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Studies of the thermal stability of briquettes based on microsilica

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ABSTRACT
The object of study in this work is microsilica. Microsilica is a pulverized technogenic waste generated during the smelting of technical silicon in industries. The intended use of this material is to obtain the highest quality grades of technical silicon in the end result without the use of expensive quartz. Microsilica is a finely dispersed powder, the direct processing (without preliminary preparation) of which into technical silicon is impossible in ore-thermal furnaces. That requires the manufacture of high-strength briquettes based on it, meeting all the requirements for raw materials for ore-thermal furnaces. In this paper, the authors present the results of a study of the thermal stability of microsilica-based briquettes in order to optimize the physicochemical properties of the resulting briquettes. The tested microsilica briquettes with various carbonaceous reducing agents (screenings of coke of thermo-oxidative coking, screenings of charcoal, etc.) were obtained on a large-scale laboratory roller briquetting press ZZXM-4. The evaluation of the thermal stability of the briquettes was carried out according to the method in which the resulting briquettes are subjected to thermal shock followed by abrasion on a special drum. The thermal resistance of a briquette is defined as the ratio of the weight of the main body of the briquette after the abrasion test to the sum of the weight of the main body and the crumbled material. The dependence of thermal stability on the granulometric composition of briquettes was also determined. The optimal granulometric composition was determined, with which the briquettes have satisfactory thermal stability. Thus, the most technologically advanced granulometric composition of the briquetting charge for reduction smelting in an ore-thermal furnace was established. It is best to use briquettes with granulometric compositions of the appropriate ratio of fractions 0-1; 1-3; 3-5 mm with the following proportions 35/35/30 and 60/20/15, as well as briquettes with particle size distribution within these ranges of variation.
Keywords: thermal stability, briquettes, microsilica, silicon briquettes, briquetting, silicon waste.

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Introduction

Due to the widespread use of silicon in various fields of industry, a lot of work is currently underway to optimize the technological process of its production [1]. An important problem of the technological process of production of technical silicon in the conditions of Tau-KenTemir LLP is the formation of a large amount of microsilica. Under the conditions of Tau-KenTemir LLP, when smelting one ton of technical silicon according to standard technology using vein quartz with a carbonaceous reducer, one ton of microsilica is captured by gas cleaning systems, with 65-70% extraction of silicon into metal [[2], [3]]. Such average annual technical and economic indicators significantly worsen the economics of production, which not only highlights the problem of the lack of technological solutions for the processing/utilization of microsilica but also creates an environmental problem associated with the storage of technogenic waste [[4], [5], [6], [7]]. This fact indicates the need for a number of research works to find a competent utilization of microsilica. It should also be noted that developments are underway around the world to improve existing and create new methods for involving finely dispersed raw materials in metallurgical processing [[8], [9]], including in the production of technical silicon.

Microsilica is an ultra-disperse material consisting of spherical particles, obtained in the process of cleaning the exhaust gases of furnaces in the production of technical silicon [10]. In the world, only a small part of these wastes has found application for the manufacture of building concrete mixtures [[11], [12]]. In turn, microsilica can be a significant quality source of raw materials for the production of technical silicon. This material in the bulk consists of a valuable component - silicon dioxide and the number of harmful impurities is not high, so it is important to use microsilica as a raw material for the production of silicon and its alloys.

Utilization and use of pulverized waste in the form of microsilica should be considered as an important direction in saving material resources [[13], [14], [15]]. The use of microsilica for the production of silicon is complicated by the fact that it is in a finely dispersed form [[16], [17], [18]], and does not meet the requirements for charge materials for smelting in ore-thermal furnaces. Therefore, it is required to develop an optimal mode of agglomeration of this material to obtain high-strength briquettes. This should be taken into account when developing a new resource-saving technology for smelting crystalline silicon using microsilica. Briquetting is considered the most effective method of agglomeration for metallurgical production due to the possibility of agglomeration of raw materials of wide-size classes in almost any ratio and any chemical composition, as well as the ergonomics of the process.

Currently, there is no state regulatory and technical base that regulates the requirements for briquettes as an element of the technological process. In this connection, the resulting briquettes in terms of their chemical composition, geometric dimensions (size), and strength must meet the requirements and features of the technological process in which they are supposed to be used. Since the chemical composition and size of briquettes are set before briquetting, one of the important characteristics of the quality of briquettes is their thermal stability. Heat resistance is characterized by the preservation of the shape of briquettes when they are kept for 3 minutes at a temperature above 600 °C.

The authors of this work, in order to optimize the process of microsilica briquetting for the smelting of technical silicon, for the first time among previous studies, took into account the factor of thermal stability of briquettes. This factor has never been taken into account before. The lack of this experience often led to the disintegration of briquettes when loaded onto the top of an ore-thermal furnace. Therefore, a negative attitude towards the possibility of using briquettes based on finely dispersed quartz materials as a feedstock in the smelting of technical silicon has firmly entrenched in the silicon industry.

