



## Obtaining and Research of the Composition and Structure of the Max- Phase of the Ti-Al-C System by Shs Method

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### Abstract

The results of the process of obtaining and studying the composition and structure of  $Ti_3AlC_2$  MAX-phases from the  $TiCo_{0.5-x}Al$  ( $x = 20, 25, 30$ ) system by the SHS method are discussed. The quantitative analysis of the obtained MAX-phases is carried out. It is shown that  $Ti_3AlC_2$ , with total MAX-phase content of 95%, and TiC content not exceeding 5,1% are obtained from the SHS - Ti-Al-C system.

**Keywords:** Multicomponent, Shs-System, Max-Phase, Structure, Composition, Titanium, Aluminum, Carbon.

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**Citation:** V.A. Martirosyan et al. (2019), Obtaining and Research of the Composition and Structure of the Max- Phase of the Ti-Al-C System by Shs Method. Int J Pharm Sci & Scient Res. 5:1, 1-5.

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**Received:** December 24, 2018

**Accepted:** December 27, 2018

**Published:** January 30, 2019

### Introduction

The development of new light, durable and heat-resistant materials, meeting the modern requirements of aircraft engine designers to the most loaded parts of aviation equipment (primarily to the blades and disks of the compressor and turbine flow parts) is the most important task of modern aviation materials science.

For these purposes, a new class of materials based on the MAX-phases is currently proposed, to whose production and properties more than a hundred publications in domestic and foreign periodicals are devoted [1,2]. MAX-phases are chemical compounds with the general formula  $M_{n+1}A X_n$ , where M is a transition metal, A – an element of the subgroup “A” of the periodic table, X - carbon or nitrogen. Currently, more than 60 triple carbides and nitrides belonging to such phases [3-4] are obtained. The growing interest in the MAX-phases is associated with an unusual and, sometimes, unique set of mechanical properties, which is due to the nature of their special crystal structure, as well as the mobility of dislocations of the basic sliding systems even at room temperature. These compounds combine the advantages of both ceramic and metal materials. Like metals, MAX-phases have high electrical and thermal conductivity, while showing high resistance to thermal shock. On the other hand, they have a small specific gravity, high

modulus of elasticity, low thermal coefficient of expansion, high heat resistance and excellent heat resistance. The layering at the level of the crystal structure leads to a pronounced laminate structure of the MAX phase grain with a layer thickness of up to tens of nanometers. This structure makes it possible to deform the material without macroscopic damages and destructions. When loading, the nanolaminate structure of the material undergoes shear deformation between the layers, and the latter's bending and stratification are observed.

A typical and most studied representative of the MAX-phases is  $Ti_3AlC_2$  [5,7]. However, the various current methods of obtaining materials based on the MAX-phases are far from being perfect. They are characterized by significant energy costs, complexity and multistage technological cycles, low productivity, and do not always provide the required quality of materials in structure and properties, as well as purity. All this requires the creation and development of new methods and technologies for their production.

The methods of self-propagating high-temperature synthesis (SHS) and thermal explosion have great potential in this regard [8]. These methods, based on the use of internal chemical energy of the initial reagents, are an example of a very profitable organization of the synthesis process from a thermal point of view.

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SHS methods have certain advantages over classical methods of obtaining the above-mentioned materials, but, unfortunately, there are also inherent shortcomings. One of the disadvantages of self-propagating high-temperature synthesis is a large specific porosity of the materials obtained, connected with the presence of impurity gas in the synthesis process. But these MAX-phases are used in powder metallurgy in the form of powders, so here the porosity does not interfere [9].

The aim of the work was to establish the possibility of synthesis of  $Ti_3AlC_2$  compound from Ti-Al-C powder mixture by SHS method in combustion mode and to study the effect of the amount of carbon on the phase formation process and parameters of the crystal structure of the obtained MAX-phase.

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## Materials and methods

The following powders were used as initial materials: titanium of PTS grade (purity 98 %, average particle size 100  $\mu\text{m}$ ), carbon black of P-701 grade (99,5 %, 2  $\mu\text{m}$ ), aluminum of PA-4 grade (99 %, 50  $\mu\text{m}$ ). To remove moisture, the powders were dried in air at a temperature of 120°C for 2 hours. Mixing of powders was performed manually in a porcelain mortar.

