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PHYSICAL AND CHEMICAL PROCESSES DURING NITRIDING OF CHROMIUM FERROSILICON BY FILTRATION COMBUSTION

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Abstract. In this paper, the nitriding of chromium ferrosilicon is carried out in the combustion mode under the condition of natural nitrogen filtration. The authors studied the effect of the key parameters (pressure of gaseous nitrogen, diameter and dispersity of starting samples) on the maximum temperature and combustion of the starting powder mixture based on chromium ferrosilicon. The combustion synthesis of chromium ferrosilicon proceeds steadily in the stationary mode with formation of a macrohomogeneous nitrided composition which, according to the results of *X*-ray phase analysis, contains two nitride phases - chromium nitride and silicon nitride. Interaction of the initial powder with gaseous nitrogen in the filtration combustion mode proceeds by the following probable chemical reaction: $3CrSi_2 + 3Si + 3FeSi_2 + 11.5N_2 = 3CrN + 5Si_3N_4 + 3Fe$. Increasing the diameter of the starting samples slightly affects the amount of absorbed nitrogen and slows the propagation of the combustion wave front. An increase in the pressure of gaseous nitrogen increases the amount of absorbed nitrogen and the combustion rate. Increasing the dispersity of the starting powder increases the amount of absorbed nitrogen and the combustion rate. It was found that the combustion reaction is not possible with a dense initial sample. The maximum combustion temperature, depending on the nitriding conditions, varies between 2400 and 2650 °C and increases with increasing gaseous nitrogen pressure, diameter of the initial samples and dispersion of chromium ferrosilicon powder. It is possible to realise nitriding of chrome ferrosilicon in the combustion mode at the pressure of gaseous nitrogen not less than 3 MPa, diameter of initial samples not less than 3.5 cm and size of initial particles not more than 100 μm. Optimal parameters of nitriding are gaseous nitrogen pressure of 5 MPa, diameter of samples 5 cm, size of initial particles less than 100 μm and bulk density of samples (2.23 g/cm^3).

Keywords: self-propagating high-temperature synthesis, combustion synthesis, nitriding, nitrides, ferroalloy, powder metallurgy

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ФИЗИКО-ХИМИЧЕСКИЕ ПРОЦЕССЫ АЗОТИРОВАНИЯ ФЕРРОСИЛИКОХРОМА В РЕЖИМЕ ФИЛЬТРАЦИОННОГО ГОРЕНИЯ

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Аннотация. В работе изучены процессы азотирования ферросиликохрома в режиме горения в условиях естественной фильтрации азота и представлены результаты исследования влияния основных параметров синтеза (давление газообразного азота, диаметр образцов и размер исходных частиц) на максимальную температуру и процесс горения исходной порошковой шихты. Горение ферросиликохрома протекает устойчиво в стационарном режиме с образованием макрооднородной азотированной композиции, которая по результатам рентгенофазового анализа содержит в своём составе две нитридные фазы – нитрид хрома и нитрид кремния. Взаимодействие исходного порошка с газообразным азотом в режиме фильтрационного горения протекает по следующей вероятной химической реакции: $3CrSi_2 + 3Si + 3FeSi_2 + 11,5N_2 = 3CrN + 5Si_3N_4 + 3Fe$. Увеличение диаметра исходных образцов незначительно влияет на количество поглощеного азота и приводит к замедлению продвижения фронта волны горения. При повышении давления газообразного реагента наблюдается увеличение количества поглощенного азота и скорости горения. Более тонкое измельчение исходного порошка позволяет увеличить количество поглощенного азота и скорость горения. Определено, что при уплотнении исходного образца реализовать реакцию горения невозможно. Максимальная температура горения в зависимости от условий азотирования изменяется в пределах от 2400 до 2650 °C и повышается при увеличении давления газообразного азота, диаметра исходных образцов и дисперсности порошка ферросиликохрома. Реализовать азоти-

рование ферросиликохрома в режиме горения возможно при давлении газообразного азота не менее 3 МПа, диаметре исходных образцов не менее 3,5 см и размере исходных частиц не более 100 мкм. Оптимальными параметрами азотирования ферросиликохрома является давление газообразного азота 5 МПа, диаметр образцов 5 см, размер исходных частиц менее 100 мкм и насыпная плотность порошка 2,23 г/см^3.

