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Research article

Научная статья



Azide self-propagating high-temperature synthesis of a highly dispersed AlN–SiC powder composition using polytetrafluoroethylene

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Abstract. Composite ceramics from aluminum nitride with silicon carbide (AlN–SiC) is promising for applications in both metallurgy and mechanical engineering as a refractory and structural material with enhanced properties, as well as in electronics and photonics as an advanced material for creating various high-performance devices. To fabricate products with optimal properties, compositions (mixtures) of highly dispersed AlN and SiC powders with particle sizes of less than 1 μm must be used. This study is dedicated to improving a simple, energy-efficient method of azide self-propagating high-temperature synthesis (SHS) for such powder compositions, using mixtures of sodium azide (NaN_3) powder and elemental powders of aluminum, silicon, and carbon with the addition of polytetrafluoroethylene (PTFE) powder as an activating and carbidizing additive. During the combustion of these mixtures in a bulk or pressed form in a reactor under 3 MPa of nitrogen gas pressure, the temperature, pressure, and yield of solid combustion products were evaluated. Scanning electron microscopy and X-ray phase analysis were employed to determine the microstructure and phase composition of the combustion products. The addition of PTFE helped to eliminate, in most cases, the drawbacks of the traditional azide SHS approach using halide salts such as $(\text{NH}_4)_2\text{SiF}_6$, AlF_3 , and NH_4F . While maintaining the high dispersity of the synthesized AlN–SiC powder compositions, their phase composition, particularly in pressed charges, became significantly closer to the targeted theoretical composition, with a substantial increase in SiC phase content and the elimination of undesirable by-products such as silicon nitride and the water-insoluble cryolite salt Na_3AlF_6 .

Keywords: aluminum nitride, silicon carbide, powder compositions, self-propagating high-temperature synthesis, sodium azide, polytetrafluoroethylene, combustion products, composition, structure

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Азидный самораспространяющийся высокотемпературный синтез высокодисперсной порошковой композиции AlN–SiC с применением политетрафторэтилена

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Аннотация. Композиционная керамика из нитрида алюминия с карбидом кремния (AlN–SiC) является перспективной для применения как в металлургии и машиностроении в качестве огнеупорного и конструкционного материала с повышенными свойствами, так и в электронике и фотонике в качестве нового материала для создания соответствующих разнообразных высокоэффективных устройств. Для изготовления из нее изделий с наилучшими свойствами необходимо использовать композиции (смеси) высокодисперсных порошков AlN и SiC с размером частиц менее 1 мкм. Настоящая работа посвящена усовершенствованию простого энергосберегающего метода азидного самораспространяющегося высокотемпературного синтеза (СВС) композиций таких порошков из смесей порошка азид натрия (NaN_3) и элементных порошков алюминия, кремния и углерода за счет применения активирующей и карбидизирующей добавки порошкового политетрафторэтилена (ПТФЭ). При сжигании этих смесей в насыпном или прессованном виде в реакторе с давлением газообразного азота 3 МПа оценивались температура и давление, а также выход твердых продуктов горения. С применением сканирующей электронной микроскопии и рентгенофазового анализа определялись микроструктура и фазовый состав продуктов горения. Применение добавки ПТФЭ позволило устранить в большинстве случаев недостатки традиционного подхода азидного СВС с использованием галоидных солей $(\text{NH}_4)_2\text{SiF}_6$, AlF_3 и NH_4F . При сохранении высокой дисперсности синтезированных композиций порошков AlN–SiC их фазовый состав, особенно в случае прессованных шихт, стал значительно ближе к задаваемому теоретическому составу, также существенно выросло содержание фазы SiC, исчезли нежелательные побочные фазы нитрида кремния и водонерастворимой соли криолита Na_3AlF_6 .

Ключевые слова: нитрид алюминия, карбид кремния, композиции порошков, самораспространяющийся высокотемпературный синтез, азид натрия, политетрафторэтилен, продукты горения, состав, структура

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Introduction

Aluminum nitride (AlN) is one of the foremost materials in technical ceramics [1]. It exhibits an exceptional combination of physical, electrical, and chemical properties: low density, high melting (or decomposition) temperature, thermal conductivity, electrical resistance, hardness, strength, wear resistance, and heat resistance, as well as resistance to thermal shock, acids, and molten metals, and stability at elevated temperatures in various gaseous environments. Owing to these characteristics, aluminum nitride has long found applications across diverse industrial sectors, particularly for high-temperature applications. It is widely used as a refractory material for lining tanks, electrolyzers, and containers in metallurgy and chemical engineering, as well as for producing protective sheaths for metallic thermocouples and manufacturing crucibles. AlN ceramics are among the most widely used electrical

insulating materials. Furthermore, aluminum nitride is employed as a structural material for parts operating in aggressive environments and in cutting tools [2]. Intensive research to enhance its physical and mechanical properties continues [3].

However, due to the unique physical properties of AlN, its application in electronics and photonics has recently expanded rapidly [4; 5]. In electronics, this expansion is driven by its excellent heat dissipation capabilities in electronic devices, attributed to its uniquely high thermal conductivity combined with high electrical resistance and a coefficient of thermal expansion (CTE) close to that of silicon. This has led to a transition to aluminum nitride in nearly all areas of electronic component production, where the highly toxic beryllium oxide was traditionally used [4]. In photonics, AlN's wide bandgap, broad transparency window (covering the range from ultraviolet to mid-infrared), and significant second-order nonlinear optical

effect have further broadened its utility. Additionally, AlN exhibits piezoelectric and pyroelectric effects, enabling its use in opto-mechanical devices and pyroelectric photodetectors, respectively [5]. However, in both these fields and in high-temperature structural applications, the use of aluminum nitride is constrained by its brittleness, specifically its relatively low fracture toughness and thermal shock resistance [6].

In this regard, significant attention is being drawn to the development of composite ceramics of aluminum nitride with silicon carbide (SiC), which, in addition to its high thermal conductivity and heat resistance, possesses substantially improved mechanical properties (hardness, fracture toughness, thermal stability, and creep resistance) [7; 8]. Silicon carbide is also attractive due to its crystal structure, which is similar to that of aluminum nitride, allowing it to form a single-phase homogeneous solid solution with AlN, enhancing flexural strength and fracture toughness, thereby reducing brittleness [9; 10]. Additionally, in a two-phase state with sintered, spatially separated powder components of AlN and SiC, AlN–SiC composites exhibit significantly better toughness and thermal stability [8; 11]. By controlling the grain size of AlN and SiC, it is possible to obtain AlN–SiC material with high thermal conductivity [12]. Notably, reducing grain size is another important approach to improving nearly all properties of AlN ceramics and AlN–SiC ceramic composites as a whole [13; 14].

AlN–SiC composite ceramics have shown promise not only for applications in metallurgy and mechanical engineering as a refractory and structural material with enhanced properties but also, as noted above, in electronics and photonics for the creation of a variety of high-performance devices [4; 5; 15].

Traditional energy-intensive methods for producing AlN–SiC ceramics include pressureless sintering of SiC and AlN ceramic powders, hot pressing, and injection molding, all of which require temperatures of around 2000 °C and prolonged holding times of several hours [16–19]. More modern and less energy-intensive methods include spark plasma sintering, microwave heating, and additive 3D-printing technologies; however, these require costly equipment [20–22].

