Study of aluminum content on the structure and phase composition of synthesized aluminum-matrix composites

Yevheniia Kyryliuk, Genadii Bagliuk, Stepan Kyryliuk, Bykov Oleksandr, Yulia Shishkina
Frantsevich Institute for Problems in Materials Science, NASU, Kyiv, Ukraine, 03142, Krzhizhanovsky str., 3

Abstract: *The work shows that there is no significant change in the phase composition of composites with a change in the synthesis temperature, so we can use pre-synthesized heats at a temperature of 950 oC to obtain hot-stamped aluminum-based composites. The best characteristics of the synthesized titanium carbide were obtained for the composition 45Al-11C-44Ti (%, wt.). The lattice period of titanium carbide for this sample is 0.4324, and the particle size of titanium carbide formed after sintering is 0.8-1.5 μm. The influence of the component composition of the initial charge on the features of the structure and the phase composition of the thermally synthesized heat of the Al-C-Ti system was established.*

KEYWORDS: *THERMAL SYNTHESIS, MASTER ALLOY, METAL MATRIX COMPOSITE, ALUMINUM, STRUCTURE, PHASE COMPOSITION, TITANIUM CARBIDE, LATTICE SPACING.*

Today, aluminum matrix composites (AMC) have found the most effective application in automotive, aviation and rocket engineering [1, 2]. This is explained by their low weight, high mechanical, tribotechnical and corrosion properties. The most widely used AMCs are reinforced with ceramic particles, since they have isotropic properties and are made according to a simpler and cheaper technology compared to fibrous and layered aluminum composites. Ceramic powders of aluminum oxide (Al_2O_3) and silicon carbide (SiC) with a size of 10-20 μm with a volume fraction of up to 25% are used as a reinforced phase in most of the manufactured AMCs. However, in recent years, attention has been paid to the use of titanium carbide (TiC) as a dispersion-strengthened phase, since TiC particles can provide AMC with a complex of properties, surpassing all other discretely strengthened AMC [3]. This is due to the fact that TiC has an fcc crystal lattice that coincides with the α-Al crystal lattice, as well as high strength, hardness, and thermodynamic stability. Both powder metallurgy and casting methods are applicable for the production of Al/TiC composites [4]. All these methods can be divided into ex-situ methods, when the reinforced particles are produced in advance, separately, outside the matrix, and then introduced into the matrix during the manufacturing process of the composite, and in-situ methods, when the reinforcing particles are synthesized by chemical reactions directly in the matrix during preparation of the composite. In the case of ex-situ methods, the surface of the powders is usually contaminated with oxides, moisture, adsorbed gases, which impair the wettability of the powders by the matrix and the adhesion between the particles and the matrix. In the case of in-situ methods, the reinforcing particles synthesized in the matrix have a clean, uncontaminated surface, which is important for ensuring strong adhesion to the matrix, they are thermodynamically stable and do not replay with the matrix, they can have a smaller size and a more uniform distribution in the matrix [5, 6], thanks to these advantages, in-situ methods are currently being intensively developed.

Therefore, promising for Al-Ti-C aluminum composites is the direction associated with the preliminary synthesis of titanium carbide in a powder mixture. Depending on the ratio of the content of titanium and carbon in the mixture, when it is heated, in-situ formation of dispersed particles of titanium carbide and ternary titanium carbide occurs, which allows you to control their dispersion and shape. Titanium carbide does not interact with the aluminum matrix and does not form undesirable compounds.

As a result of previous studies [7-10], the authors have accumulated and systematized a certain amount of information on obtaining new composite materials, technologies for preparing powder mixtures (mechanical methods: grinding, chemo-thermal methods: processing in argon and vacuum), consolidation conditions (sintering in controlled gas environments, hot pressing and stamping), which will create new powder composite materials.

The purpose of the work is to study the phase and structure formation of alumino-matrix composite materials strengthened by titanium carbide during their thermal synthesis, as well as to study the influence of the phase composition of composites on the synthesis temperature.

The achievement of the set goal will occur as a result of using the effects of obtaining the required dispersed phases during thermal synthesis, which will allow to purposefully influence the growth rate of grains and artificially create heterogeneity of the composite and thereby form a fine-grained microheterogeneous structure with high physical, mechanical and tribotechnical properties. A new approach in the creation of such powder materials is that refractory reinforcing additives can be formed by recrystallization in thermal synthesis from elementary powders.

Objects and research methods

Aluminum, titanium and graphite powders were used as raw materials for preparing the charge mixture which was subsequently thermal synthesized. To evaluate the effect of blending ratio on the structure and properties of the samples after sintering four compounds of row charge mixtures were chosen. The materials used in this study are summarized in Table 1.

