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The study of the effect of heat treatment on the properties of the AMg2–10%TiC and AMg6–10%TiC composite materials produced by self-propagating high-temperature synthesis

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Abstract: Dispersion-strengthened composite materials belong to the group of promising structural materials characterised by a diverse combination of properties. The paper considers examples of the creation and heat treatment of composite materials based on aluminium alloys strengthened by the titanium carbide dispersed phase characterised by high hardness, elastic modulus, and good melt wettability. At present, self-propagating high-temperature synthesis (SHS) is the most accessible, inexpensive and effective way to obtain them. The authors substantiate the expediency and show their successful experience of the formation in the composition of the AMg2 and AMg6 industrial alloys of a titanium carbide dispersed phase with a particle size of 130 nm in an amount of up to 10 wt. % using the SHS method, which makes it possible to increase the hardness of the alloys. Additional heating of the AMg2–10%TiC and AMg6–10%TiC samples after synthesis also contributes to the further increase in hardness. The complex of studies of physical, mechanical and operational characteristics presented in the paper was carried out to compare the properties of the work-hardened matrix alloys and the samples of composite materials before and after heating. The test results showed that heat treatment reduces the porosity of the composites and significantly increases their hardness and microhardness. A slight decrease in compressive strength at a significant increase in wear resistance is observed. It was found that composite materials are characterised by high corrosion resistance to carbon dioxide and hydrogen sulfide corrosion corresponding to the level of matrix alloys. The results obtained allow recommending the developed materials for the production of parts of the connecting rod and piston group, bearings and other wear-resistant parts of friction units.

Keywords: composite material; AMg2–10%TiC; AMg6–10%TiC; titanium carbide; heat treatment; self-propagating high-temperature synthesis.

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INTRODUCTION

One of the most promising ways to improve the mechanical characteristics of conventional aluminium alloys, is to introduce dispersed particles of an additional phase into their composition, for which ceramic compounds – oxides, carbides, nitrides, borides, etc. are most often used [1; 2]. However, in the case of an aluminium matrix, the most suitable phase for reinforcement is the titanium carbide phase, which has crystal lattice parameters as close as possible to aluminium, and possesses high hardness, elastic modulus, low density, and good wettability [3; 4]. The most common way to produce such composite materials is the method of mechanical mixing of particles into an aluminium melt, however, this approach excludes the possibility of obtaining a highly dispersed titanium carbide phase, since the introduced particles are prone to agglomeration, and often contain impurity adsorbed compounds, that prevent the complete assimilation of particles in the melt. In this regard, the most appropriate option is the formation of dispersed particles of titanium carbide directly in the melt from the initial elemental powders of titanium and carbon or their compounds [5–7].

This technology based on the method of self-propagating high-temperature synthesis (SHS) was developed and used at the Chair “Metal Science, Powder Metallurgy, Nanomaterials” of Samara State Technical University. According to the results of the studies, the possibility of successful synthesis of composite materials of the Al–10%TiC, Al–5%Cu–10%TiC, Al–5%Cu–2%Mn–10%TiC, etc. compositions characterised by increased mechanical characteristics has already been shown [8; 9].

A review of current publications showed a steady tendency towards reinforcement of industrial alloys, that have long been developed and actively used with the titanium carbide phase [10]. For example, in the study [11], the Al–10%TiC addition alloy was introduced into the composition of the 2014 aluminium matrix alloy, which allowed increasing the strength from 118 to 147 MPa, and the hardness from 61 to 94 HV. In [12], based on the AA 6063 alloy (analogue of AD 31), hybrid-reinforced samples were produced using the SHS method, including 5 vol. % Al_2O_3 and 5 vol. % TiC. Then, since the matrix alloy is a thermally hardenable one, the resulting composite was subjected to T6 treatment in the form of quenching at 530 °C and artificial ageing at 175 °C. It was found that composite material samples demonstrated acceleration in aging kinetics. To achieve a maximum hardness of 78 HB after ageing, they required 2–4 h, whereas for an unreinforced alloy, this time was 6–8 h, and the material hardness did not exceed 65 HB. The authors explain the observed accelerated aging by an increase in the dislocation density near dispersed particles. This is associated with a large difference in the thermal expansion coefficient of these particles and the matrix alloy (Al_2O_3 and TiC particles have a TLEC of $8\cdot10^{-6}$/K^{-1}, Al – $23\cdot10^{-6}$/K^{-1}), as well as accelerated diffusion of dissolved atoms and modification of the base alloy. It is obvious that the presence of additional phase dispersed particles affects the order and intensity of structural transformations in the composition of conventional alloys. On the other hand, in addition to this, completely new effects may arise that are not typical for matrix alloys.