In both methods, the starting material must be a composition (mixture) of AlN and SiC powders. To achieve the best properties of the resulting AlN–SiC ceramics, these powders should be as fine as possible: highly dispersed (submicron) with particle sizes $d < 1 \mu\text{m}$ or even nanoscale with $d < 100 \text{ nm}$ (0.1 μm) [13; 23]. There are two approaches to producing AlN and SiC powder mixtures: *ex-situ* and *in-situ*. The first approach is the simplest and most common for producing com-

posite ceramics and involves mixing ready-made AlN and SiC powders, compacting, and sintering them. However, for highly dispersed powders, especially nanopowders, two issues arise: high cost and the near impossibility of achieving uniform mechanical mixing due to the strong tendency of nanoparticles to form durable agglomerates that are challenging to break up during mixing. The *in-situ* processes for producing composite ceramics involve the chemical synthesis of AlN and SiC powder particles within the composite from a mixture of significantly cheaper starting reagents, allowing for better dispersion of the synthesized particles. These technologies are thus more favorable for obtaining mixtures of highly dispersed and nanoscale AlN and SiC powders [13; 23].

Traditional *in-situ* methods for producing mixtures of AlN and SiC powder components, or their solid solutions (furnace method, plasma-chemical synthesis, carbothermal synthesis, gas-phase deposition, etc.), are known for their high energy consumption, complex equipment requirements, and inconsistent ability to yield nanoscale powders and nanopowder compositions [10; 13; 14; 16; 24–26].

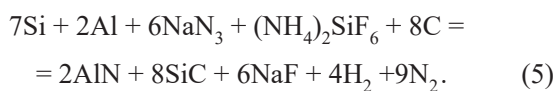
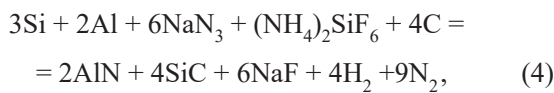
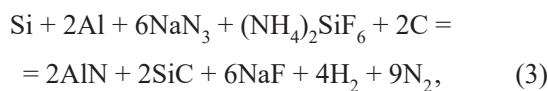
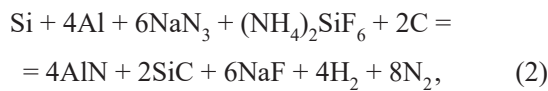
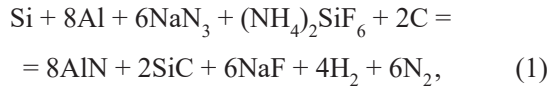
Self-propagating high-temperature synthesis (SHS), or “combustion synthesis”, presents distinct advantages over conventional technologies [16; 27–29]. The *in-situ* SHS approach is notably more cost-effective, as the AlN–SiC composite synthesis is driven by self-sustained combustion heat, requiring only simple equipment and low-cost precursor reagents, such as Al, Si, C (carbon black), Si_3N_4 powders, and gaseous N_2 . This method has therefore garnered significant research interest, with various powder mixtures being examined for their combustion efficiency in producing AlN–SiC composites [27; 30–33]. A review of these studies [34] reveals that, in all cases, the synthesized AlN–SiC ceramics exhibit micron-sized particle structures.

To achieve a composition of highly dispersed ($d < 1 \mu\text{m}$) AlN–SiC powders, the authors investigated a variant of synthesis known as azide SHS technology, where sodium azide (NaN_3) powder serves as the nitriding agent. Additionally, various activating halide salts are introduced alongside elemental reagents, promoting relatively low combustion temperatures, the formation of significant intermediate vapor-gas reaction products, and the generation of condensed and gaseous by-products that effectively separate target powder particles, thus preventing their agglomeration into larger particles [34–37]. The main findings of these investigations are summarized as follows.

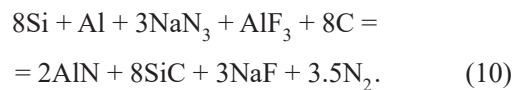
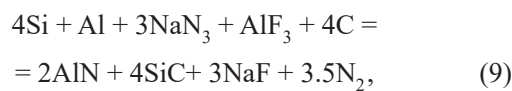
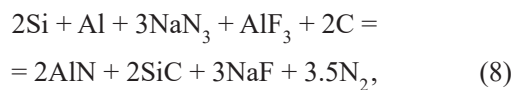
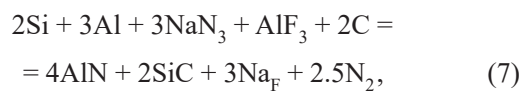
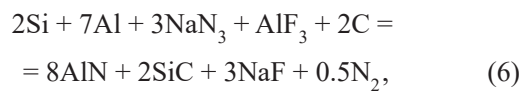
Azide SHS technology was employed to synthesize AlN–SiC powder compositions at five molar ratios of the target phases aluminum nitride and silicon car-

bide AlN:SiC = 4:1, 2:1, 1:1, 1:2, and 1:4, according to stoichiometric equations involving the use of halide salts $(\text{NH}_4)_2\text{SiF}_6$, AlF_3 and NH_4F [37].

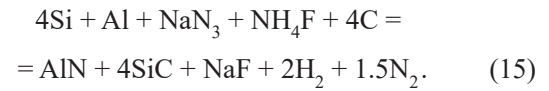
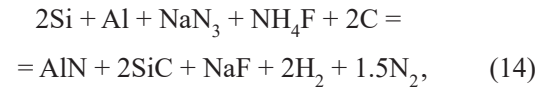
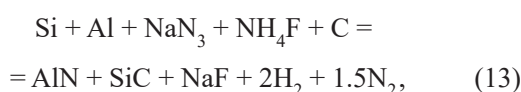
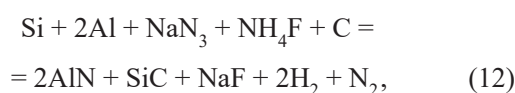
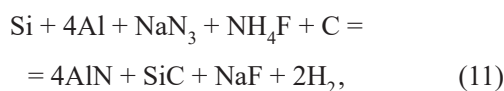
$x\text{Si}-y\text{Al}-\text{NaN}_3-(\text{NH}_4)_2\text{SiF}_6-(x+1)\text{C}$ system



$x\text{Si}-y\text{Al}-\text{NaN}_3-\text{AlF}_3-x\text{C}$ system



$x\text{Si}-y\text{Al}-\text{NaN}_3-\text{NH}_4\text{F}-x\text{C}$ system



The initial reagent mixtures (charges) from equations (1)–(15) were burned in bulk form with a relative density of 0.4 in a tracing paper cup with a diameter of 30 mm and a height of 45 mm inside a 4.5 L azide SHS reactor at a nitrogen gas pressure of 4 MPa. The cooled combustion product was removed from the reactor, crushed to a loose powdered state in a porcelain mortar, and washed with water to remove the by-product sodium fluoride (NaF). In most cases, the combustion product consisted of a highly dispersed powder of complex composition, appearing as equiaxed submicron particles with sizes ranging from 100 nm to 1 μm and fibers with diameters of 50–500 nm and lengths up to 5 μm , with a tendency toward an increased proportion of finer particles as the SiC content in the AlN–SiC composition increased. This is illustrated in Fig. 1 for the system with the halide salt NH_4F , and in study [34] for the system with $(\text{NH}_4)_2\text{SiF}_6$. The results comparing the phase composition of the washed solid combustion products from charges (1)–(15) (determined experimentally) with the theoretical composition of the target phases expected according to the stoichiometric equations (1)–(15) are presented in Table 1. As shown, the experimental phase composition of the azide SHS products significantly deviates from the expected theoretical composition of the AlN–SiC powder compositions. First, the content of the target phases AlN and SiC is lower than theoretically expected, particularly the SiC phase, whose quantity is on average half of the theoretical amount. Second, an undesirable side phase of silicon nitride in α - and β -modifications is present in considerable amounts (up to 22.1 and 15.2 wt. %, respectively). Third, there is a noticeable amount (from 4.1 to 9.8 wt. %) of an unwanted water-insoluble impurity, the cryolite salt Na_3AlF_6 .

