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Effect of Si, Al, Cu, Cr, and TiSi₂ on the formation of the Ti₃SiC₂ MAX phase during self-propagating high-temperature synthesis in air

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Abstract. This study examines how additions of Si, Al, Cu, and Cr powders to the stoichiometric 3Ti–Si–2C (at. %) charge influence the formation of the Ti₃SiC₂ MAX phase during self-propagating high-temperature synthesis (SHS) performed in air within a sand bed, without a sealed reactor or controlled atmosphere. The effect of partially or fully substituting elemental Ti and Si powders with TiSi₂ on the Ti₃SiC₂ yield is also assessed. Microstructural characterization of the SHS products was conducted using scanning electron microscopy equipped with energy-dispersive spectroscopy, and the phase composition was quantified by X-ray diffraction. An addition of 0.1 mol Si to the stoichiometric mixture increases the Ti₃SiC₂ content in the product to approximately 70 vol. %. Incorporating 0.1 mol Al decreases the Ti₃SiC₂ fraction to 39 vol. % and results in the formation of TiAl. In contrast, combining a silicon excess with 0.1 mol Al in the 3Ti–1.25Si–2C + 0.1Al system markedly enhances the Ti₃SiC₂ yield, reaching ~89 vol. %. For synthesis in the TiSi₂–C system, the share of the MAX phase decreases while secondary phases become more prevalent; the maximum Ti₃SiC₂ content in this system is 56 vol. %. When TiSi₂ fully replaces elemental silicon in the 2.5Ti–0.5TiSi₂–2C mixture, the Ti₃SiC₂ fraction drops to 20 vol. %.

Keywords: self-propagating high-temperature synthesis (SHS), reactorless SHS, Ti₃SiC₂ MAX phase, powders, microstructure, X-ray diffraction

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Влияние Si, Al, Cu, Cr и TiSi₂ на получение МАХ-фазы Ti₃SiC₂ методом самораспространяющегося высокотемпературного синтеза на воздухе

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Аннотация. В работе рассмотрено влияние добавок порошков Si, Al, Cu и Cr к стехиометрическому составу шихты 3Ti–Si–2C (ат. %) при получении МАХ-фазы Ti₃SiC₂ в режиме самораспространяющегося высокотемпературного синтеза (СВС) на воздухе в засыпке из песка без применения закрытого реактора и специальной атмосферы или вакуума. Показано влияние частичной или полной замены элементных порошков шихты Ti и Si на TiSi₂ на выход Ti₃SiC₂. Проведен анализ микроструктуры полученных СВС-продуктов с помощью растрового электронного микроскопа с приставкой для энергодисперсионной спектроскопии. Исследованы качественный и количественный фазовые составы порошковых СВС-продуктов с помощью рентгеновского дифрактометра. Установлено, что добавка 0,1 моль порошка кремния к стехиометрическому составу шихты увеличивает количество МАХ-фазы Ti₃SiC₂ в продукте до 70 % от объема. Введение 0,1 моль Al-порошка в исходную шихту приводит к уменьшению количества Ti₃SiC₂ до 39 % от объема и появлению новой фазы TiAl. При этом совместный избыток кремния вместе с добавкой 0,1 моль Al в системе 3Ti–1,25Si–2C + 0,1Al существенно повышают содержание Ti₃SiC₂ до ~89 об. %. Установлено, что при синтезе в системе TiSi₂–C в продуктах реакции доля МАХ-фазы уменьшается, а количество побочных фаз возрастает. Максимальное содержание Ti₃SiC₂ в продукте при синтезе в системе TiSi₂–C достигает 56 % от общего объема. При увеличении количества TiSi₂ до полной замены элементного кремния в исходной шихте 2,5Ti–0,5TiSi₂–2C доля МАХ-фазы Ti₃SiC₂ в продукте падает до 20 %.