Table 1 *- Chemical composition of charge mixtures for the synthesis of composites*

Number	Composition, % (mass.)		
	Al	C	Ti
1	45	11	44
2	50	10	40
3	55	9	36
4	60	8	32

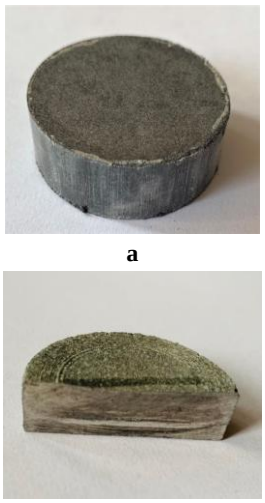
The initial mixtures were pressed under a pressure of 500 MPa, thermal synthesis was carried out in a vacuum furnace of induction heating at a temperature of 950 ° C and held for 1 hour with a sintering rate of 5-10 degrees./ min. Composites was studied using a scanning electron microscope with an energy-dispersive microanalyzer REM 106i, which allows obtaining images of the structure with high spatial resolution and depth of field in reflected (BSE) and secondary (SE) electrons, as well as provides information about the chemical composition and structure. Etching of the samples was performed in a 40% NaOH solution. Microspectral analysis, X-ray phase analysiscand differential thermal analysis (DTA) was also performed.

Results and discussion

The composition of powder mixtures used for the synthesis of Al-Ti-C composites is shown in Table. 1. as a result of synthesis, the initial samples (fig. 1. b, c) turned into fairly strong and relatively dense compacts. After sintering, there was no significant increase in volume and no change in the shape of the compacts due to thermal synthesis at a low heating rate of 5-10 degrees/min. The

advantage of this method is the production of dispersed particles of titanium carbide in the aluminum matrix as a result of the in-situ reaction between the original elementary powders of Al, Ti and C. It is assumed that as a result of the in-situ reaction during the synthesis of the material, the intergrain boundaries between the particles of the strengthening phase and aluminum will be free from oxides, which significantly increases the interfacial strength, and the titanium carbide particles themselves will have a submicron size and a fairly homogeneous volume distribution, which leads to an increase in the mechanical properties of the composite [11, 12].

By adjusting the heating rate, the final porosity of the synthesized compacts can be controlled. The porosity of the synthesized composites varies from 8 to 25%, depending on the aluminum content (45-60% by mass) and the sintering rate from 5 to 10 degrees/min.

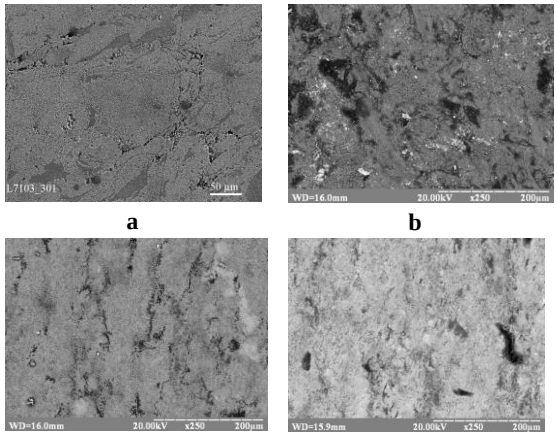


a **b**

c

Figure 1 *- Initial pressing (a) and characteristic macrostructure of samples after thermal synthesis (b, c)*

The results of scanning electron microscopy (SEM) of the samples after thermal synthesis presented in Figure 2 and show that the studied samples have mainly a two-phase structure - a gray field, on which light small particles are evenly distributed. It should be noted that the number of light particles significantly decreases with the increase in the amount of aluminum in the samples.



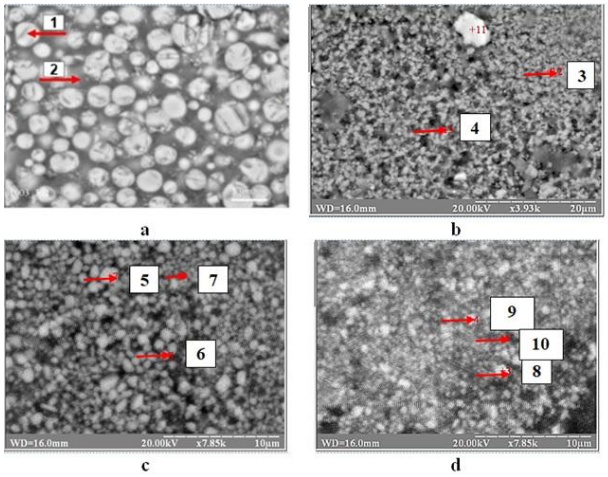
a **b**

c **d**

a – 45Al-11C-44Ti (%, мас.); b – 50Al-10C-40Ti (%, мас.); c – 55Al-9C-36Ti (%, мас.); d – 60Al-8C-32Ti (%, мас.)