Ключевые слова: самораспространяющийся высокотемпературный синтез, фильтрационное горение, азотирование, нитриды, ферросплав, порошковая металлургия

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Introduction

The method of self-propagating high-temperature synthesis (SHS) is based on highly exothermic reactions that occur in the form of a combustion wave in a self-propagating mode. The SHS method has undeniable advantages: energy efficiency, short synthesis time, environmental friendliness, and simplicity of equipment [1 – 3].

Currently, a large number of materials, particularly nitrides, have been obtained by the filtration SHS method in a nitrogen environment [4 – 6]. Several nitride materials possess unique physicochemical properties [7 – 9]. They can be used in the production of gas turbine components [10], heat-dissipating radiators [11], cutting tools [12; 13], photocatalysts [14], semiconductors [15], and so on.

The most promising application in the filtration SHS method is the use of accessible and relatively inexpensive ferroalloys. Using ferroalloys in SHS processes can produce nitride material at a relatively low cost without losing product quality [16; 17]. Iron, which is part of the ferroalloys, has a catalytic effect on the nitriding process of other elements included in the initial mixture [18]. Thus, iron increases the intensity and depth of nitriding of the initial material. There is a considerable amount of work dedicated to the filtration combustion of simple ferroalloys. The patterns of nitriding of ferrosilicon [19 – 21], as well as the filtration combustion of ferrochrome and ferrovanadium [22], have been thoroughly studied. The study [23] investigated the combustion of industrial ferrotitanium in nitrogen. The monograph [24] described the nitriding of ferroboron and ferroniobium in a combustion mode.

However, the use of complex ferroalloys in filtration combustion processes is interesting and little studied. Complex ferroalloys are alloys of iron with two or more elements. At present, SHS combustion of ferro-silicoaluminium [25] and ferro-aluminum-silicon-zirconium [2] has been studied.

The aim of this work was to study the combustion processes of chromium ferrosilicon in a self-propagating mode under conditions of natural nitrogen filtration to obtain a nitride-containing composite material based on chromium nitride and silicon nitride.

Materials and methods

Chromium ferrosilicon (CFS) was used as the starting material. *X*-ray phase analysis showed that this ferroalloy is multiphase and contains $CrSi_2$, Si, and $FeSi_2$ (Fig. 1). According to the chemical analysis, the composition of CFS is as follows (wt. %): 49.4 Si, 29.7 Cr, 20.7 Fe, and the rest are oxides. For nitriding in the self-propagating mode, the initial CFS was ground in a ball mill and dried in a vacuum drying oven at a temperature of 150 °C for 3 h.

Nitriding of the initial CFS was carried out in a constant pressure setup with a volume of 3 liters. For the synthesis, the initial powder mixture was placed in a gas-permeable container mounted on a non-conductive stand. An igniting composition was poured over the initial charge. A coil was connected to the igniting composition to conduct an electric pulse from a transformer. After the electric pulse was applied, the combustion reaction of the igniting composition was initiated. Then, the heat released as a result of the combustion of the igniting composition initiated the combustion reaction of the initial CFS powder. After the combustion wave front passed and complete cooling occurred, the unreacted nitrogen was vented, and the nitrided samples were removed for further physicochemical studies.

The phase composition was studied using a Shimadzu XRD-6000 diffractometer. The oxygen and nitrogen contents were determined using a LEKO-ONH 836 instrument. The maximum combustion temperature was measured using the thermocouple method with tungsten-rhenium thermocouples (WRe5-WRe20) on an LA20USB.