Ключевые слова: самораспространяющийся высокотемпературный синтез (СВС), безреакторный синтез, МАХ-фаза Ti₃SiC₂, порошки, структура, рентгенофазовый анализ

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Introduction

The Ti₃SiC₂ MAX phase is a promising layered ternary carbide that combines key ceramic and metallic properties, including excellent oxidation resistance, high thermal and electrical conductivity, thermal-shock resistance, high-temperature plasticity, creep resistance, low density, and good machinability [1; 2]. These properties make Ti₃SiC₂ a potential alternative to conventional ceramics.

Many published synthesis approaches require expensive equipment, long high-temperature dwell times, and protective atmospheres, all of which increase the cost and complexity of MAX-phase production [3–6]. In contrast, the highly exothermic and economically efficient process of self-propagating high-temperature synthesis (SHS) significantly simplifies the fabrication route, requires no special hardware, and proceeds much faster than furnace sintering [7; 8]. A simple method

for producing MAX-phase cermets was recently proposed, based on infiltrating molten metals into a porous Ti₃SiC₂ skeleton synthesized via SHS in air [9]. During Ti₃SiC₂ formation, the reaction temperature may reach 2260 °C [10], while the maximum adiabatic combustion temperature is reported to be 2735 °C [11].

Formation of Ti₃SiC₂ proceeds through several stages. Initially, TiC particles and a Ti–Si melt form simultaneously. At the next stage, TiC dissolves into the Ti–Si liquid, followed by crystallization of Ti₃SiC₂ platelets [12–14]. Because SHS evolves extremely rapidly – values below 3–4 s are typical for the first stage – post-ignition control of the process is practically impossible. Therefore, establishing optimal synthesis parameters is essential to maximize the purity of the final Ti₃SiC₂ product. Deviations from charge stoichiometry and insufficient high-temperature dwell associated with fast post-reaction cooling commonly

lead to increased fractions of secondary phases such as TiC and TiSi₂.

The reactions leading to TiC and the Ti–Si melt compete for the available titanium, as both intermediates form simultaneously within a single reactive system. Consequently, a deficiency of one intermediate and the excess of the other inevitably decreases the Ti₃SiC₂ yield in the SHS product.

TiC is frequently reported as the primary secondary phase in Ti₃SiC₂ synthesis, indicating an insufficient amount of Ti–Si melt available for MAX-phase platelet growth. Therefore, many studies [15–22] use chemical precursors instead of elemental Ti and Si powders. One such precursor is titanium disilicide (TiSi₂), which has the lowest crystallization temperature among Ti–Si compounds (1330 °C).

The Ti–Si melt crystallizes within the 1480–1570 °C range, where Ti₃SiC₂ formation becomes significantly slower. Additions of aluminum are known to reduce the crystallization temperature of the Ti–Si melt, thereby increasing the time during which TiC can react with the liquid and form the MAX phase during cooling. For example, the SHS system 3Ti–Si–2C–0.1Al (at. %) synthesized under argon after vacuum drying yielded a product containing 89 wt. %¹ Ti₃SiC₂ [23]. The authors noted that the addition of aluminum to the stoichiometric 3Ti + Si + 2C mixture suppresses the reaction responsible for TiC formation, which in turn increases the Ti₃SiC₂ yield. In the SHS system 3Ti + 1.2Si + 2C + 0.1Al, the Ti₃SiC₂ phase yield reached approximately 83 %, accompanied by 13 % TiC and 4 % Ti₅Si₃ in the product [24]. The beneficial role of a slight silicon excess has also been repeatedly emphasized [25–27], and may likewise be attributed to the crystallization behavior of the Ti–Si melt [28–30].

According to the Ti–Si phase diagram, at 50 at. % Si the crystallization temperature is 1570 °C; a slight Si excess reduces it to about 1480 °C, and at >67 at. % Si it decreases further to approximately 1330 °C.

Copper additions of 5–10 % to Ti and Si also reduce the melting temperatures of the corresponding binary systems [31; 32] and may therefore lower the crystallization temperature of the Ti–Si–Cu melt, potentially promoting an increased Ti₃SiC₂ yield. However, Ti₃SiC₂ is known to decompose in contact with molten Cu via Si deintercalation, forming Cu(Si) and TiC_x [33; 34].