In this context, the aim of this study was to bring the experimental composition of the target highly dispersed AlN and SiC powder mixture closer to the theoretical composition according to the stoichiometric equations of azide SHS Technology by adjusting the composition of the initial reagents and the conditions of the azide SHS process. Primarily, it is essential to significantly increase the content of the SiC carbide phase in the azide SHS product. Several approaches are known [38] to promote the formation of SiC during the combustion of a silicon and carbon black powder mixture via the weakly exothermic reaction

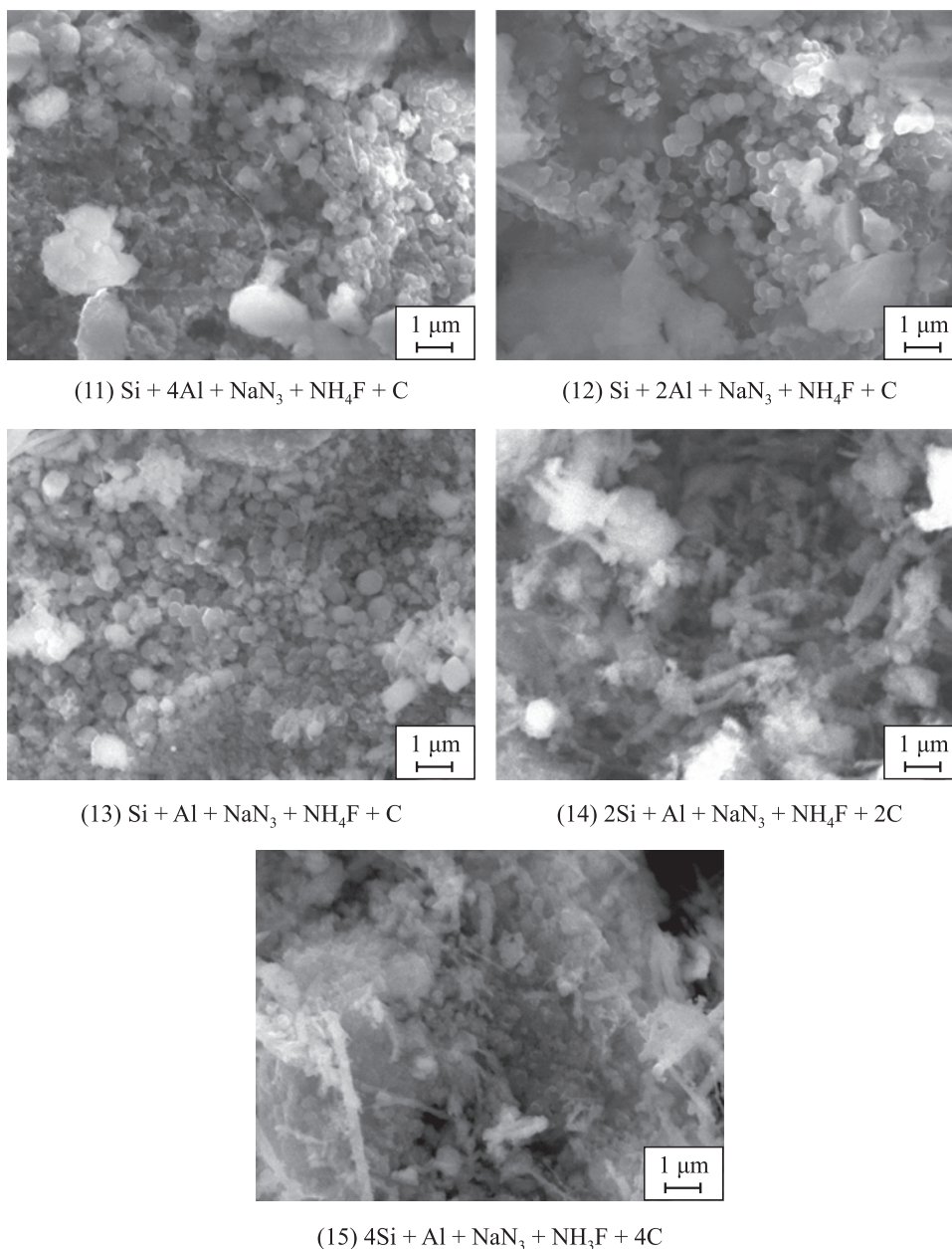


Fig. 1. SEM images of combustion products of charges with NH_4F according to equations (11)–(15)

Рис. 1. Микроструктура продуктов горения шихт с NH_4F в соответствии с уравнениями (11)–(15)



- preheating the charge;
- applying an electric field;
- mechanical activation of the charge;
- conducting combustion in a gaseous nitrogen or air atmosphere;
- chemical activation with catalytic powder additives.

Most of these approaches can lead to submicron or even nanoscale SiC powders in the combustion mode. In this study, the simplest approach was chosen – using

the most effective catalytic additive, powdered polytetrafluoroethylene (PTFE) [39; 40].

In the first study on this topic, the chemical transformation mechanism and combustion modes in the silicon–carbon–PTFE system were investigated, depending on the ratios of the starting components, the pressure of the inert atmosphere, ignition source temperature, and sample diameter [39]. Subsequently, combustion in this system was studied in a nitrogen atmosphere at varying pressures to produce SHS composite ceramic powders of Si_3N_4 –SiC and silicon carbide [41]. It was found that without PTFE, the synthesized Si_3N_4 –SiC composites contained between 5 and 60 wt. % SiC, whereas the use

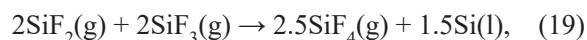
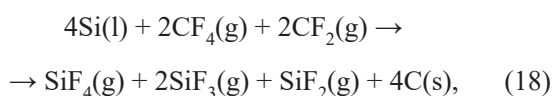
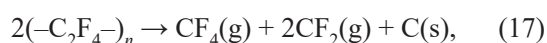
Table 1. Theoretical and experimental phase composition of washed solid products of azide SHS

Таблица 1. Теоретический и экспериментальный фазовые составы промытых твердых продуктов азидного СВС