Experimental part

High-strength briquettes based on microsilica with various carbonaceous reducing agents (screenings of coke of thermo-oxidative coking, screenings of charcoal, etc.) were obtained on a large laboratory briquetting press ZZXM-4. By achieving close contact in the briquette between microsilica silicon oxides and solid carbon of carbonaceous reducing agents, the most favorable conditions for the reduction and smelting of silicon are achieved. In this case, it becomes possible to involve scarce low-ash carbonaceous reducing agents as fine fractions reducing agents. The liquid glass was used as a binder. The optimal consumption of liquid glass per ton of charge mixture was established - 0.067 t / t at a charge moisture content of 8%. The resulting briquettes were subjected to natural drying at a temperature of 18-24 ^{0}C during the day.

Until now, with the use of briquette raw materials, not only in the production of silicon but in many electrothermal industries, little attention has been paid to the thermal stability index of briquettes. Many researchers only qualitatively described the fact of scattering of briquettes when they hit the top of an ore-thermal furnace as a result of a sharp change in temperature [[19], [20]].

To optimize the process of smelting technical silicon from briquette mono-charge, the authors of this work carried out studies to determine the thermal stability of briquettes obtained from microsilica and carbonaceous reducing agents. Here a method for the quantitative description of this property is proposed. This method (technique) for quartz was proposed by SMS Demag (Germany) [21].

According to this technique, quartz samples with a fraction of 20 - 30 mm are placed in a crucible and kept at 1300°C for one hour. The sample is taken out and cooled to room temperature. A fraction of + 20 mm is taken and placed in a drum with a diameter of 200 mm, a depth of 100 mm with four 17 mm ribs on the side. The drum is rotated 80 times at a rotation speed of about 40 rpm. Next, the sample is sieved on 20, 10, 4, and 2 mm sieves, and a granulometric curve is built. The temperature resistance is evaluated according to the reference curves - figure 1, developed by SMS Demag.

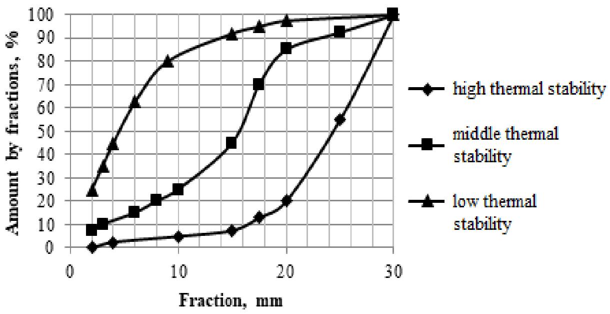

Figure 1 - Particle-size curves for assessing the thermal stability of quartz raw materials

In this work, the evaluation of the thermal stability of briquettes was carried out by a similar method, with the difference that the briquettes after thermal shock and the subsequent abrasion test were not subjected to screening, but the fraction of fines that crumbled as a result of the test relative to the main body of the briquette was estimated. The thermal shock was carried out at a temperature of 900 0C with exposure for 1 hour. After testing for abrasion on a special drum, the main body of the briquette is weighed, and the material that has fallen off the surface of the briquette during the test is collected and weighed. The thermal resistance of a briquette is defined as the ratio of the weight of the main body of the briquette after the abrasion test to the sum of the weight of the main body and the crumbled material.

It should be especially noted that the achievement of the highest thermal stability is achieved by optimizing the drying mode. Two options for removing moisture from the briquette were considered. In the first version, the briquettes were dried in a laboratory oven at a temperature of 250-2700C with high intensity and forced air blowing of the furnace space for 30 minutes from the initial moisture content of 20-25% to the residual moisture content of 1-2%. The second option is closer to practical implementation in industrial production and includes three stages. In the first stage, wet briquettes from the briquetting press are kept under natural drying conditions for about 24 hours until a residual moisture content of about 12-18%. This is necessary in order to be able to reload wet briquettes using special equipment and load them into bunkers without significant destruction of the briquette structure. Next, the briquettes are loaded into a chamber-type oven with natural air circulation in the oven space, where the briquettes are dried to a residual moisture content of 10-12% for 8 hours. After that, dried briquettes should gain strength within 5-7 days. In this case, the residual external humidity of the briquette will be about 5%. Part of the moisture in the process of setting the briquette will turn into a crystalline form.