Additions of 10 at. % Cr reduce the melting temperatures of Ti–Cr and Si–Cr alloys from 1670 to 1550 °C and from 1414 to 1305 °C, respectively [35; 36].

No data have been reported on the interaction of molten Cr with Ti₃SiC₂, which is attributed to the high melting temperature of chromium (1856 °C), while Ti₃SiC₂ begins to decompose at about 1450 °C [1].

Controlling the mechanism of Ti₃SiC₂ formation under SHS conditions offers the possibility of developing energy-efficient and technologically simple synthesis routes for MAX phases. Typically, SHS of Ti₃SiC₂ is carried out in sealed reactors under protective atmospheres or in vacuum, which significantly increases production costs and limits scalability. Therefore, the present study aims to identify a simpler and more accessible method for synthesizing Ti₃SiC₂ with minimal secondary phases. This work focuses on evaluating the effects of Si, Al, Cu, Cr, and TiSi₂ additions on Ti₃SiC₂ formation during reactor-free SHS in air under a sand bed.

Materials and methods

Powders used as starting reagents for synthesis included porous titanium powder TPP-7 with a coarse particle size ($d \sim 300 \mu\text{m}$, purity 98 %), technical carbon T900 ($d \sim 0.15 \mu\text{m}$, agglomerates up to $10 \mu\text{m}$, purity 99.8 %), colloidal graphite C-2 ($d \sim 15 \mu\text{m}$, purity 98.5 %), silicon Kr0 ($d \sim 1\text{--}15 \mu\text{m}$, purity 98.8 %), aluminum PA-4 ($d \sim 100 \mu\text{m}$, purity 98 %), copper PMS-1 ($d \sim 100 \mu\text{m}$, purity 99.5 %), chromium Kh99N1 ($d \sim 100 \mu\text{m}$, purity 99.0 %), and titanium disilicide TiSi₂ ($d \sim 100 \mu\text{m}$, purity 99.0 %).

The starting powders were weighed on a laboratory balance with an accuracy of 0.01 g and mixed in a ceramic mortar for 5 min to obtain homogeneous mixtures corresponding to the following systems: 3Ti–Si–2C + 0.1Al, 3Ti–Si–2C + 0.1Cu, and 3Ti–Si–2C + 0.1Cr, as well as TiSi₂–C, where elemental Si and Ti were replaced by TiSi₂ in amounts of 15, 50, and 100 % (complete substitution), calculated for the formation of Ti₃SiC₂ MAX phase.

Cylindrical compacts with a diameter of 23 mm and a mass of 20 g were produced from the prepared powder mixtures by single-action pressing at 22.5 MPa. SHS (combustion) was initiated using an electrically heated Ni–Cr coil. The samples were synthesized by combustion in air under a sand layer, which reduces oxidation of the combustion products [25]. The general scheme of the experiment is shown in Fig. 1. As seen, the pressed powder mixture is completely isolated from the ambient air by the sand bed to limit oxidation of the reaction products. After the SHS reaction, secondary Ti₃SiC₂ formation processes proceed in the cooling sample, also under the sand layer.

Microstructural examination and chemical analysis were performed using a Tescan Vega3 (Czech Republic)

¹ Unless otherwise noted, all compositions are in mass percent (wt. %).