Experiments to obtain the MAX-phase were carried out in the reactor (Fig. 1), which is a metal container consisting of two parts. The lower part is filled with quartz sand, the upper part is a conical cover, open at the top. In the middle of the lower part of the reactor, in the quartz

sand, recesses were made, where a sample of the charge consisting of the initial mixtures of the stoichiometric composition was placed. The pre-mixed charge was placed in a pit of quartz sand and closed with a conical cover. In the center of the sample the initiator (Ti+C) was filled. Combustion was carried out by means of a tungsten spiral heated by electric current from the upper end of the sample. Under these conditions, a chemical reaction is excited in the surface layers of the mixture and a combustion wave is formed, propagating at a constant speed along the entire length of the sample, i.e., there occurs a SHS. The combustion proceeds during 10...15 sec. within the temperature range of 2300...2500°C. When cooling the combustion products, a porous mass is formed as a result of whose grinding the powder is obtained.



Fig 1: Laboratory SHS installation

Microscopic measurements were carried out using a scanning electronic microscope of the type (SEM) VEGA TS 5130MM, Tescan, Czech Republic, Microanalysis Sistem INCA Energy 300, the x-ray phase study - using a radiograph of the brand DRONE-3.0 using  $\text{CuK}\alpha$  - radiation and a Nickel filter in the following mode: voltage - 25 kV, current - 10 mA, recording speed-420  $\text{mm}\times\text{h}^{-1}$  [10].

## Discussion of the results

The sequence of SHS of the combustion of the  $3\text{Ti}+2\text{Al}+2\text{C}$  mixture is the following: prior to initiating the combustion, there are source components of a mixture Ti, Al, and C. At the time of passage of the combustion front through the range, Ti and Al start to melt and gradually the interaction of carbon begins. During that period, the titanium carbide, being in the crystalline state, is surrounded by a fusion Ti-Al. After the passage of the combustion wave into the depth, the formation of the MAX-phases,  $\text{Ti}_3\text{AlC}_2$  and  $\text{Ti}_2\text{AlC}$  starts. It can be assumed that the crystals of titanium carbide formed in the combustion wave

dissolve in the fusion of Ti-Al. The fusion is saturated with carbon, and, while cooling, the crystallization of MAX-phases of ternary compounds takes place.

Thus, when burning a mixture of  $3\text{Ti}+2\text{Al}+2\text{C}$ , the formation of the material occurs in stages. At the first stage, the dominant reaction is the synthesis of titanium carbide, providing the main heat release and propagation of the combustion front. As a result, TiC crystals are formed, surrounded by the fusion TiAl. After the passage of the combustion front, the dissolution of titanium carbide in the surrounding fusion occurs with subsequent crystallization of the MAX - phase.

The X-ray phase analysis of the synthesis products (Fig. 2), showed that the resulting product is multiphase. Also, intermetallic phases  $\text{Al}_3\text{Ti}$  and  $\text{Al}_5\text{Ti}_2$  were found in the Ti-Al-C system, when the amount of aluminum increases, the radiographs  $\text{TiCo},5 - 25\% \text{Al}$ ,  $\text{TiCo},5 - 30\% \text{Al}$ , almost do not differ from each other, only the ratio of the obtained products changes quantitatively (Table 1).