Thus, in the work [13], a composite material based on the AMg1 alloy containing 5 wt. % SiC was obtained by the mechanical mixing method, and then the possibility of its thermal hardening was shown for the first time. In particular, it was found that quenching at a temperature of 550 °C and subsequent ageing at a temperature of 160 °C leads to an increase in hardness from 770 to 1000 HB and strength to 152 MPa, and in combination with subsequent rolling, it leads to an increase in hardness to 1530 HB and strength up to 236 MPa.

Such an increase in the strength characteristics of aluminum-magnesium alloys is extremely important, since they are widespread due to their low cost, good deformability, corrosion resistance, and weldability; however, they are not good in strength [14]. The alloys under consideration contain microadditives of alloying elements (Fe, Si, Mn, Ti, etc.), which contribute to solid solution strengthening, but their quantity is too small to significantly increase the strength characteristics, so the alloys are additionally hardened by plastic deformation. However, the use of cold hardening leads to a decrease in ductility, so annealing is the final stage after plastic deformation, during which partial or complete removal of strain hardening occurs, which leads to a decrease in strength [15; 16].

Previously, studies on the production of AMg2–10%TiC and AMg6–10%TiC composite materials by SHS method were carried out, which showed that in both cases, an active and rapid SHS reaction was observed, and the fractures of the samples were characterised by a uniform grey colour without the remains of unreacted charging material [17]. After synthesis, according to X-ray microanalysis and X-ray phase analysis, the composition of the composites contained the target phase of titanium carbide (with a particle size of 130 nm), as well as magnesium, apparently in the precipitated β-phase (Al_3Mg_2), which was not detected due to its small amount. Hardness measurements showed an increase in the values for the AMg2 base from 59.4 to 64.4 HB, for the AMg6 base – from 83 to 90.9 HB. Then the samples were additionally heated with following cooling in air. It was found that heating at 150 °C and holding for 2 h leads to an increase in the hardness of AMg2–10%TiC to 67.6 HB, and heating at 230 °C and holding for 3 h of the AMg6–10%TiC sample leads to the hardness of 93 HB. Using phase analysis of the samples, the β-phase was detected in both cases, which indicates its additional precipitation after heating [17]. However, other properties than hardness of the obtained samples were not studied.

The purpose of the work is to study and compare the basic physical, mechanical and operational characteristics, of the AMg2–10%TiC and AMg6–10%TiC composite materials before and after heat treatment.

METHODS

To compare the results, all tests were carried out on matrix alloys in a cold-worked state (AMg2N and AMg6N) and composite materials based on them. AMg2 and AMg6 alloys were used as a matrix for creating melts with digital markings of these alloys as 1520 and 1560, respectively, according to GOST 4784-2019 (Table 1).

To obtain a charge mixture, titanium powders (grade TPP-7, TU1715-449-05785388) and carbon (P-701, GOST 7585-86), taken in a stoichiometric ratio, were mixed with 5 % by weight of the Na_2TiF_6 salt charge (GOST 10561-80). The resulting composition was divided into 3 equal portions, each of which was alternately introduced into AMg2 or AMg6 melts heated to a temperature of 900 °C in a graphite crucible of a PS-20/12 melting furnace, then synthesised and poured into a steel casting mold. Thermal treatment of the samples was carried out in a laboratory chamber furnace SNOL, with an operating temperature of up to 1300 °C. The experimental determination of the density of the samples was carried out using hydrostatic weighing in accordance with GOST 20018-74. The theoretical maximum possible density of a nonporous casting composite was calculated using the formula

$$\rho_T = \frac{\rho_1\rho_2}{n\rho_1 + (1-n)\rho_2},$$

where ρ_T is theoretical density, kg/m^3;
ρ_1 is crystalline aluminium density, kg/m^3;
ρ_2 is second phase (titanium carbide) density, kg/m^3;
n is titanium carbide mass content in the composite.

The calculation of actual porosity was carried out using the formula

$$P = 1 - \frac{\rho_e}{\rho_T},$$

where ρ_e is experimentally measured density, kg/m^3;
P is porosity, %.

When calculating, the density of aluminium was taken to be 2700 kg/m^3, the density of the titanium carbide phase was 4920 kg/m^3, n=0.1.