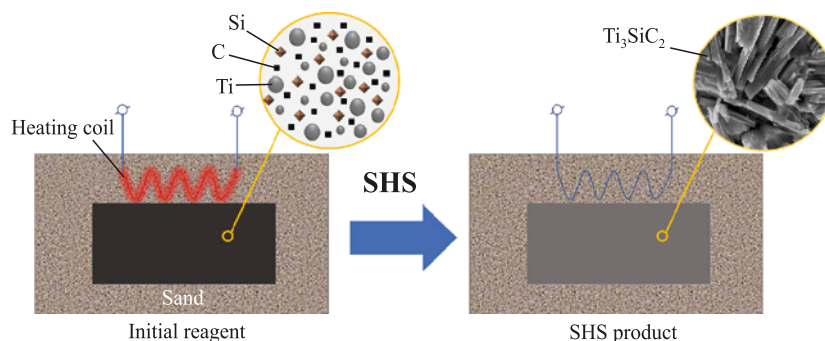


Fig. 1. Scheme for SHS of Ti₃SiC₂ under a sand layer

Рис. 1. Принципиальная схема синтеза Ti₃SiC₂ под слоем песка

scanning electron microscope equipped with an X-ray energy-dispersive spectroscopy attachment. The phase composition was determined by X-ray diffraction using an ARL X'tra-138 diffractometer (Switzerland) with CuK_α radiation, operated in continuous-scan mode over $2\theta = 5\text{--}80^\circ$ at a scan rate of $2^\circ/\text{min}$. Quantitative phase analysis was carried out using the reference intensity ratio (RIR) method.

Results and discussion

The SHS reaction produces a porous skeleton, which has been repeatedly described in previous studies [9; 12; 25]. After mechanical pulverization, the SHS skeleton is converted into a fine powder, and its particle-size distribution is adjusted using sieves of the required mesh size.

To evaluate the possibility of increasing the Ti₃SiC₂ MAX-phase yield during SHS in air, a series of experiments was performed in which 10 % of Si, Cr, Al, or Cu was added to the 3Ti–1Si–2C charge composition.

In the sample obtained with an excess of silicon (Fig. 2, *a*), the unreacted titanium carbide is non-stoichiometric and corresponds approximately to TiC_{0.6}. Elemental analysis of the lighter Ti–Si regions showed an atomic ratio Si:Ti = 60:40, which is consistent with TiSi₂. The layered morphology and elemental ratios also confirm the presence of Ti₃SiC₂ regions. A similar microstructural pattern is observed when 10 % Cr is added (Fig. 2, *b*); however, in this case only trace amounts of Ti₃SiC₂ are present, while chromium is concentrated within the TiSi₂ phase. In the Cu-containing sample (Fig. 2, *d*), the light-grey regions contain predominantly copper and silicon in a ratio of about 50:15, along with up to 10 at. % carbon. No Ti₃SiC₂ MAX phase is detected, and the non-stoichiometric titanium carbide corresponds to TiC_{0.5}. Despite the presence of significant amounts of TiSi₂ in direct contact with TiC_{0.5}, Ti₃SiC₂ does not form under these conditions.

These findings suggest that additions of 10 % Cu or Cr inhibit the formation of Ti₃SiC₂; however, the mechanism governing their influence on MAX-phase formation under SHS conditions requires further study. This may be due to the fact that Cu and Cr cannot occupy the A-site in MAX phases, and their presence in the Ti–Si melt hinders Ti₃SiC₂ structural formation.

In the sample containing aluminum (Fig. 2, *c*), thin dark-grey regions surrounding the Ti₃SiC₂ platelets were observed. EDS results indicate that these regions correspond to a mixture of TiAl₂ and TiSi₂. The crystallization temperature of TiAl₂ (1175 °C) is significantly lower than that of the Ti–Si melt (1330–1480 °C, depending on the Ti/Si ratio). Considering that Ti₃SiC₂ forms via the interaction of solid TiC with liquid Ti–Si, it can be inferred that Al reduces the crystallization temperature of the Ti–Si melt, extending the time during which the melt remains liquid under SHS conditions. This prolongs its interaction with TiC and allows Ti₃SiC₂ structure formation to continue for a longer duration, ultimately increasing the MAX-phase content in the SHS product.

These observations are consistent with previous findings reported in [25], where the addition of Al increased the Ti₃SiC₂ yield under reactor-based SHS conditions. The authors showed that a combined excess of 20 % Si and 10 % Al in the Ti:Si:C:Al = 3:1.2:2:0.1 system increases the Ti₃SiC₂ content from 64 to 83 %.