Results and discussion

Combustion of chromium ferrosilicon proceeds in a stationary mode. The nitrided samples obtained based

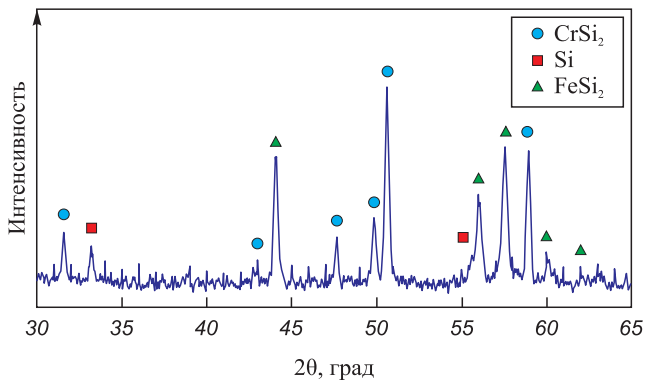

Fig. 1. *X*-ray diffraction pattern of chromium ferrosilicon

Рис. 1. Рентгеновская дифрактограмма ферросиликохрома

on CFS are macrohomogeneous. An image of the nitrided CFS is shown in Fig. 2.

The probable chemical reactions of the interaction of the initial charge based on CFS with nitrogen are given below:

$$3CrSi_2 + 5.5N_2 = 3CrN + 2Si_3N_4; \quad (1)$$

$$3Si + 2N_2 = Si_3N_4; \quad (2)$$

$$3FeSi_2 + 4N_2 = 2Si_3N_4 + 3Fe. \quad (3)$$

The overall chemical reaction equation is as follows:

$$3CrSi_2 + 3Si + 3FeSi_2 + 11.5N_2 = \\ = 3CrN + 5Si_3N_4 + 3Fe. \quad (4)$$

Reaction (4) corresponds to the complete nitriding of the initial CFS (with a conversion degree of 1). Due to the rapid processes of SHS, the initial charge is in the reaction zone for a relatively short time and does not fully react with nitrogen. It is theoretically calculated that the maximum amount of absorbed nitrogen by chromium ferrosilicon is 28.99 %.

The nitrided CFS product is a multiphase material containing β-Si_3N_4, α-Fe, CrN, Cr and $CrSi_2$. The presence of Cr and $CrSi_2$ indicates the incomplete nitriding reaction of the initial powder (Fig. 3).

Parameters such as the pressure of the gaseous reactant, sample diameter, particle size, and density of the initial material significantly influence the maximum temperature, process, and feasibility of filtration combustion in a self-propagating mode.

Fig. 2. Sample of nitrided chromium ferrosilicon

Рис. 2. Образец азотированного ферросиликохрома

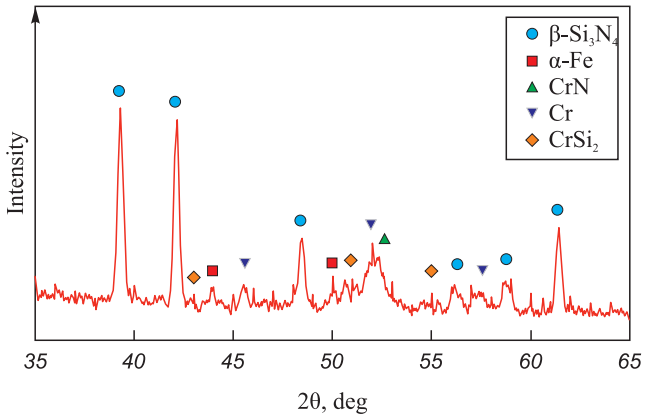

Fig. 3. *X*-ray diffraction pattern of nitrided chromium ferrosilicon

Рис. 3. Рентгеновская дифрактограмма азотированного ферросиликохрома

Fig. 4 shows the dependence of the amount of absorbed nitrogen and the combustion rate on the diameter of the initial samples. The influence of the diameter was studied in the range of 35 to 65 mm. Combustion of CFS can be initiated with initial sample diameters of at least 35 mm. Increasing the diameter slightly affects the amount of absorbed nitrogen and leads to a decrease in the combustion rate from 0.11 to 0.021 mm/s. This slight change in the amount of absorbed nitrogen is due to the increased difficulty of nitrogen filtration reaching the reaction zone as the diameter increases. At the same time, due to the slowdown in the combustion wave front, the residence time of the initial CFS particles in the reaction zone increases. The slowdown of the combustion wave front is related to the increased volume of the powder mixture, which