Reaction equation	AlN:SiC (mol)	Content, wt. %						
		Theoretical		Experimental				
		AlN	SiC	AlN	SiC	α -Si ₃ N ₄	β -Si ₃ N ₄	Na ₃ AlF ₆
$x\text{Si}-y\text{Al}-\text{NaN}_3-(\text{NH}_4)_2\text{SiF}_6-(x+1)\text{C}$								
(1)	4:1	80.4	19.6	69.3	8.5	8.0	7.0	7.2
(2)	2:1	67.2	32.8	54.6	10.3	13.2	12.1	9.8
(3)	1:1	50.6	49.4	38.0	19.2	22.1	13.2	7.5
(4)	1:2	33.8	66.2	29.9	28.0	21.4	14.0	6.7
(5)	1:4	20.4	79.6	18.5	42.1	19.6	15.2	4.6
$x\text{Si}-y\text{Al}-\text{NaN}_3-\text{AlF}_3-x\text{C}$								
(6)	4:1	80.4	19.6	73.6	8.2	5.7	8.4	4.1
(7)	2:1	67.2	32.8	58.0	17.9	11.2	7.4	5.5
(8)	1:1	50.6	49.4	47.0	24.6	13.0	9.1	6.3
(9)	1:2	33.8	66.2	23.9	34.8	19.7	15.0	6.6
(10)	1:4	20.4	79.6	13.5	47.2	21.4	12.6	5.3
$x\text{Si}-y\text{Al}-\text{NaN}_3-\text{NH}_4\text{F}-x\text{C}$								
(11)	4:1	80.4	19.6	72.5	7.9	8.2	7.1	4.3
(12)	2:1	67.2	32.8	64.0	12.9	9.6	8.5	5.0
(13)	1:1	50.6	49.4	39.0	25.0	18.1	11.9	6.0
(14)	1:2	33.8	66.2	25.4	33.5	21.2	13.6	6.3
(15)	1:4	20.4	79.6	14.4	46.2	21.9	12.3	5.2

of an activating additive at 5–15 wt. % increased the SiC content to 100 %. Study [42] demonstrated that partially replacing carbon with PTFE led to complete carbidizing in the reaction $\text{Si} + 0.9\text{C} + 0.05\text{C}_2\text{F}_4 = \text{SiC} + 0.1\text{F}_2$ in a nitrogen atmosphere at 3 MPa, producing SiC particles with an average size of around 200 nm. Study [43] showed that PTFE can be used not only as an activating additive but also as a carbidizing reagent when fully replacing technical carbon during combustion of a bulk Si + PTFE mixture in gaseous argon at 0.5 MPa, resulting in the synthesis of silicon carbide in the form of fibers with diameters of 100–500 nm and equiaxed particles of 0.5–3.0 μm , which aggregate into clusters. However, the yield of SiC synthesis was very low: only about 10 % of the total charge mass reacted to form fibrous SiC, with the remainder consisting of unreacted carbon and silicon [43].

The reactions for silicon carbide formation involving polytetrafluoroethylene $(-\text{C}_2\text{F}_4-)_n$ can be represented as follows [43; 44]:



The first stage (17) involves the exothermic decomposition of PTFE in the preheating zone into gaseous fluorides and solid carbon particles. The intermediate stages (18) and (19) represent interactions between the gaseous fluorides with each other and with molten silicon particles, while the final stage (20) depicts the reaction between silicon and carbon particles (both the original carbon in the form of carbon black and the carbon generated from PTFE decomposition) to form the target silicon carbide. The completion of all stages results in the formation of highly dispersed SiC, whereas only the first stage yields gaseous fluorides and carbon black particles [43]. Combustion involving PTFE occurs at a high rate with intense gas release, which may cause dispersion of the charge components, preventing the silicon and carbon particles from reacting with each other, a reaction that is unlikely to proceed in the gas phase [43]. Therefore, a PTFE-containing charge should be in a pressed, rather than bulk, form, as a briquette with a diameter of at least 30 mm, and combusted under excess gas pressure in the SHS reactor to prevent PTFE

decomposition products from escaping the reaction zone [39; 42–44].

In the present study, these findings were used to increase the SiC phase content in the target composition of highly dispersed AlN–SiC powders produced via azide SHS technology with PTFE, aiming to bring the experimental composition of the target highly dispersed AlN and SiC powders closer to the theoretical composition in accordance with the stoichiometric equations of azide SHS.

Research methodology

In the study of AlN–SiC composition synthesis processes using azide SHS technology with PTFE, the following initial charge components were used (here and throughout: wt. %):

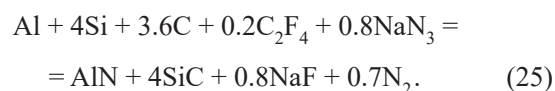
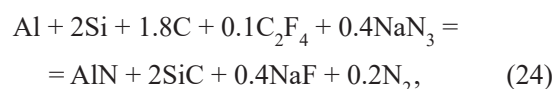
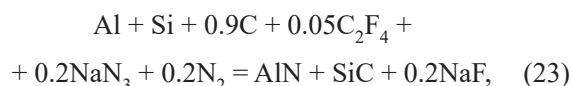
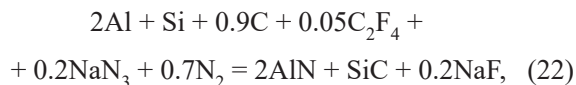
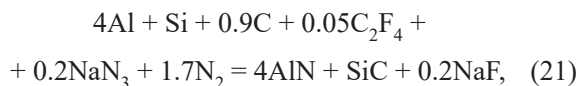
- silicon powder, grade Kr00 (main substance content ≥ 99.9 %, average particle size $d = 40$ μm);
- aluminum powder, grade PA-4 (≥ 98.0 %, $d = 100$ μm);
- sodium azide powder, analytical grade (≥ 98.71 %, $d = 100$ μm);
- polytetrafluoroethylene (PTFE), grade PN-40 (≥ 99.0 %, $d = 40$ μm);
- technical carbon black, grade P701 (≥ 88.0 %, $d = 70$ nm, in the form of agglomerates up to 1 μm).

PTFE was used as an activating and carbidizing additive, partially replacing technical carbon black. Based on the findings from studies [39; 41; 42], a carbidizing mixture of technical carbon and PTFE of various compositions was used to carbide silicon, equivalent to 1 mole of carbidizing carbon:

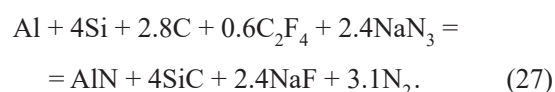
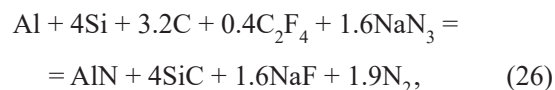


These mixtures correspond to PTFE concentrations of 5 to 15 %, necessary for obtaining pure SiC through reaction (16) and achieving an increased SiC content in composites with silicon nitride [41]. Sodium azide (NaN_3) was added to the charge in an amount sufficient to neutralize the fluorine released during the complete decomposition of PTFE, binding it into the water-soluble compound NaF, which can be easily removed from the azide SHS product by washing with water. As a result, the stoichiometric equations of the azide SHS for AlN–SiC powder compositions with 5 molar ratios of target phases $\text{AlN}:\text{SiC} = 4:1, 2:1, 1:1, 1:2$ and

1:4 using carbidizing mixture (A) with PTFE under combustion in gaseous nitrogen are as follows:



To achieve an AlN–SiC composition with the maximum silicon carbide phase content ($\text{AlN}:\text{SiC} = 1:4$) carbidizing mixtures (B) and (C) with an increased PTFE content were also used:



The initial reagent mixtures (charges) from equations (21)–(27), with an average mass of 22 g, were burned in a 4.5 L azide SHS reactor under a nitrogen gas pressure of 3 MPa, both in bulk form in a tracing paper cup with a diameter of 30 mm and height of 45 mm, and as briquettes pressed at a pressure of 7 MPa with a diameter of 30 mm and an average height of 22 mm. Combustion was initiated with an electric tungsten coil. The maximum gas pressure generated in the reactor during combustion was recorded using a manometer, and the maximum combustion temperature was measured with a tungsten-rhenium thermocouple of 200 μm in diameter, inserted into the charge. The cooled combustion product was removed from the reactor, ground to a loose powder state in a porcelain mortar and washed with water to remove the by-product sodium fluoride (NaF).