It is reasonable to assume that further optimization of the 3Ti–1xSi–2C–yAl system, with respect to both the silicon excess (*x*) and the aluminum addition (*y*), may produce even higher Ti₃SiC₂ yields during SHS in air. To verify this, several charge compositions containing different silicon excesses and Al additions were investigated. The results are presented in Table 1. A silicon excess of 15–25 % combined with 10 % Al substantially increases the Ti₃SiC₂ content, reaching a maximum of ~89 vol. % in the 3Ti–1.25Si–2C + 0.1Al system. Varying the Al amount in the absence of excess

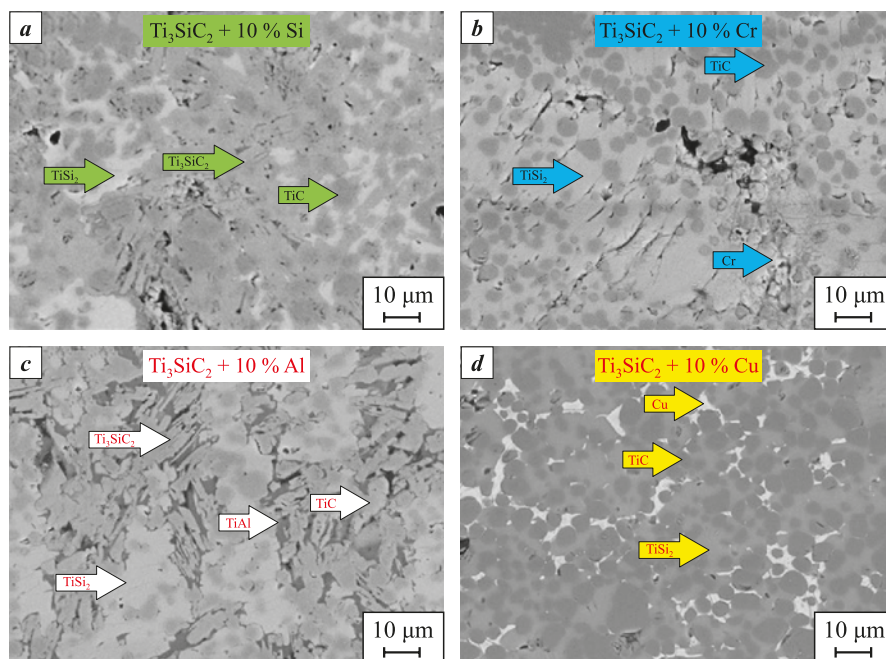


Fig. 2. Microstructures of the samples after introducing 10 % (0.1 mol) of Si (*a*), Cr (*b*), Al (*c*) and Cu (*d*) into the 3Ti–1Si–2C system

Рис. 2. Микроструктуры образцов после введения в систему 3Ti–1Si–2C по 10 % (0,1 моль) Si (*a*), Cr (*b*), Al (*c*) и Cu (*d*)

Si does not significantly affect the Ti₃SiC₂ yield, which remains within 38–47 vol. %. Microstructural images of the sample containing the maximum Ti₃SiC₂ content are shown in Fig. 3. The sample consists predominantly of characteristic, randomly oriented Ti₃SiC₂ platelets, while the pore surfaces are coated with a thin (10–15 μm) layer of densely packed equiaxed TiC particles. The width of most Ti₃SiC₂ platelets ranges from 2 to 5 μm, and their length varies between 10 and 50 μm.

Diffraction patterns of the SHS products with the highest and lowest Ti₃SiC₂ contents are shown in Fig. 4. Based on these data (Fig. 4, *a*), the intensity ratio of the main Ti₃SiC₂ peaks (39.5 and 42.4°) to the TiC peaks (36.0 and 41.8°) corresponds to approximately 80 vol. % Ti₃SiC₂. In Fig. 4, *b*, the TiC peaks are sig-

nificantly more intense than those of Ti₃SiC₂, which is consistent with ~30 % Ti₃SiC₂ in the product. In addition, distinct graphite peaks (at ~26.5°) are observed in all samples listed in Table 1, as illustrated in Fig. 4, *b*. This indicates that part of the carbon does not completely react during SHS. Notably, the initially amorphous carbon black used as a reagent becomes crystallized into graphite during combustion, as described previously for Ti–C SHS systems [37; 38]. A slight shift of certain Ti₃SiC₂ peaks is also observed, which may indicate partial incorporation of Al into the Ti₃SiC₂ lattice during SHS, a phenomenon previously noted in the literature [23].

