture content of clay in the drying process decreases from 15-20 to 7-10%. The residence time of clay in the drying drum is within 20-30 minutes with a drum length of 8-14 m. The capacity of the drying drum is 10-14 t/h. It is not recommended to over-dry the clay, since when grinding such clay is very dusty and may lose its plastic properties.

The grinding of dry clay is carried out in hammer or ball mills [9].

Waste gases containing dust up to 25 microns in size are sent to a dust cleaning system consisting of cyclones, the efficiency of which is not higher than 70-75%. For more efficient purification of gases coming from drying drums, two-stage systems are used, where more highly efficient devices are used in the second stage. Such devices are electrofilters and bag filters, with a degree of dust capture which is at least 98%.

Strict quality standards for mullite-silicon products also extend to the binding clay. The levels of undesirable impurities, such as iron oxides, calcium, magnesium, and others, must be kept to a minimum.

Mullite-silicon products are produced using a multi-chamotte process, with the clay binder comprising 10-20% of the mixture. Different preparation methods for mullite-silicon chamotte are employed depending on the raw materials used.

Utilizing technical alumina or alumina-containing waste (such as slags, spent catalysts, or dust from cleaning processes) allows for the production of all types of mullite-silicon products [10].

The experimental part

The ratio of clay to technical alumina is calculated based on the final product requirements, ensuring that the Al2O3 content in the chamotte is 5-10% higher than in the finished product. This is necessary because the clay used for binding contains a lower Al2O3 content.

Technical alumina, in the form of spherulites, has poor sintering properties, so it is finely ground in vibrating mills along with the dry portion of the binder clay. Steel balls, 10-15 mm in diameter, are used as grinding media. The fineness of the grind is controlled by the residue left on a sieve with 10,000 rel./cm^2, which should not exceed 2% [11].

The mixing of young alumina and clay is carried out in a mixer of the SM-115 type with simultaneous moistening of the mixture to 17-18%. Mixing with waterlogging up to 25-28% is carried out in a two-shaft mixer with subsequent processing and compaction in a vacuum belt press. The resulting blanks with dimensions of 50 × 50 × 50 mm are sent for firing into a rotating furnace

The chamotte is fired at higher temperatures, and the higher the content of A12O3 in the charge. The firing temperature ranges from 1350-1550 ° C. The water absorption of burnt chamotte should be no more than 1-3%. The chamotte coming out of the rotary kiln has the following grain composition, %:

Fraction, mm	>10	10-3	3-0.5	< 0.5
Content, %	30-50	25-30	25-30	5-10

The dust captured by the gas cleaning system contains a lot of technical alumina, so it is fully returned to production at the briquette forming stage [[12], [13]].

Several reactions are possible between the aluminosilicate refractory and the main components of the electrolyte – cryolite and sodium fluoride:

$$18(NaF)_l + 2(Al_2O_3 2SiO_2)_s + 5(SiO_2)_s = 9(NaAlSiO_4)_l + 3(Na_3AlF_6)_l \quad (1)$$

$$6(NaF)_l + 3(SiO_2)_s + 23(Al_2O_3 2SiO_2)_s = 3(NaAlSiO_4)_l + 3(Na_3AlF_6)_l \quad (2)$$

$$6(Na_3AlF_6)_l + 23(SiO_2)_l + 23(Al_2O_3 2SiO_2)_s = 18(NaAlSiO_4)_l + 9(SiF_4)_g \quad (3)$$

The appearance of the refractory samples after operation during one cupola movement is shown in Fig. 1.2. and 1.3.

Figure 1.2 – Samples of refractory after operation in the cupola shaft

Figure 1.3 – Samples of refractory after operation in the furnace of the cupola

The product after use in the shaft (Fig.1.2) carbonized throughout the depth and has molten crusts on the treated surfaces. The shape of the untreated surfaces is mostly preserved, and the crusts from processing are evenly erased. Slag is also visible on the sides, which indicates the penetration of molten components and the vapor phase into the seams between the materials. There is no mechanical destruction of the samples [[14], [15]].

The product after use in the oven (Fig. 1.3) has irregularities, especially on treated surfaces, where molten crusts up to 5 mm thick are visible. Carburization is not observed. The sample is fragmented by internal cracks formed as a result of mechanical action. Melting and compression zones are observed inside the product and at the joints with neighbouring products [[16], [17]].