The phase composition of the synthesized products was determined using an ARL X'TRA powder X-ray diffractometer (Thermo Fisher Scientific, Switzerland) equipped with a copper anode X-ray tube. Diffraction pattern analysis and quantitative phase composition assessment were performed using the Rietveld method

in the HighScore Plus software with the COD-2024 crystallographic database. The morphology and particle size of the synthesized compositions were examined using a JSM-6390A scanning electron microscope (Jeol, Japan).

Results and discussion

The results of experimental determination of the parameters for azide SHS of AlN–SiC powder compositions according to equations (21)–(25) are presented in Table 2. As the data indicate, combustion of bulk mixtures occurs more intensively than that of pressed mixtures, with higher maximum temperatures and pressure spikes, accompanied by greater dispersion of the SHS reactants and products. The higher the aluminum content in the charge, the greater the combustion parameters (temperature and pressure); however, at maximum aluminum content, the mass loss of the product due to dispersion is offset by mass gain from increased absorption of gaseous nitrogen during aluminum nitride formation. Mass loss is highest in the synthesis of AlN–SiC compositions with roughly equal molar ratios, especially in the case of bulk mixtures. However, this loss decreases as the SiC content in the combustion product increases, accompanied by a reduction in combustion parameters

The X-ray diffraction (XRD) patterns of phase analysis of water-washed solid combustion products from bulk and pressed mixtures are shown in Figs. 2 and 3, respectively.

The data in Fig. 2 show that the XRD patterns of water-washed combustion products from bulk char-

ges used for synthesizing AlN–SiC composites with higher and equal molar content of the AlN phase, i.e., AlN:SiC = 4:1, 2:1 and 1:1, contain strong reflections only for the target phases AlN and SiC, along with weak reflections from free silicon impurities. For the combustion products with increased SiC phase content, i.e., AlN:SiC = 1:2 and 1:4, in addition to the AlN, SiC, and Si reflections, distinct peaks of the undesirable by-product phase Si_3N_4 appear, particularly noticeable in the sample with the maximum SiC content. The XRD patterns of combustion products from pressed charges shows only the target phases AlN and SiC for four ratios AlN:SiC = 4:1, 2:1, 1:1 and 1:2 (with a Si impurity for AlN:SiC = 2:1) and the appearance of an additional undesirable by-product phase, Si_3N_4 , in one case, AlN:SiC = 1:4, with maximum SiC content (Fig. 3).

Table 3 presents the results of quantitative analysis of the XRD patterns, showing the quantitative phase content in the washed combustion products from charges (21)–(25) with carbidizing mixture (A) and minimal PTFE content, as well as for equations (26) and (27) to obtain an AlN–SiC composition with the maximum silicon carbide phase content (AlN:SiC = 1:4) using mixtures (B) and (C) with increased PTFE content. These experimental data are compared with the theoretical results for the target phases AlN and SiC content in the reaction products according to stoichiometric equations (21)–(27).

Table 3 shows that, compared to the azide SHS products without PTFE presented in Table 1, the use of equations (21)–(25) with the first carbidizing mixture (A), containing minimal PTFE ($0.9 + 0.05\text{C}_2\text{F}_4$), led to several notable changes:

Table 2. Combustion parameters of initial powder mixtures for reactions (21)–(25)

Таблица 2. Параметры горения исходных порошковых смесей реакций (21)–(25)

Reaction equation	Initial powder mixture	Maximum combustion temperature, K	Maximum pressure, MPa	Yield of solid products, g	Mass loss, %
Bulk initial powder mixtures					
(21)	$4\text{Al} + \text{Si} + 0.9\text{C} + 0.05\text{C}_2\text{F}_4 + 0.2\text{NaN}_3$	3270	4.38	26.18	0.38
(22)	$2\text{Al} + \text{Si} + 0.9\text{C} + 0.05\text{C}_2\text{F}_4 + 0.2\text{NaN}_3$	3270	4.20	25.07	0.23
(23)	$\text{Al} + \text{Si} + 0.9\text{C} + 0.05\text{C}_2\text{F}_4 + 0.2\text{NaN}_3$	2870	4.14	17.76	26.27
(24)	$\text{Al} + 2\text{Si} + 1.8\text{C} + 0.1\text{C}_2\text{F}_4 + 0.4\text{NaN}_3$	2790	4.19	14.26	38.90
(25)	$\text{Al} + 4\text{Si} + 3.6\text{C} + 0.2\text{C}_2\text{F}_4 + 0.8\text{NaN}_3$	2620	4.09	15.99	26.68
Pressed mixtures					
(21)	$4\text{Al} + \text{Si} + 0.9\text{C} + 0.05\text{C}_2\text{F}_4 + 0.2\text{NaN}_3$	3180	3.89	26.25	0.11
(22)	$2\text{Al} + \text{Si} + 0.9\text{C} + 0.05\text{C}_2\text{F}_4 + 0.2\text{NaN}_3$	3120	3.61	25.10	0.11
(23)	$\text{Al} + \text{Si} + 0.9\text{C} + 0.05\text{C}_2\text{F}_4 + 0.2\text{NaN}_3$	2720	3.51	11.41	26.27
(24)	$\text{Al} + 2\text{Si} + 1.8\text{C} + 0.1\text{C}_2\text{F}_4 + 0.4\text{NaN}_3$	2690	3.81	20.61	11.69
(25)	$\text{Al} + 4\text{Si} + 3.6\text{C} + 0.2\text{C}_2\text{F}_4 + 0.8\text{NaN}_3$	2550	3.71	20.80	4.60