Porous SHS skeletons were also synthesized using TiSi₂ as a chemical reagent to replace elemental Ti and Si in quantities of 10, 50, and 100 %. Analysis of the XRD patterns revealed that such substitution results in an increased amount of secondary phases compared to the Ti₃SiC₂ MAX phase (Fig. 5). A new secondary phase, SiC, also appears. The maximum Ti₃SiC₂ content in this series – 56 % – was obtained when silicon was replaced by 10 % TiSi₂. However, increasing the TiSi₂ fraction to 100 % reduced the Ti₃SiC₂ content to 20 %. For comparison, SHS conducted using elemental Ti, Si, and C powders in the stoichiometric 3Ti–1Si–2C charge composition yields up to 66 % Ti₃SiC₂ [25]. This reduction may be attributed to insufficient temperature in the TiSi₂-containing SHS systems, which shortens the lifetime of the Ti–Si melt and prevents the Ti₃SiC₂ structure-formation process from fully completing.

Phase composition of SHS products in the 3Ti–xSi–2C + yAl system

Содержание фаз в CBC-продукте системы 3Ti–xSi–2C + yAl

Charge composition	Ti ₃ SiC ₂ , vol. %	TiC, vol. %
3Ti–1.00Si–2C	36	64
3Ti–1.15Si–2C + 0.1Al	88	12
3Ti–1.20Si–2C + 0.1Al	78	22
3Ti–1.25Si–2C + 0.1Al	89	11
3Ti–1.00Si–2C + 0.05Al	38	62
3Ti–1.00Si–2C + 0.08Al	61	39
3Ti–1.00Si–2C + 0.12Al	47	53

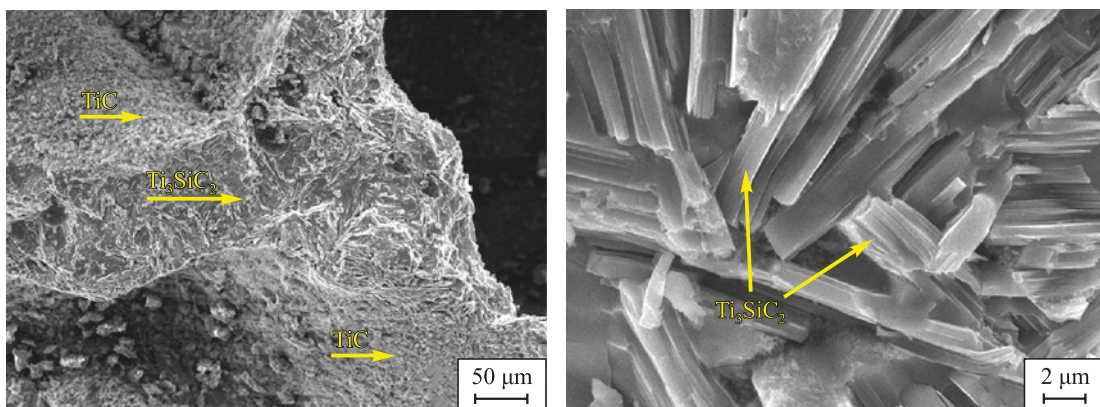


Fig. 3. SEM images of the microstructure of the sample synthesized from the 3Ti–1.25Si–2C + 0.1Al system

Рис. 3. СЭМ-изображения микроструктуры образца, синтезированного из системы 3Ti–1,25Si–2C + 0,1Al