The hardness of the obtained experimental samples was determined using a TSh-2M hardness tester according to GOST 9012-59. The microhardness of the samples was studied using a PTM-3 standard microhardness tester according to GOST 9450-76 by indentation of a diamond pyramid with a square base and an interface angle at the apex of 136°. The weight on the indenter was 100 g. Compression tests were carried out on the III type samples, with a diameter of 20 mm according to GOST 25.503-97. The moment of appearance of the first cracks was determined visually. Corrosion resistance was tested according to GOST 13819–68 in the Coat Test 3.3.150.150 autoclave complex under the following conditions: aqueous solution of 5%NaCl; gas phase 1 MPa CO_2, 0.5 MPa H_2S, 3.5 MPa N_2 at a temperature of 80 °C; duration is 240 h; total pressure is 5 MPa. Corrosion resistance parameters were calculated, according to GOST 9.908-85. Tribological tests were carried out using the "Universal-1B" universal tribological complex, according to the ring–plane test scheme; counterbody material – steel 40X; normal contact load is 380 N; counterbody rotation speed – 600 rpm; test duration is 30 min or until complete setting occurs.

RESULTS

As a result of determining the physical properties (Table 2) of the AMg2N, AMg6N alloys, and the AMg2–10%TiC and AMg6–10%TiC composite materials, it was identified that the density of the composite materials is higher than the density of the matrix alloys, which is obviously related to the presence of a reinforcing ceramic phase of titanium carbide. Test results show, that additional heating leads to a decrease in the porosity of composite materials, due to an improvement in the adhesive bond between the matrix and the filler.

The study of mechanical characteristics (Table 3) showed that the ceramic phase reinforcement of matrix alloys leads to an increase in their hardness and microhardness. Additional heating of the AMg2–10%TiC and AMg6–10%TiC composite materials promotes an increase in hardness by 13 and 12 %, respectively, and microhardness by 22 and 7 %, respectively. Reinforcement, with a highly dispersed titanium carbide phase in combination with heat treatment does not have a strong negative effect on the yield strength and relative strain.

The results of determining the tribological characteristics (Table 4) of the AMg2N, AMg6N alloys and AMg2–10%TiC, AMg6–10%TiC composite materials before and after heat treatment, showed that reinforcement in combination with heat treatment leads to a significant decrease in the friction ratio and wear rate. The lowest tribological properties are observed in the original AMg2N and AMg6N alloys: they showed wear during setting and abrasive

Table 1. *Chemical composition of the AMg2 and AMg6 alloys*
Таблица 1. *Химический состав сплавов АМг2 и АМг6*

Alloy	Element content, %						
	Al	**Mg**	**Fe**	**Si**	**Mn**	**Cu**	**Ti**
AMg2	95.3–98.00	1.8–2.8	<0.4	<0.4	0.2–0.6	<0.1	<0.1
AMg6	91.1–93.68	5.6–6.8	<0.4	<0.4	0.5–0.8	<0.1	<0.1

Table 2. *Physical properties of the AMg2, AMg6 alloys and AMg2–10%TiC, AMg6–10%TiC composite materials before and after heat treatment*
Таблица 2. *Физические свойства сплавов АМг2, АМг6 и композиционных материалов АМг2–10%TiC, АМг6–10%TiC до и после термической обработки*

Alloys and composite materials on their base	Theoretical density, ρ_T, g/cm^3	Experimental density, ρ_e, g/cm^3	Porosity, P, %
AMg2N	2.690	–	–
AMg2–10%TiC, without HT	2.820	2.797±0.05	0.82
AMg2–10%TiC, after HT	2.820	2.820±0.03	0.00
AMg6N	2.640	–	–
AMg6–10%TiC, without HT	2.768	2.739±0.06	1.00
AMg6–10%TiC, after HT	2.768	2.768±0.04	0.00

Note. HT (heat treatment) is heating at T=150 °C for 3 h.
Примечание. НТ (термическая обработка) – нагрев при T=150 °C в течение 3 ч.

wear, which led to rapid surface layer destruction; high values of the friction ratio and wear rate indicate unacceptable processes occurring in the friction zone. AMg2–10%TiC and AMg6–10%TiC samples showed significantly better tribological characteristics compared to the matrix alloy, however, they had scuff marks, on which friction was established with a ratio of about 0.1. The same samples, after additional heating according to the recommended conditions, showed the lowest values of the friction ratio, low wear rate and good conformability.