Discussion of the results

This study observes that the product exhibits a denser structure after operation in the cupola mine than after service in the furnace. This behavior is unusual and suggests the presence of volatile components in the charge melt, which act as stronger sintering agents for aluminosilicate refractories compared to iron oxide and metallic iron, the primary components of the furnace workspace. The uniform influence of the sintering factor on the overall structure of the refractory across the thickness of the lining is noteworthy. For instance, in previous studies [E. A. Sidorina, A. Z. Isagulov, I. D. Kashcheev, K. G. Zemlyanoi, New refractories No. 5 2022 "Development of technology for superdense slag-resistant aluminosilicate refractories" Similar results have also been reported regarding the behavior of aluminosilicate refractories under various operating conditions [[18], [19]]. On the basis of aluminosilicate raw materials of the Republic of Kazakhstan, a technology for producing superdense aluminosilicate refractories with increased slag resistance for thermal processes of utilization of fluorinated carbonaceous waste is proposed. The use of "ceramic" technology, which allows to obtain a structure with a water absorption of less than 1% for both chamotte and refractory products with different Al2O3 content. Further, products after operation in the cupola furnace show a clearly defined melted crust in the working area, formed at the point of contact with molten cast iron. This area, formed as a result of impregnation by molten cast iron and/or slag into the refractory, shows significant modification. In contrast, the transitional and impregnated (slightly modified) zones exhibit structures that differ little from the original refractory, even though the sintering factor in the furnace shaft impacts the structure's density, including the furnace lining. Comparing these findings with other studies on refractory behavior under similar conditions could provide additional insights into the factors influencing the observed structural changes. When selecting a grain composition, an intermittent grain composition is employed, where clay and chamotte are ground together. Grinding high-alumina chamotte presents significant challenges due to its high hardness. Large chamotte pieces are initially crushed in a jaw crusher and then further reduced in size using a ball mill with self-seeding [[20], [21]]. The chamotte stream undergoes repeated magnetic separation to remove metallic iron, ensuring that the content of metallic iron in the coarse fraction does not exceed 0.05%, and in the fine fraction, it does not exceed 0.7%. From the analysis of the literature data, it follows that in order to increase the economic efficiency of metallurgical production, various man-made carbon wastes can be used as fuel and reducing agent instead of significantly more expensive coal and coal coke. The issue of utilization of fluorine and alkali-containing carbonaceous waste from aluminum production is particularly relevant for the Republic of Kazakhstan.

Conclusions

In practice, the following charge compositions are used:

- mullitosilicon products with A12O3 content of 28-45 wt. % contain chamotte fractions 3-1 mm 55-65% and joint grinding of clay and chamotte 35-45%. In the joint grinding, the clay content is 35, and the chamotte is 65%. The total clay content in the charge is ~ 25 %;
- mullite products with A12O3 content > 45 wt. % contain chamotte fractions 3-1 mm 55% and joint grinding of clay and chamotte 45%. In the joint grinding, the clay content is 25, and the chamotte is 75%. The total clay content in the charge is ~ 13%

Mixing is carried out on runners with heavy rollers with strict observance of the order of feeding materials into the mixer. First, a large fraction of chamotte is loaded and a clay slip with a density of 1.30-1.35 g / cm3 is introduced. After 5 minutes of mixing, a finely ground mixture of joint grinding is loaded. The total mixing cycle is 15-20 min. The humidity of the prepared mass is 5-6%.

Pressing of products is carried out on hydraulic presses. To improve the quality of products, the number of presses per minute is reduced to four. The density of the formed raw material, depending on the type of press used, is in the range of 2.70-2.85 g/ cm^3.

Drying of mullite-silica products does not cause difficulties and is carried out in tunnel dryers combined with a tunnel furnace. The final moisture content of the raw material after drying is not more than 2%.

The products are fired in tunnel furnaces with a flat arch of 156 m long and 3.1 m wide. The height from the bottom of the trolley to the arch is 1.1 m . The height of the cage is small, since the firing temperature, depending on the content of Al2O3, ranges from 1350-1550 ° C. The firing duration is 80 h. The low height of the cage is necessary in this case for uniform firing and exclusion of delamination when the coolant moves along the height of the tunnel furnace channel.

Conflict of interest. On behalf of all the authors, the correspondent author declares that there is no conflict of interest.