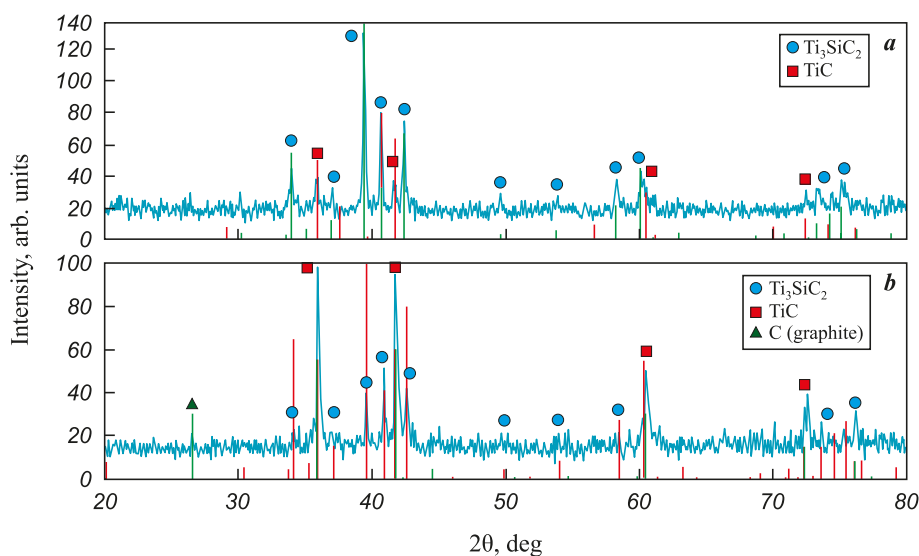


Fig. 4. XRD patterns of the 3Ti–1.25Si–2C + 0.1Al (a) and 3Ti–1.00Si–2C + 0.05Al (b) systems

Рис. 4. Дифрактограммы систем 3Ti–1,25Si–2C + 0,1Al (a) и 3Ti–1,00Si–2C + 0,05Al (b)

Conclusions

1. It was established that the combined addition of excess Si and Al in the 3Ti–1.25Si–2C + 0.1Al charge composition substantially increases the Ti_3SiC_2 content, reaching ~89 vol. % under SHS conditions in air using a sand layer without a reactor or protective atmosphere.

2. Additions of Cu and Cr in the 3Ti–1Si–2C + 0.1Cu and 3Ti–1Si–2C + 0.1Cr systems lead to an almost complete suppression of Ti_3SiC_2 formation in the SHS product.

3. Partial (10 and 50 %) and complete (100 %) substitution of the elemental Ti and Si powders with TiSi_2 significantly reduces the yield of the Ti_3SiC_2 MAX phase during SHS in air.

4. It is likely that further optimization of the 3Ti–1.25Si–2C + 0.1Al system with respect to the par-

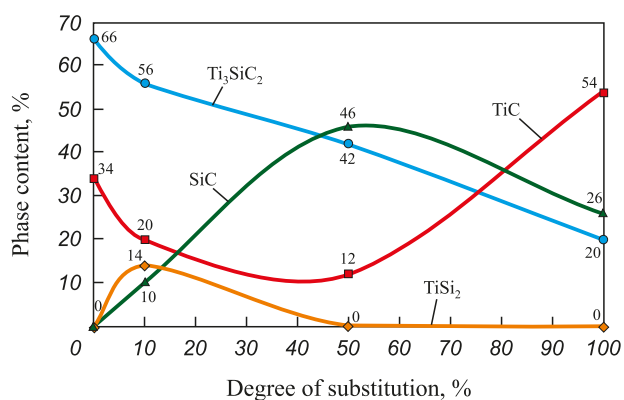


Fig. 5. Dependence of the phase composition of the SHS product on the degree of Si and Ti substitution by TiSi_2 in the 3Ti–1Si–2C green mixture

Рис. 5. Зависимость содержания фаз в продукте СВС от степени замещения Si и Ti на TiSi_2 в исходной шихтовой смеси 3Ti–1Si–2C

ticle-size distribution of the starting powder reagents, as well as scale-up factors, may allow the Ti₃SiC₂ content to exceed 90 % under reactorless SHS conditions.

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