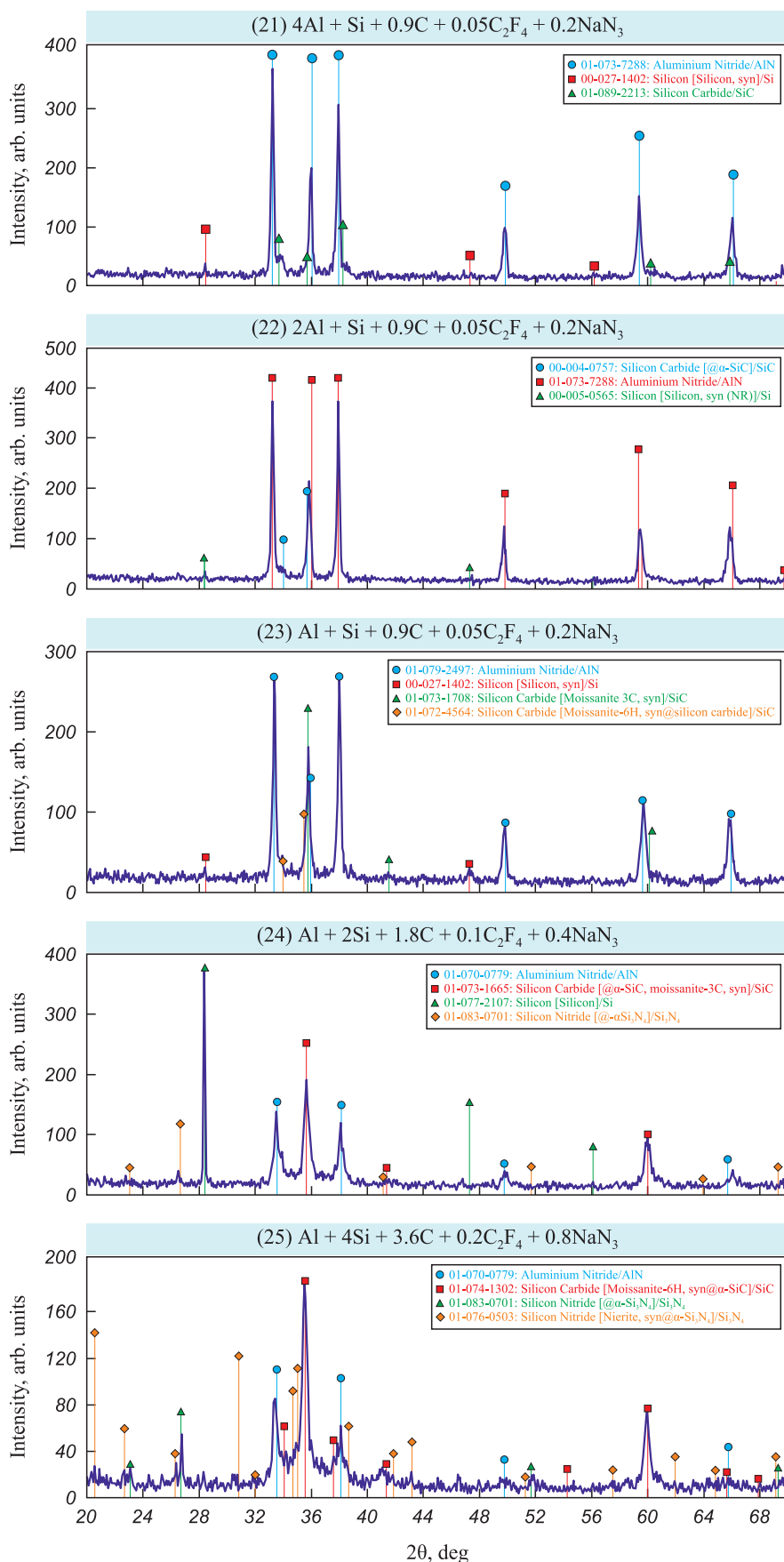


Fig. 2. XRD patterns of combustion products from bulk charges (21)–(25)

Рис. 2. Рентгеновские дифрактограммы продуктов горения насыпных шихт (21)–(25)

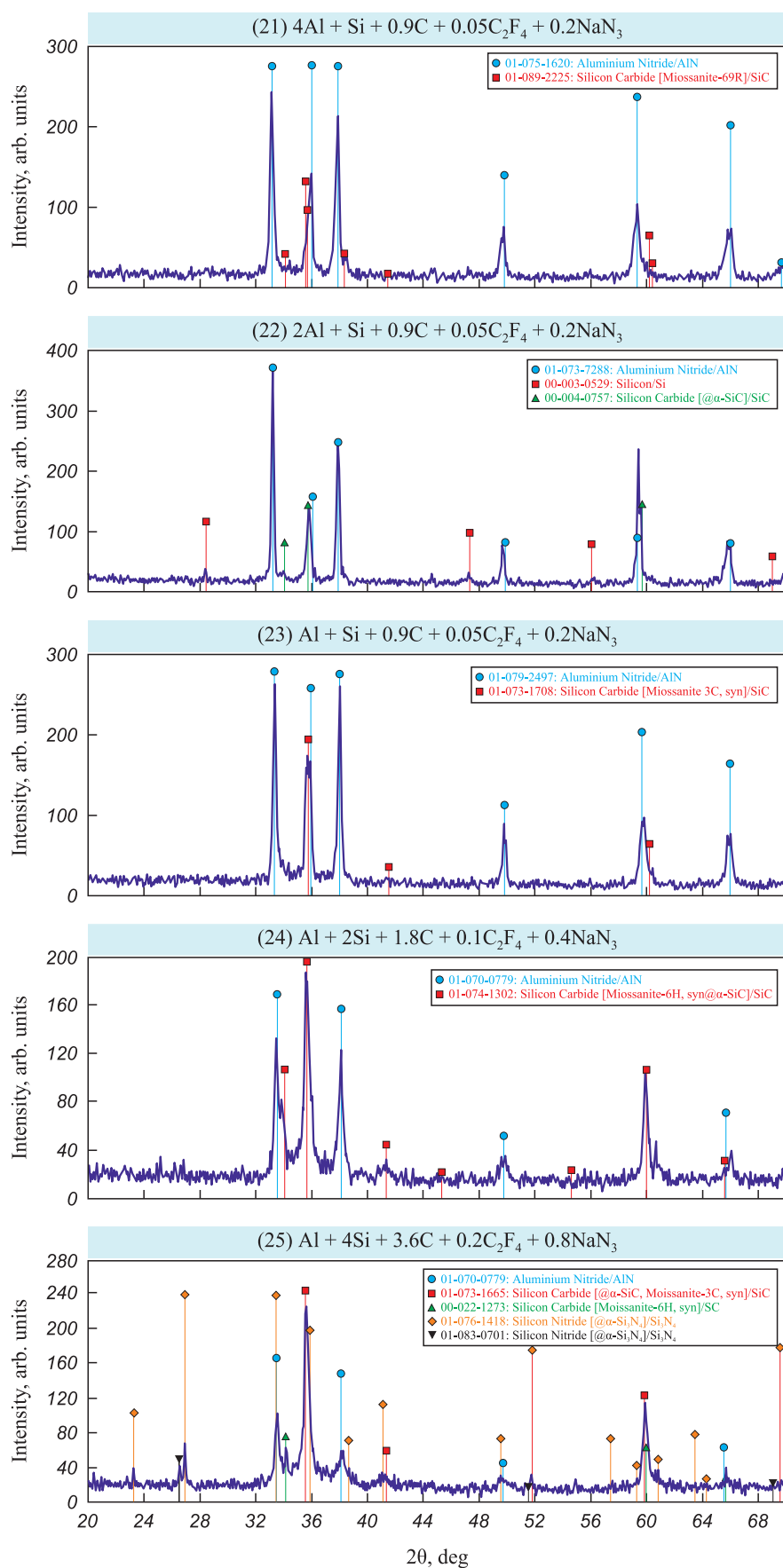


Fig. 3. XRD patterns of combustion products from pressed charges (21)–(25)

Рис. 3. Рентгеновские дифрактограммы продуктов горения прессованных шихт (21)–(25)

– the undesirable, water-insoluble impurity of cryolite salt Na_3AlF_6 completely absent from the combustion products of both bulk and pressed charges, which represents a significant improvement;

– the presence of the undesirable Si_3N_4 by-product phase is eliminated or substantially reduced to 15 % (observed in bulk charges with AlN:SiC ratios of 1:2 and 1:4, and in pressed charges with an AlN:SiC ratio of 1:4);

– impurities of free silicon and carbon are reduced or even absent, particularly in pressed charges, where only 2 % free silicon was detected at an AlN:SiC ratio of 2:1;

– the experimental content of the target phases AlN and SiC for most ratios closely aligns with the theoretical content, especially in pressed charges, with the exception of bulk charges at AlN:SiC ratios of 1:2 and 1:4 and pressed charges at an AlN:SiC ratio of 1:4.