CRediT author statement: **E. Sidorina**: Conceptualization, Methodology, Software. **A. Isagulov**: Data curation, Writing- Original draft preparation. **M. Rabatuly:** Visualization, Investigation. **Y**. **Yang**: Software, Validation.

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Құрамында фторы бар қалдықтарды өңдейтін қондырғылар үшін алюмосиликатты отқа төзімді бұйымдарды өндіру технологиясы

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Түйіндеме
Алюминийді криолит-глиноземді балқымаларды электролиздеу негізінде алу өзара байланысты, бірізді және параллельді технологиялық үдерістер кешенін қамтиды, олардың әрқайсысы инженерлік-технологиялық дамудың белгілі бір жеткен деңгейімен сипатталынады. Қазіргі алюминий өнеркәсібінің эволюциясы қайталама қорларды және техногендік қалдықтарды өңдеуге бағытталған қор үнемдейтін және экологиялық технологияларды әзірлеумен және енгізумен байланысты. Фторлы көміртекті материалдар қыздырылған кезде фторды газ фазасына салыстырмалы түрде төмен температурада шығарады, содан кейін фторлы қалдықтарды өңдейтін жылу қондырғыларының жұмыс кеңістігінде ол газ фазасында болады, сонымен қатар сол қалдықтарда болатын сілтілі металдар. Демек, пеш атмосферасының коррозиялық әсеріне төсемнің беріктігін арттыру үшін матрицадағы ең жоғары тығыздығы (кеуектілігі төмен) және максималды муллит мөлшері (шихтаның ұсақ ұнтақталған бөлігі) бар отқа төзімді заттарды қолдану қажет. Мұндай қасиеттер жиынтығын тек жартылай құрғақ қалыптау әдісімен алынған отқа төзімді өнімдерден алуға болады, бұл дәндердің жоғары тығыздығын қамтамасыз етеді және керамикалық муллит байламы алынғанға дейін күйдіріледі. Муллитті кремний диоксиді немесе қысыммен күйдірілген муллитті отқа төзімді бұйымдар фторкөміртекті қалдықтарды отын немесе тотықсыздандырғыш ретінде пайдаланатын жылу қондырғыларын төсеу үшін қолданылады.

Түйін сөздер: құрамында фтор бар көміртекті материалдар, ұсақтау, муллитті отқа төзімді заттар, жоғары пластиналы саз, ұнтақтау, муллит-кремнийлі бұйымдар, байлам, шамот, кептіру, күйдіру.

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Технология производства алюмосиликатных огнеупоров для агрегатов, перерабатывающих фторсодержащие отходы

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Поступила: *31 мая 2024* Рецензирование: *19 июня 2024* Принята в печать: *2 октября 2024*	**Аннотация** Процесс производства алюминия с использованием электролиза криолит-глинозёмных расплавов включает комплекс взаимосвязанных, последовательных и параллельных технологических этапов, каждый из которых характеризуется определённым уровнем инженерно-технологического развития. Современная алюминиевая промышленность развивается благодаря разработке и внедрению технологий, ориентированных на экономию ресурсов и охрану окружающей среды, что включает переработку вторичных ресурсов и техногенных отходов. При нагревании фторсодержащие углеродные материалы выделяют фтор в газообразной форме уже при относительно низких температурах, что приводит к его присутствию в газовой фазе в тепловых агрегатах, перерабатывающих такие отходы, наряду с щелочными металлами, содержащимися в этих отходах. Для повышения устойчивости футеровки к коррозионными воздействиям печной атмосферы необходимо использовать огнеупоры с максимальной полностью (низкой пористостью) и высоким содержанием муллита в матрице (тонкоизмельчённой части шихты). Достичь такого набора свойств возможно только при производстве огнеупорных изделий полусухим методом формования, который обеспечивает высокую плотность упаковки зёрен и обжиг до получения керамической муллитовой связки. Муллитовые кремнезёмные или мулитовые огнеупорные изделия, обожжённые под давлением, применяются для футеровки тепловых агрегатов, использующих фторуглеродные отходы в качестве топлива или восстановителя.
	Ключевые слова: фторсодержащие углеродистые материалы, дробление, муллитовые огнеупоры, высокопластинчатые глина, помол, муллитокремнеземистые изделия, связка, шамот, сушка, обжиг.
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