Table 5 shows that the titanium carbide ceramic phase reinforcement of the AMg2 and AMg6 alloys leads to a decrease in the corrosion resistance level.

DISCUSSION

Since composite materials, especially those obtained by the SHS method, are characterised by increased porosity, which has a significant impact on their properties, the density and porosity of the resulting materials were initially determined. The study of the porosity of the samples (Table 2) showed that after synthesis, the deviation from the calculated value does not exceed 1 %, and after heat treatment, it decreases to zero, which may be caused by a change in the composition and structure of the interphase boundaries and an improvement in the quality of the "matrix – filler" bond.

The titanium carbide phase is characterised by increased hardness, and, accordingly, low ductility, therefore, the main mechanical characteristic of composite materials containing it, as a rule, is compressive strength. However, since complete destruction of such samples does not occur, the yield strength value was used as an evaluation criterion, which corresponds to the temporary fracture resistance upon the occurrence of the first cracks. The obtained values of the strength, of the as-cast material (Table 3) comparable to the values after cold hardening, are obviously caused by the following factors. The first is the action of the Hall – Petch mechanism determined by the role of dispersed particles as the alloy crystallisation centres. The second is the Orowan mechanism, the essence of which is that the resistance to motion of dislocations, increases with decreasing distance between particles. The third is the emergence of difficulties for the motion of dislocations due to the formation of additional dislocations caused by the mismatch of the coefficients of thermal expansion and elastic modulus of the matrix material and the reinforcing phase particles. The study [18] showed that the introduction of reinforcing TiC particles with a size of 40–100 μm, into the AK12M2MgN aluminium alloy, using the mechanical mixing method, leads to a decrease in the compressive deformation degree from 17.01 to 12.65 %, and the ultimate compressive strength from 489 to 470 MPa, while the hardness increases by 30–50 HB. One can conclude that the presence of the carbide phase does not lead to an increase in the strength characteristics, but does contribute to an increase in hardness.

Since it was found that the presence of carbide phase particles contributes to an overall increase in the hardness of the resulting materials, it was assumed that this could have a positive effect on their wear resistance, so the tribological properties were further investigated. Low values of the friction ratio, low wear rate and good conformability of AMg2–10%TiC and AMg6–10%TiC composite materials after the optimal heat treatment mode, are obviously related to an increase in the quality of the interfacial bond, as well as additional precipitation of the solid intermetallic β-phase (Al_3Mg_2) [19].

One of the main advantages of aluminum-magnesium alloys is their high corrosion resistance. The study of this characteristic in an environment of CO_2 and H_2S gases at an elevated temperature of 80 °C, showed that the samples

Table 3. *Mechanical characteristics of the AMg2, AMg6 alloys and AMg2–10%TiC, AMg6–10%TiC composite materials before and after heat treatment*

Таблица 3. *Механические характеристики сплавов АМг2, АМг6 и композиционных материалов АМг2–10%TiC, АМг6–10%TiC до и после термической обработки*

Alloys and composite materials on their base	Uniaxial compressive strength test		Hardness HB	Microhardness HV, MPa
	$\sigma_т^c$, MPa	ε, %		
AMg2N	290±10	69.19	59.4±20	608±10
AMg2–10%TiC, without HT	271±13	59.70	59.4±20	736±15
AMg2–10%TiC, after HT	298±10	61.50	67.6±20	745±18
AMg6N	449±15	32.00	83.0±19	991±21
AMg6–10%TiC, without HT	403±18	19.00	90.9±19	1020±20
AMg6–10%TiC, after HT	395±19	14.00	93.0±19	1069±19

Note. HT (heat treatment) is heating at T=150 °C for 3 h.
Примечание. HT (термическая обработка) – нагрев при T=150 °C в течение 3 ч.

***Table 4.** Tribotechnical properties of the AMg2, AMg6 alloys and AMg2–10%TiC, AMg6–10%TiC composite materials before and after heat treatment*
***Таблица 4.** Триботехнические свойства сплавов АМг2, АМг6 и композиционных материалов АМг2–10%TiC, АМг6–10%TiC до и после термической обработки*

Alloys and composite materials on their base	Wear rate, μm/h	Friction ratio	Self-heating temperature, °C
AMg2N	37.6±5.2	<0.3	71
AMg2–10%TiC, without HT	6.4±1.6	0.11…0.12	65
AMg2–10%TiC, after HT	4.0±1.3	0.07…0.08	56
AMg6N	15.5±4.1	0.13...0.15	70
AMg6–10%TiC, without HT	3.5±0.6	0.07…0.09	59
AMg6–10%TiC, after HT	4.2±1.2	0.08…0.10	66

Note. HT (heat treatment) is heating at T=150 °C for 3 h.
Примечание. НТ (термическая обработка) – нагрев при T=150 °C в течение 3 ч.