At the same time, increasing the amount of PTFE in carbidizing mixtures (B) and (C), along with a higher proportion of sodium azide NaN_3 for fluorine neutralization in equations (26) and (27), leads to the appearance of the undesirable cryolite salt impurity Na_3AlF_6 (from 4 to 8 %) in the combustion products of both

bulk and pressed charges. It also significantly increases the content of the undesirable Si_3N_4 by-product phase and, consequently, substantially reduces the proportion of the target phases AlN and SiC compared to their theoretical content.

The microstructures of the washed powder combustion products from bulk and pressed charges according to the stoichiometric reaction equations (21)–(25) are shown in Figs. 4 and 5. These figures reveal a trend of decreasing particle size in the combustion products as the SiC content increases in the AlN–SiC powder compositions, corresponding to the trend of decreasing maximum combustion temperature as the SiC content in the product rises (see Table 2). The powders are largest at an AlN:SiC ratio of 4:1, with particles up to 5 μm for bulk charges and up to 2 μm for pressed charges. In other compositions with lower AlN content, the synthesized powders are highly dispersed, consisting of submicron equiaxed particles ranging from 100 nm to 1 μm and nanofibers with diameters of 50–500 nm and lengths up to 5 μm . As the SiC content in the product increases, the proportion of the smallest equiaxed particles, measuring 100–300 nm, also increases. The AlN:SiC ratio of 1:4 composition, with the highest

Table 3. Theoretical and experimental phase composition of water-washed solid products of azide SHS according to equations (21)–(27) for bulk and pressed charges

Таблица 3. Теоретический и экспериментальный фазовые составы промытых твердых продуктов азидного СВС согласно уравнениям (21)–(27) для насыпных и прессованных шихт

Equation reaction	AlN:SiC (mol)	Content, wt. %							
		Theoretical		Experimental					
		AlN	SiC	AlN	SiC	Si ₃ N ₄	Si	C	Na ₃ AlF ₆
Bulk charges									
(21)	4:1	80.4	19.6	81.0	15.0	—	4.0	—	—
(22)	2:1	67.2	32.8	62.0	36.0	—	2.0	—	—
(23)	1:1	50.6	49.4	47.5	51.5	—	1.0	—	—
(24)	1:2	33.8	66.2	20.0	65.0	15.0	—	—	—
(25)	1:4	20.4	79.6	33.7	49.5	15.9	—	0.9	—
(26)	1:4	20.4	79.6	6.1	46.4	36.5	3.0	4.0	4.0
(27)	1:4	20.4	79.6	11.1	42.4	34.4	4.0	—	8.1
Pressed charges									
(21)	4:1	80.4	19.6	80.0	20.0	—	—	—	—
(22)	2:1	67.2	32.8	66.0	32.0	—	2.0	—	—
(23)	1:1	50.6	49.4	51.0	49.0	—	—	—	—
(24)	1:2	33.8	66.2	38.0	62.0	—	—	—	—
(25)	1:4	20.4	79.6	18.0	67.0	15.0	—	—	—
(26)	1:4	20.4	79.6	19.0	46.4	27.6	3.0	4.0	7.0
(27)	1:4	20.4	79.6	17.0	42.4	31.6	4.0	—	5.0

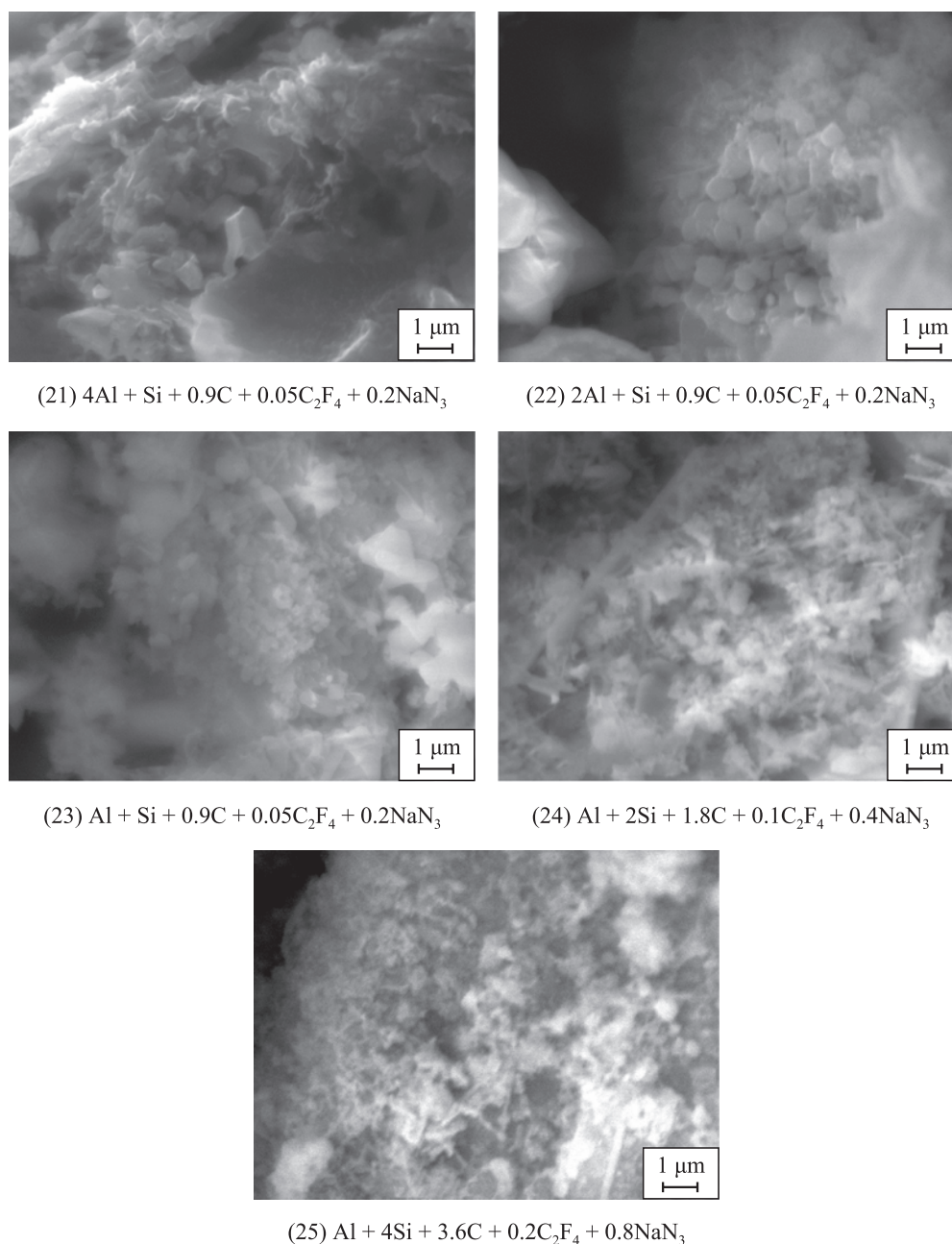


Fig. 4. SEM images of combustion products from bulk charges (21)–(25)

Рис. 4. Микроструктура продуктов горения насыпных шихт (21)–(25)

silicon carbide content, forms agglomerates of these small particles.