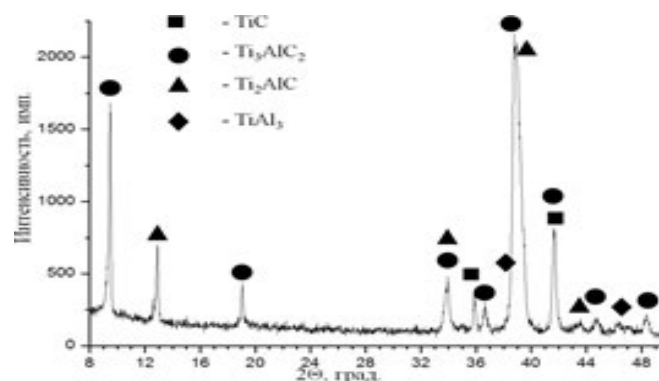


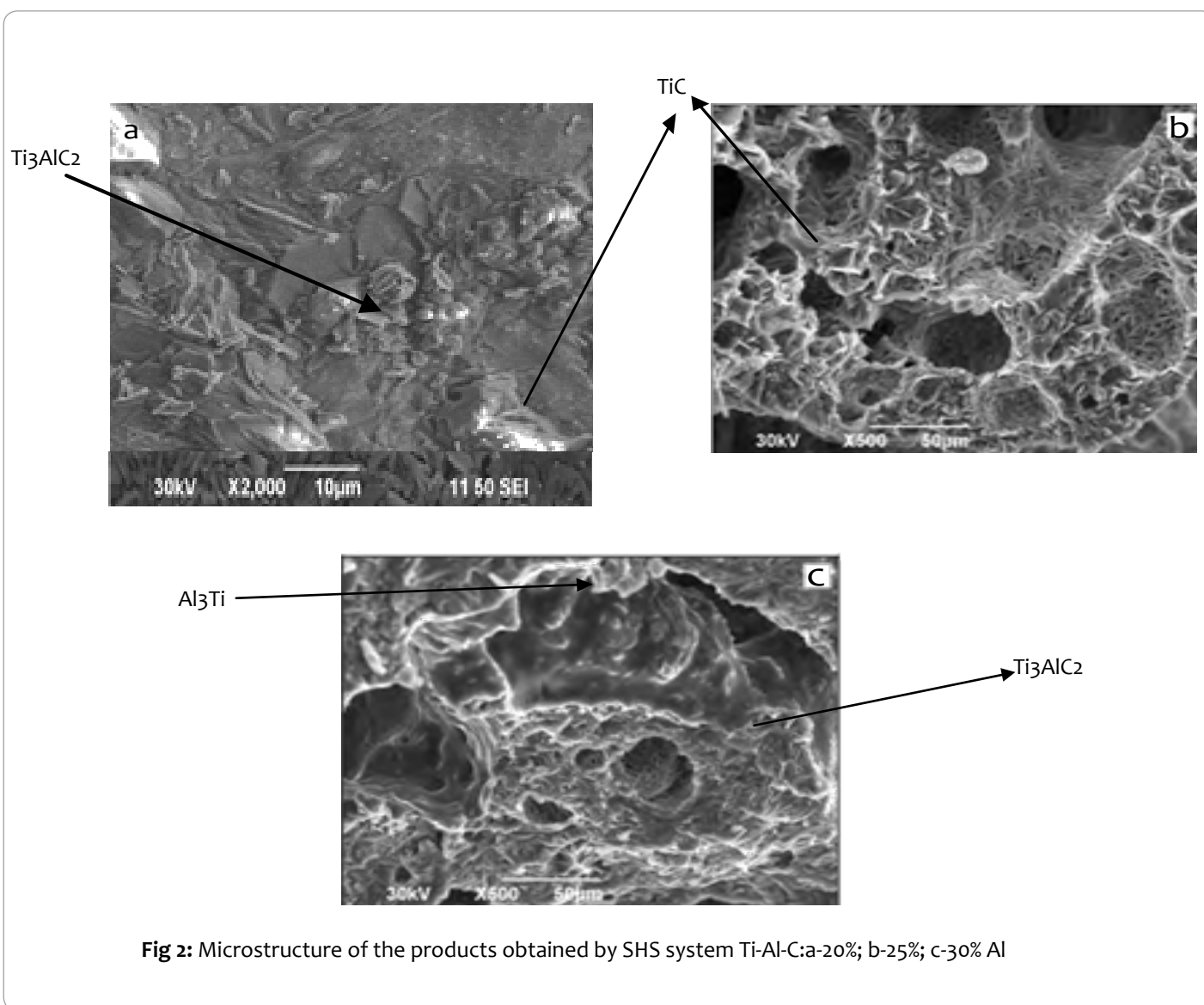
Fig 2: Radiograph of the products at  $\text{TiCo},5 - 0\% \text{Al}$  synthesized from the mixture  $3\text{Ti}+2\text{Al}+2\text{C}$