***Table 5.** Corrosion resistance of the AMg2, AMg6 alloys and AMg2–10%TiC, AMg6–10%TiC composite materials before and after heat treatment*
***Таблица 5.** Коррозионная стойкость сплавов АМг2, АМг6 и композиционных материалов АМг2–10%TiC, АМг6–10%TiC до и после термической обработки*

Alloys and composite materials on their base	Factor		
	Weight loss per unit area, Δm, kg/m^2	Corrosion rate, V, g/(m^2·h)	Corrosion depth index, π, mm/year
AMg2N	0.160	0.666±0.04	0.0021
AMg2–10%TiC, without HT	0.095	0.416±0.02	0.0014
AMg2–10%TiC, after HT	0.108	0.450±0.03	0.0014
AMg6N	0.231	0.962±0.06	0.0030
AMg6–10%TiC, without HT	0.151	0.627±0.04	0.0021
AMg6–10%TiC, after HT	0.208	0.868±0.02	0.0027

Note. HT (heat treatment) is heating at T=150 °C for 3 h.
Примечание. НТ (термическая обработка) – нагрев при T=150 °C в течение 3 ч.

both of matrix alloys and of composite materials, before and after heating, have a depth corrosion rate at the level of 0.001–0.005 mm/year (Table 5). This indicates the high corrosion resistance of the AMg2–10%TiC and AMg6–10%TiC composite materials and allows classifying them as rather resistant metals [20].

CONCLUSIONS

According to the results of a set of studies of properties, the developed composite materials AMg2–10%TiC and AMg6–10%TiC produced by the SHS method and subjected to additional heating showed, a higher level of hardness, microhardness, wear and corrosion resistance compared to the matrix alloys AMg2 and AMg6 in the cold hardening state. Thus, reinforcement with a highly dispersed phase of titanium carbide in combination with heat treatment, is an appropriate way to increase mechanical and operational characteristics, as it helps to avoid the labour-intensive operation of cold strain hardening (cold hardening). Based on the data obtained, composite materials can be recommended to produce connecting rod and piston group parts, bearings, and other wear-resistant parts of friction units.

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Влияние термической обработки на свойства композиционных материалов АМг2–10%TiC и АМг6–10%TiC, полученных методом самораспространяющегося высокотемпературного синтеза

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Аннотация: Дисперсно-упрочненные композиционные материалы относятся к группе перспективных конструкционных материалов, отличающихся разнообразным сочетанием свойств. В работе рассмотрены примеры создания и термической обработки композиционных материалов на основе алюминиевых сплавов, упрочненных дисперсной фазой карбида титана, для которой характерны высокая твердость, модуль упругости и хорошая смачиваемость расплавом. В настоящее время наиболее доступным, недорогим и эффективным способом получения этих материалов является самораспространяющийся высокотемпературный синтез (СВС). Обоснована целесообразность и показан собственный успешный опыт формирования в составе промышленных сплавов АМг2 и АМг6 дисперсной фазы карбида титана с размером частиц от 130 нм в количестве до 10 мас. % методом СВС, что позволяет увеличить твердость сплавов. Проведение после синтеза дополнительного нагрева образцов АМг2–10%TiC и АМг6–10%TiC также способствует последующему повышению твердости. Представленный в статье комплекс исследований физических, механических и эксплуатационных характеристик выполнен с целью сравнения свойств матричных сплавов в нагартованном состоянии и образцов композиционных материалов до и после нагрева. Результаты испытаний показали, что проведение термической обработки способствует снижению пористости композитов и значительному повышению их твердости и микротвердости. Наблюдается также незначительное снижение прочности на сжатие при существенном повышении износостойкости. Установлено, что композиционные материалы характеризуются высокой коррозионной стойкостью к углекислотной и сероводородной коррозии, соответствующей уровню матричных сплавов. Полученные результаты позволяют рекомендовать разработанные материалы для изготовления деталей шатунно-поршневой группы, подшипников и других износостойких деталей узлов трения.

Ключевые слова: композиционный материал; АМг2–10%TiC; АМг6–10%TiC; карбид титана; термическая обработка; самораспространяющийся высокотемпературный синтез.

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