Nanofibers are practically absent in compositions with a high AlN content in the combustion products of both bulk and pressed charges, as well as in compositions with a high SiC content in the combustion products of pressed charges. The highest presence of nanofibers is observed in compositions with an equal phase content of AlN and SiC in the combustion products of both types of charges, as well as in compositions with a high SiC content in the combustion products of bulk charges.

Conclusion

The results of this study indicate that incorporating polytetrafluoroethylene (PTFE) in azide SHS technology has a highly positive impact on the production of AlN–SiC ceramic powders. While the traditional approach of azide SHS using sodium azide (NaN_3) as a nitriding agent and activating halide salt additives $(\text{NH}_4)_2\text{SiF}_6$, AlF_3 , and NH_4F enabled the production of highly dispersed AlN–SiC powder compositions with particle sizes below $1\text{ }\mu\text{m}$ from a mixture of elemental aluminum, silicon, and carbon pow-

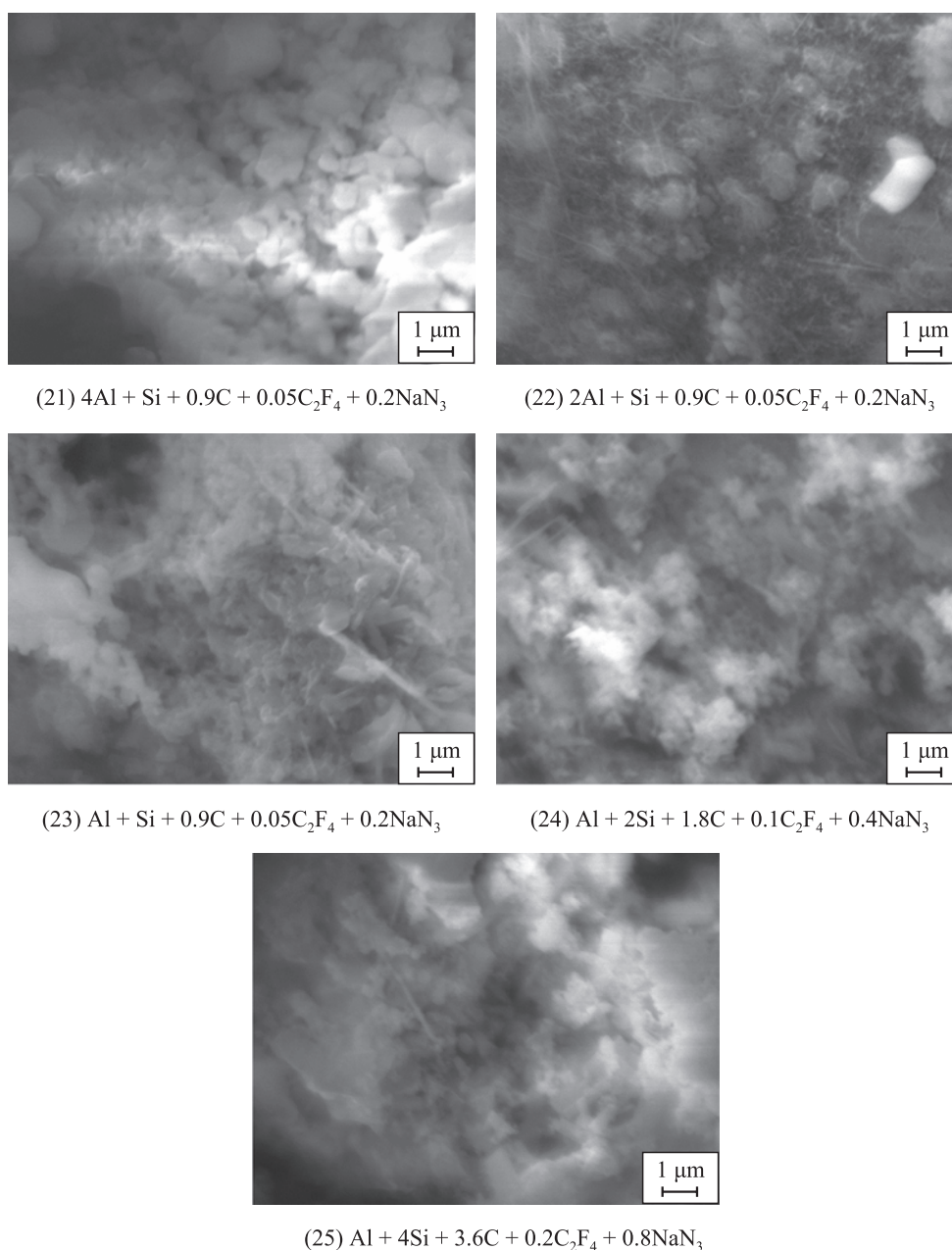


Fig. 5. SEM images of combustion products from pressed charges (21)–(25)

Рис. 5. Микроструктура продуктов горения прессованных шихт (21)–(25)

ders – a clear advantage of this technology – the phase composition of these compositions had significant drawbacks. Ideally, the phase composition should match the various targeted theoretical ratios of aluminum nitride and silicon carbide phases in accordance with the initial stoichiometric equations. However, the experimentally obtained target phases AlN and SiC were substantially below the theoretical values, particularly for the SiC phase, whose quantity was, on average, half of the theoretical amount. Additionally, significant amounts of undesirable by-product phases, including silicon nitride and the water-insoluble cryolite salt Na_3AlF_6 , were present.

Introducing PTFE as an activating and carbidizing additive, partially replacing carbon in the carbidizing mixture $0.9\text{C} + 0.05\text{C}_2\text{F}_4$ during azide SHS, resolved most of the traditional approach's limitations across various AlN and SiC target ratios.

This adjustment maintained the high dispersity of the synthesized AlN–SiC powders while bringing their phase composition – especially in pressed charges – much closer to the theoretical target. The SiC phase content also increased significantly, and the unwanted by-products of silicon nitride and the water-insoluble cryolite salt Na_3AlF_6 were elimi-

nated. However, for AlN–SiC compositions with the highest relative SiC content, it was not possible to completely avoid the formation of the silicon nitride by-product phase.

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A. P. Amosov – defined the purpose of the work, summarized the obtained results, wrote and edited the manuscript.
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I. A. Uvarova – conducted experiments, prepared and organized the experimental results, contributed to the analysis and discussion, and helped write the article.
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