Results and discussion

Table 1 presents the results of the thermal stability of briquettes of various compositions at various stages of the drying process, as well as in different variants of its implementation. As can be seen from the table, the final thermal strength of the briquettes is higher when drying according to the second option. In this regard, when assessing the impact of the granulometric composition of the briquette mixture and the briquetting force on the physicochemical properties of the briquettes, the data obtained as a result of the three-stage method of drying the briquettes were taken

Figure 2 shows graphs of the thermal strength of briquettes for different types of granulometric composition of the briquette charge. Analyzing the data presented in Figure 2, it should be noted that all dependencies, except for the dependence for fine mixtures, pass through a maximum. Reducing the proportion of fine fractions from 60 to 35% provides a slight shift of the maximum towards greater optimal briquetting forces from 300 to 370 kgf/cm^2. This achieves a large value of thermal stability of the briquettes - about 90%. Further reduction of fines fraction 0-1 mm in the charge to 15% leads to a sharp deterioration in the conditions of briquetting and requires an increase in effort at optimal heat resistance up to 780 kgf/cm^2. Optimum thermal stability under these conditions is reduced to 65%. When finely dispersed mixtures are used in the process of briquetting, the thermal strength of the briquettes sharply increases to a force of 300 kgf/cm^2. After this value, the growth rate decreases significantly. The dependence in this area has a monotonically increasing character up to a force value of 880 kgf/cm2. The value of thermal strength at this force is quite high, but does not exceed the option with a fine fraction of 35%. Thus, from the point of view of the thermal strength of the briquette, the optimal ratio of fractions is 0-1; 1-3; 3-5 mm in proportions 35/35/30.

Table 1 - Comparative analysis of thermal stability of briquettes, % at various stages of the drying process

Briquette composition	Drying stage			
	Drying 24 hours up to 12-18%	Drying in a chamber furnace - 8 hours up to 10-12%	Drying - 5-7 days up to 5%	Quick furnace dry 30 minutes
Special coke - 40% Microsilica - 60%	40	58	75	50
Charcoal - 10% Special coke – 20% Microsilica - 70%	32	40	82	48

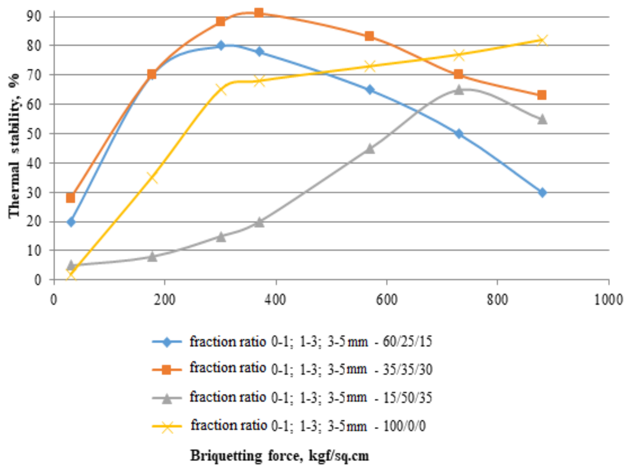

Figure 2 - The dependence of the thermal stability of briquettes on the briquetting force and particle size distribution of the briquetting charge

The presence of particles of different pore diameters in the briquette improves the gas permeability of the layer of reaction products due to the formation of macrocavities in the body of the briquette during the gasification of carbon inclusions. The negative point in this is a decrease in the thermal stability of briquettes with content of particles with a diameter of 1-3 and 1-5 mm increased by more than 30%. The briquettes subjected to the thermal stability test, formed from a charge, including microsilica, screenings of quartz, and screenings of oxidative thermocoking coke with a particle size ratio close to optimal, acquire a specific macrostructure, shown in Figure 3. They can be identified by the presence of macrocavities (pores) on the body of the briquette, the solid body of the briquette is microsilica, white inclusions are quartz, black are coke screenings.

a b

Figure 3 - Macrostructure on the fracture of a briquette of fractions 0-1; 1-3; 3-5 mm at a ratio of 35/35/30 (a - briquette at a pressure of 175 kgf / cm^2; b - briquette at a pressure of 300 kgf / cm^2)

Conclusions

Thus, the most technologically advanced granulometric composition of the briquetting charge for reduction smelting in an ore-thermal furnace was established. It is best to use briquettes with granulometric compositions of the appropriate ratio of fractions 0-1; 1-3; 3-5 mm with the following proportions 35/35/30 and 60/20/15, as well as briquettes with particle size distribution within these ranges of variation. A briquette with a finer fractional composition is characterized by greater thermal stability, which is undesirable when smelted in ore-thermal furnaces. Less fine briquette shows the opposite characteristics. Which of the factors is more significant can be determined as a result of the use of these briquettes in ore-thermal smelting.

It is also worth noting that in all cases the high thermal stability was combined with the high mechanical strength of the briquette. Therefore, this indicator was not studied in this work.

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Conflict of interest. The corresponding author declares that there is no conflict of interest.