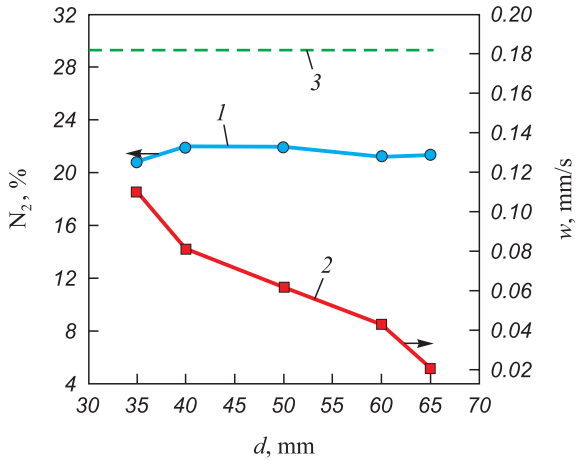

Fig. 4. Dependence of the content of absorbed nitrogen (*1*) and combustion rate (*2*) on diameter of the starting samples (*3* – theoretically calculated maximum amount of absorbed nitrogen) at P = 5 MPa, D > 100 μm and ρ = 2.23 g/cm^3

Рис. 4. Зависимость количества поглощенного азота (*1*) и скорости горения (*2*) от диаметра образцов (*3* – теоретически рассчитанное максимальное количество поглощенного азота) при P = 5 МПа, D > 100 мкм и ρ = 2,23 г/см^3

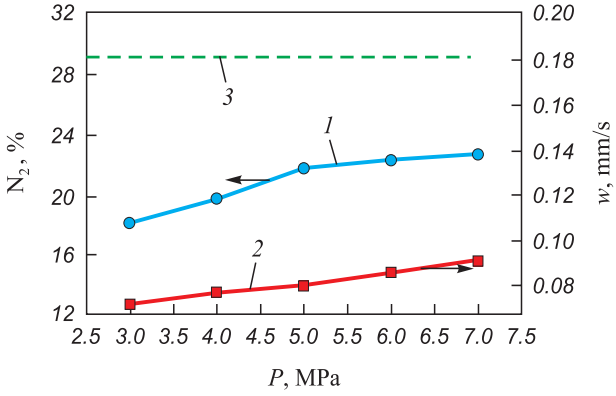

Fig. 5. Dependence of the content of absorbed nitrogen (*1*) and combustion rate (*2*) on pressure of gaseous nitrogen (*3* – theoretically calculated maximum amount of absorbed nitrogen) at d = 50 mm, ρ = 2.23 g/cm^3 and D > 100 μm

Рис. 5. Зависимость количества поглощенного азота (*1*) и скорости горения (*2*) ферросиликохрома от давления газообразного азота (*3* – теоретически рассчитанное максимальное количество поглощенного азота) при d = 50 мм, ρ = 2,23 г/см^3 и D > 100 мкм

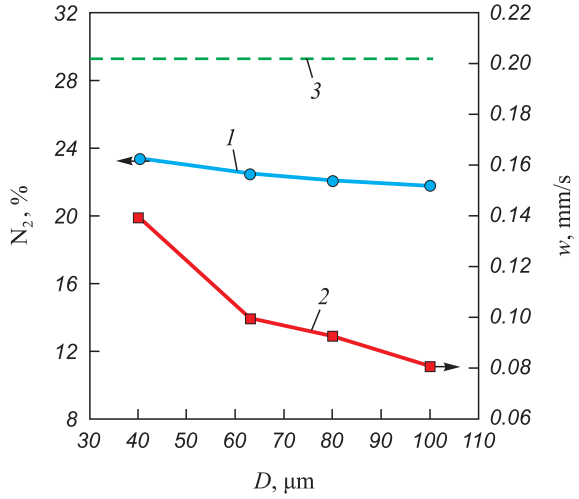

Fig. 6. Dependence of the content of absorbed nitrogen (*1*) and combustion rate (*2*) on dispersity of the starting powder (*3* – theoretically calculated maximum amount of absorbed nitrogen) at d = 50 mm, P = 5 MPa and ρ = 2.23 g/cm^3

Рис. 6. Зависимость количества поглощенного азота (*1*) и скорости горения (*2*) от дисперсности порошка ферросиликохрома (*3* – теоретически рассчитанное максимальное количество поглощенного азота) при d = 50, P = 5 МПа и ρ = 2,23 г/см^3

requires a large amount of heat for heating. When changing the diameter, the maximum amount of absorbed nitrogen was about 22 %, which is 6.99 % less than the theoretically calculated amount of nitrogen absorption. As the diameter of the initial powder mixture increases, the maximum combustion temperature of CFS changes from 2400 to 2650 °C.