Fig. 2 *- SEM microstructure of Al-Ti-C composites after thermal synthesis*

Quantitative analysis showed that the gray field in the photographs of microstructures (Fig. 3) consists mainly of aluminum (Table 2, spectra 2), or a mixture of aluminum and titanium carbide particles (spectra 4, 7-10), light rounded particles (spectra 3, 5 and 6) correspond to the close to stoichiometric composition of titanium carbide. In addition, an increase in the amount of aluminum from 45 to 60% leads to an increase of oxygen in the samples, the percentage of which gradually increases in the samples 55Al-9C-36Ti (%, wt.) and 60Al-8C-32Ti (%, wt.).



a **b**

c **d**

a – 45Al-11C-44Ti (%, мас.); b – 50Al-10C-40Ti (%, мас.); *c* – 55Al-9C-36Ti (%, мас.); d – 60Al-8C-32Ti (%, мас.)

Fig. 3 *-.SEM microstructure of Al-Ti-C composites after thermal synthesis*

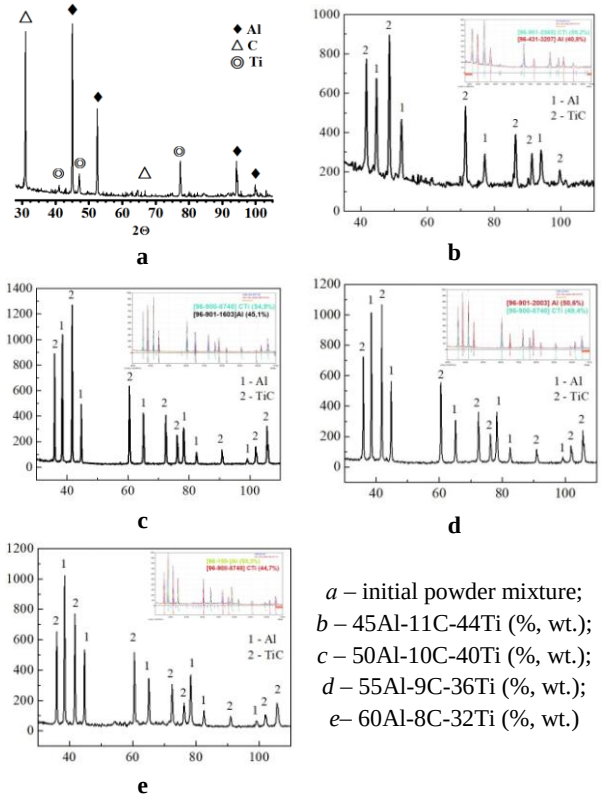
Table 2 *- Content (wt.) of elements at different points synthesized ligatures of the Al-Ti-C system (see Fig. 2)*

№ spectrum	Al, %	Ti, %	C, %	O,%
1	30,33	60,36	9,3	-
2	55,1	44,1	3,86	-
3	15,48	47,01	24,26	13,25
4	30,91	26,18	31,36	11,55
5	8,94	55,40	22,70	12,97
6	20,56	45,38	16,33	17,73
7	21,5	41,60	23,66	13,24
8	35,75	25,06	20,57	19,62
9	35,91	30,13	17,52	16,44
10	45,19	23,52	18,07	13,20

The dispersion of titanium carbide particles in the sample with the composition 45Al-11C-44Ti (%, wt.) lies in the range from 0.8 to 1.5 μm (Fig. 3, a), which is less than in other samples in which the grain size 2-4 μm, respectively (Fig. 3, b - d).

This is explained by the different content of aluminum: the greater its amount surrounds the titanium carbide particles formed in the synthesis process, the larger the diffusion path becomes and the smaller the driving force that promotes the growth of the particles.

Fig. 4 presents the XRD pattern of initial mixture and composites. It can be seen that the initial mixture contains Al, Ti and C as shown in Fig 4a. The XRD results revealed the presents of TiC and Al in all specimens Fig 4 (b, c, d, e)



a – initial powder mixture;
b – 45Al-11C-44Ti (%, wt.);
c – 50Al-10C-40Ti (%, wt.);
d – 55Al-9C-36Ti (%, wt.);
e– 60Al-8C-32Ti (%, wt.)

Fig. 4 *- Diffraction patterns of the initial powder mixture (a) and synthesized Al-Ti-C master alloys (b, c, d, e)*

The influence of the aluminum content on the parameters of the titanium carbide lattice was investigated (Fig. 5). In all samples, the diffraction lines of titanium carbide are at approximately the same angle, which explains the close values of the period of the TiC lattice (Fig. 5).