Ti <sub>2</sub> AlC	Ti <sub>3</sub> AlC <sub>2</sub>	TiC	TiAl <sub>3</sub>	C	TiAl
36,5	38,8	5,1	–	–	19,6
24,5	56,0	5,0	–	>0	14,5
20,7	58,9	–	8,9	5,1	–

**Table 1:** Phase composition of SHS product depending on the carbon additive

The total content of MAX-phases is 94,9%, and the content of TiC does not exceed 5,1%. In Fig. 2, the microstructures of SHS system Ti-C-Al are presented. The microstructure is presented by lamellar grains of Ti<sub>3</sub>AlC<sub>2</sub> with a length of about 10 microns and a thickness of 1-2 microns. Another distinctive feature of

the compositions TiC<sub>0,5</sub> – 20% Al, TiC<sub>0,5</sub> – 25% Al is the presence of round inclusions of TiC in the structure with a size of about 1 micron. The presence of the second phase – Al<sub>3</sub>Ti – is observed in the composition of TiC<sub>0,5</sub> -30% Al along the boundaries of lamellar grains.



**Fig 2:** Microstructure of the products obtained by SHS system Ti-Al-C:a-20%; b-25%; c-30% Al

Rated composition	Phase composition of the MAX- phase	Content of the phase , % mass.
TiCo,5 – 20 % Al	Ti <sub>3</sub> AlC <sub>2</sub>	94,5
	TiC	5,1
TiCo,5 – 25 % Al	Ti <sub>3</sub> AlC <sub>2</sub>	95,0
	TiC	5,0
TiCo,5 – 30 % Al	Ti <sub>3</sub> AlC <sub>2</sub>	100
	Al <sub>3</sub> Ti	-

**Table 2:** The results of the quantitative x-ray phase analysis

products are presented. It is shown that during SHS process, at the aluminum content of 20 and 25%, all the aluminum participates in the formation of the MAX-phase, and the remaining titanium and carbon form a TiC compound. At the aluminum content of 30%, the excess aluminum, which is not involved in the MAX-phase formation, forms an intermetallic Al<sub>3</sub>Ti with titanium.

From the analysis of the microstructure and phase composition of SHS products it follows that the obtained MAX-phase is multiphase. Micro-images show that with the increase in the amount of aluminum, the structure of the MAX-phase becomes more porous. And, on the contrary, with the increase in the amount of titanium, the structure of the MAX-phase becomes denser.

### Conclusions

Thus, the comparison of microscopic images and radiographs showed that in all cases, in the Ti-Al-C system there is a so-called MAX-phase of Ti<sub>3</sub>AlC<sub>2</sub> in the form of the main phase. The Ti<sub>3</sub>AlC<sub>2</sub> phase has a layered structure, where the Ti<sub>3</sub>C<sub>2</sub> carbide layers are bonded together by a monoatomic layer of aluminum (Ti-Al bonds) and have a thickness of about 1 nm [6]. This structure allows to combine simultaneously the increased properties of both hardness and plasticity. In the SHS product of the rated composition of TiCo,5 – 20% Al and TiCo,5 – 25% Al, in addition to the main phase, there is a phase of titanium carbide – TiC, and in the SHS product TiCo,5 -30% Al it lacks, instead, the phase of the intermetallic Al<sub>3</sub>Ti is revealed.

Thus, the multi-component SHS-MAX-phases are multiphase with a homogeneous distribution of structural components that have different physical and chemical properties. The main component of the structure is the MAX-phase Ti<sub>3</sub>AlC<sub>2</sub>, which has rather high thermal stability characteristics, which favorably affects the quality of the powders obtained.

The study was performed with the financial support from the SCS of the RA Ministry of Education and Science in the framework of Armenian-Belarusian joint research project No. 16AB - 48. The experimental data were obtained in the Belarusian State Scientific and Production Corporation of Powder Metallurgy.

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