In a laboratory setup with a volume of 3 liters, it is preferable to implement combustion of samples with a diameter of 50 mm.

Increasing the pressure accelerates the filtration of gaseous nitrogen and, accordingly, increases the concentration of the reagent gas in the chemical reaction zone. Combustion of CFS at a nitrogen pressure of less than 3 MPa could not be achieved. Increasing the pressure of gaseous nitrogen leads to an increase in the amount of absorbed nitrogen from 18.3 to 22.8 % and the combustion rate from 0.073 to 0.092 mm/s. In the nitrogen pressure range from 5 to 7 MPa, the change in the amount of absorbed nitrogen becomes less pronounced. Increasing the nitrogen pressure above 7 MPa is not advisable because, at 5 MPa, the influence of pressure on the combustion process becomes insignificant (Fig. 5). When implementing CFS combustion at a pressure of 7 MPa, the sample contains 6.29 % less nitrogen than the theoretically calculated value. As the pressure of gaseous nitrogen increases from 3 to 7 MPa, the maximum combustion temperature rises from 2350 to 2600 °C.

Combustion of CFS with particle sizes greater than 100 μm and without fine fractions (less than 63 μm) could not be achieved. As the particle size of the initial material decreases, the amount of absorbed nitrogen increases from 21.9 to 23.5 % and the combustion rate from 0.081 to 0.140 mm/s. The increase in the dispersity of the initial material leads to an increase in the specific surface area capable of reacting (Fig. 6). The reduction in particle size of CFS results in an increase in the maximum temperature from 2400 to 2490 °C.

The increase in the density of the initial powder mixture was achieved by pressing the initial powder into tablets with a diameter and height of 40 mm in molds. Combustion of the pressed powder, which retains the shape of the tablet (ρ = 2.52 g/cm^3), could not be achieved. Therefore, only samples with a bulk density (2.23 g/cm^3) were used.

Conclusions

The combustion of chromium ferrosilicon proceeds in a stationary mode, resulting in homogeneous nitrided samples without melt droplets or cracks.

Increasing the diameter leads to a decrease in the combustion rate from 0.11 to 0.021 mm/s and slightly affects the amount of absorbed nitrogen. Increasing the nitrogen pressure results in an increase in the amount of absorbed nitrogen (18.3 – 22.8 %) and the combustion rate (0.073 – 0.092 mm/s) of chromium ferrosilicon. Reducing the dispersity of the initial material allows for an increase in the amount of absorbed nitrogen from 21.9 to 23.5 % and the combustion rate from 0.081 to 0.140 mm/s. A slight increase in the density of the initial powder prevents achieving the combustion reaction of chromium ferrosilicon in a nitrogen environment.

The maximum combustion temperature increases with a rise in gaseous nitrogen pressure from 3 to 7 MPa (from 2350 to 2600 °C), the diameter of the initial samples from

35 to 65 mm (from 2400 to 2650 °C), and a decrease in the particle size of the initial charge from less than 100 to less than 40 μm (from 2400 to 2490 °C).

Stable combustion in the self-propagating mode of chromium ferrosilicon powder samples is possible at a nitrogen pressure of at least 3 MPa, a sample diameter of at least 3.5 cm, a particle size of less than 100 μm with the presence of fine fractions (less than 63 μm), and a sample density not exceeding 2.23 g/cm^3. It is optimal to carry out nitriding of the initial chromium ferrosilicon under conditions of natural nitrogen filtration at a pressure of 5 MPa, a sample diameter of 5 cm, a particle size of the initial material less than 100 μm, and a bulk sample density of 2.23 g/cm^3.

The product of chromium ferrosilicon nitriding contains β-Si_3N_4, CrN, α-Fe, Cr and $CrSi_2$. The presence Cr and $CrSi_2$ indicates the incomplete nitriding reaction of the initial chromium ferrosilicon. The nitrogen saturation is 21.9 %, which is 7.09 % less than the theoretically calculated maximum amount of absorbed nitrogen.

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