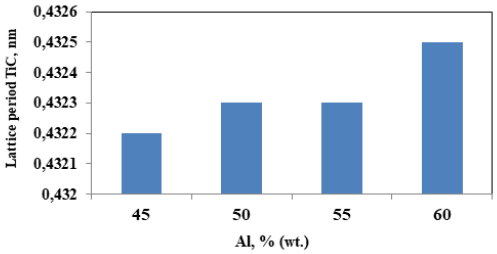
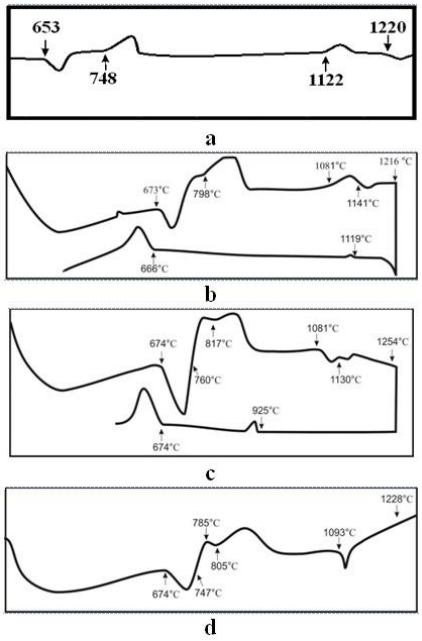


Fig. 5 *- The value of the period of the TiC lattice on the content of aluminum in composites*

However, it should be noted that with an increase in the amount of aluminum in the samples, it can be seen that the value of the lattice period increases. The low value of the period of the TiC lattice may be related to too little amount of aluminum, which in this system at the synthesis temperature of 950 ºC is an environment that facilitates the interaction between titanium and carbon, and its deficiency affects the completeness of the carbide formation reaction.

The differential thermal analysis of the studied systems showed that for all compositions of the initial mixtures, the DTA curves have pronounced endothermic and exothermic peaks (Fig. 6). For samples with a content of 50 and 60 wt. %. aluminum, the minima of the endothermic and the maxima of the exothermic peaks practically coincide (at temperatures of about 640 °C and 800 °C, respectively). In a sample with a content of 45 wt. % of aluminum, endothermic peaks with minima at 635°C are observed, and exothermic peak maxima correspond to temperatures of 812 °C and 800 °C. The endothermic effect at temperatures of 635 - 675 °C is explained by the appearance of a liquid phase. The exothermic peaks on the DTA curves at 800 °C and 812 °C correspond to the in-situ formation of titanium carbide as a result of the solid-phase interaction of titanium with carbon, where the aluminum melt acts as a medium that intensifies the formation of titanium carbide. Based on the fact that this reaction is one of the most thermodynamically advantageous, it can be assumed that the formation of titanium carbide proceeds in this way. The temperature of formation of TiC on DTA may be underestimated, because measurements of a small mass were used for the research. During the synthesis of ligatures, heating of the presses to an average of 1100 °C was recorded.



a – 45Al-11C-44Ti (%, мас.); *b* – 50Al-10C-40Ti (%, мас.);
c – 55Al-9C-36Ti (%, мас.); *d* – 60Al-8C-32Ti (%, мас.)

Fig. 6 *-.Differential thermal analysis of Al-Ti-C powder mixture*

Thus, the results of microspectral analysis, X-ray phase analysis, and differential thermal analysis of samples obtained by thermal synthesis from powder mixtures of different compositions made it possible to establish that for all investigated compositions of the starting mixtures, when they are heated, in-situ separation of TiC titanium carbide particles occurs. The alloys synthesized from a mixture of the compositions 45Al-11C-44Ti (%, wt.) and 50Al-10C-40Ti (%, wt.) are characterized by the greatest dispersion of the particles of the strengthening phase, which were chosen for obtaining aluminum composites by the method of hot stamping, together with the sample composition 60Al-8C-32Ti (%, wt.), in which complex titanium carbide appears.

Conclusions

1 The results of microspectral analysis, X-ray phase analysis, and differential thermal analysis of samples synthesized from powder mixtures of different compositions made it possible to establish that for all the compositions of the initial mixtures, upon

heating, in-situ formation of TiC titanium carbide particles and a small amount of titanium aluminides occurs.

2. It is shown that there is no significant change in the phase composition of the composites with a change in the synthesis temperature, therefore, in the future, we can use pre-synthesized heats at a temperature of 950 °C to obtain hot-forged aluminum-based composites. The best characteristics of the synthesized titanium carbide were obtained for the composition 45Al-11C-44Ti (%, wt.). The lattice period of titanium carbide for this sample is 0.4324, and the particle size of titanium carbide formed after sintering is 0.8-1.5 μm.

3. The influence of the component composition of the initial charge on the structural features and phase composition of the thermally synthesized heat of the Al-C-Ti system was determined. It is shown that in the case of using a charge with a stechiometric carbon content in relation to titanium, the predominant strengthening phase of the alloy is titanium carbide.

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