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Микрокремнезем негізіндегі брикеттердің термиялық беріктігін зерттеу

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ТҮЙІНДЕМЕ
Осы жұмыстағы зерттеу объектісі микрокремнезем - өндірістерде техникалық кремнийді балқыту кезінде пайда болатын шаң тәрізді техногендік қалдықтар болып табылады. Бұл материалды болжамды пайдалану-қымбат кварцты пайдаланбай, техникалық кремнийдің ең жоғары сапалы брендтерін алу. Микрокремнеземді қайта өңдеу проблемасы микрокремнеземнің кенді-термиялық пештерде техникалық кремнийге тікелей өңдеу (алдын ала дайындықсыз) мүмкін болмайтын ұсақ дисперсті ұнтақ болып табылатындығына байланысты. Бұл кенді-термиялық пештерге арналған шикізат материалдарына қойылатын барлық талаптарды қанағаттандыратын оның негізінде жоғары беріктігі бар брикеттерді дайындауды талап етеді. Бұл жұмыста авторлар алынған брикеттердің физика-химиялық қасиеттерін оңтайландыру мақсатында микрокремнезем негізіндегі брикеттердің термиялық тұрақтылығын зерттеу нәтижелерін ұсынды. Әр түрлі көміртекті тотықсыздандырғыштары бар сыналатын микрокремнезем брикеттері (Термо тотықтырғыш кокстеу коксы, көмір скринингі және т.б.) ZZXM-4 ірі зертханалық роликті брикеттеу прессінде алынды. Брикеттердің термиялық беріктігін бағалау әдістеме бойынша жүргізілді, онда алынған брикеттер термиялық соққыға ұшырайды, содан кейін оларды арнайы барабанда тоздырады. Брикеттің термиялық тұрақтылығы абразивті сынақтан кейінгі брикеттің негізгі дене салмағының және құлаған материалдың қосындысына қатынасы ретінде анықталады. Сондай-ақ, термиялық тұрақтылықтың брикеттердің гранулометриялық құрамына тәуелділігі анықталған болатын. Брикеттердің қанағаттанарлық термиялық төзімділігі бар оңтайлы гранулометриялық құрамы анықталды. Егер брикеттелген шихтаның ең технологиялық гранулометриялық құрамын таңдаса, онда 0-1; 1-3; 3-5 мм фракцияларының тиісті қатынасы бар түйіршікті құрамы бар брикеттер сәйкесінше 35/35/30 және 60/20/15 пропорцияларында, сондай-ақ осы вариация шегінде түйіршікті құрамы бар брикеттер кенді-термиялық пеште тотықсыздандырғыш балқу үшін жақсырақ екенін атап өтуге болады.

Түйін сөздер: термиялық төзімділік, брикеттер, микрокремнезем, кремний брикеттері, брикеттеу, кремний қалдықтары

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Исследования термической стойкости брикетов на основе микрокремнезема

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АННОТАЦИЯ
Объектом исследования в настоящей работе является микрокремнезем - пылевидный техногенный отход, образующийся при выплавке технического кремния на производствах. Предпологаемое использование данного материла - получение в конечном результате наиболее высококачественных марок технического кремния без использования дорогостоящего кварца. Проблема переработки микрокремнезема связана с тем, что микрокремнезем представляет собой мелкодисперсный порошок, непосредственная переработка (без предварительной подготовки) которого на технический кремний невозможна в рудно-термических печах. Что требует изготовления высокопрочных брикетов на его основе, удовлетворяющих всем требованиям, предъявляемым к сырьевым материалам для рудно-термических печей. В данной работе авторами представлены результаты исследования термической стойкости брикетов на основе микрокремнезема с целью оптимизации физико-химических свойств получаемых брикетов. Испытываемые брикеты из микрокремнезема с различными углеродистыми восстановителями (отсевы кокса термоокислительного коксования, отсевы древесного угля и др.) были получены на крупно-лабораторном валковом брикетировочном прессе ZZXM-4. Оценку термической стойкости брикетов выполняли по методике, в которой полученные брикеты подвергаются термическому удару с последующим их истиранием на специальном барабане. Термическая стойкость брикета определяется как отношение веса основного тела брикета после испытания на истираемость к сумме веса основного тела и осыпавшегося материала. Также была определена зависимость термической стойкости от гранулометрического состава брикетов. Определен оптимальный гранулометрический состав, с которым брикеты обладают удовлетворительной термической стойкостью. Если выбирать наиболее технологичный гранулометрический состав брикетированной шихты, то можно отметить, что лучше подходят к восстановительной плавке в рудно-термической печи брикеты с гранулометрическими составами соответствующим соотношением фракций 0-1; 1-3; 3-5 мм в следующих пропорциях 35/35/30 и 60/20/15 соответственно, а также брикеты с гранулометрическим составом в этих пределах варьирования.

Ключевые слова: термическая стойкость, брикеты, микрокремнезем, кремниевые брикеты, брикетирование, кремниевые отходы

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