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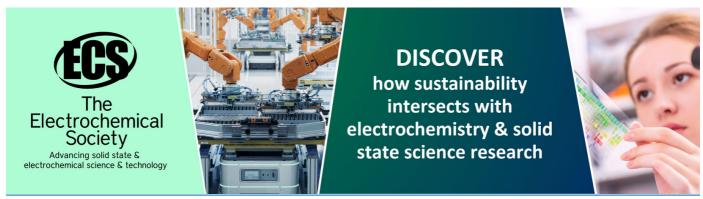
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Effect of alloying on structure and properties of particle – reinforced aluminum matrix composites Al/TiC produced by **SHS** in aluminum melt

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Abstract. Publications devoted to the properties and methods of fabrication of particlereinforced aluminum matrix composites and nanocomposites of Al/TiC were analyzed. The emphasis was on the application of the method of self-propagating high-temperature synthesis (SHS) for preparation of Al/TiC composites. The results of our own studies of fabrication of Al/10wt.%TiC composite using SHS in the aluminum melt in the presence of alloying additives of copper (5 wt.%) and manganese (2 wt.%) were presented. It was shown that the alloying of Al/10wt.%TiC cast composite with copper and manganese leads to a more uniform distribution of the synthesized nano - and ultrafine particles of titanium carbide, as well as to nearly a twofold increase of strength while maintaining a sufficiently high level of ductility.

1. Introduction

Today aluminum matrix composites (AMCs) have found greatest use among metal matrix composites in industry, including automobile, aircraft and rocket manufacture [1, 2]. This is due to their low weight, high specific strength, corrosion resistance, good processing properties, high load bearing capacity and wear resistance in wide temperature and power range of operation. AMCs, discretely reinforced with ceramic particles, are the most widely used because they have isotropic properties and are produced by more simple and cheap technology compared with fibrous and layered AMCs. Ceramic powders of aluminum oxide (Al₂O₃) and silicon carbide (SiC) of size 10-20 µm with a volume fraction up to 25% are used as a reinforcing phase in most produced AMCs. However, in recent years, much attention is paid to the use of titanium carbide (TiC) as a reinforcing phase, because the particles of TiC can give to AMCs a complex of properties, surpassing all other discretely reinforced AMCs [3]. This is because TiC possesses the FCC crystal lattice, coincident with the α -Al crystal lattice with the closest size, higher strength, hardness, thermodynamic stability.

Both methods of powder metallurgy with initial matrix aluminum in the form of a powder and casting methods with initial matrix aluminum in the form of a melt are applicable for the fabrication of Al/TiC composites [4]. All these methods can be divided into ex-situ methods, when the reinforcing particles are made in advance, separately, outside the matrix, and then are introduced into the matrix in the process of fabrication of the composite, and in-situ methods, when the reinforcing particles are synthesized by chemical reactions directly in the matrix during fabrication of the composite. In the case of ex-situ methods, the surface of powders is normally contaminated with oxides, moisture,

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adsorbed gases, which deteriorate the wettability of powders with matrix and the adhesion between particles and matrix. In the case of in-situ methods, the reinforcing particles, synthesized in the matrix, have clean uncontaminated surface, which is important to ensure strong adhesion with the matrix, they are thermodynamically stable and do not enter into chemical reactions with the matrix, they can have a smaller size and more uniform distribution in the matrix [5, 6]. Due to these advantages, the in-situ methods have been intensively developed, and currently, there are many such methods as mechanical alloying, reactive hot pressing, exothermic dispersion, direct melt oxidation, flux-assisted synthesis, self-propagating high-temperature synthesis (SHS) or combustion synthesis [4, 6].

Distinctive features of the SHS method are ease of performance and high efficiency at low energy consumption. In the early 90s of the last century, the SHS was used to obtain a composite with high content of ceramic phase (Al/30 vol.%TiC) through combustion of the mixture of elemental powders of Ti, C and Al, followed by high pressure consolidation of the hot SHS product [7]. This approach can be attributed to in-situ powder methods. Further, more development was given to casting methods of in-situ producing AMCs with carrying out the combustion synthesis of reinforcing phase TiC from elemental Ti and C powders (and sometimes Al) in the aluminum melt, which are characterized by simple equipment, the possibility of producing castings of more complex shapes and large dimensions [3]. In the liquid-phase SHS processes, a more continuous contact between TiC particles and the aluminum matrix with stronger adhesive bond is achieved. In fairness it should be noted, the main drawback of liquid-phase methods lies in the fact that the reinforcing ceramic particles form clusters and agglomerates along the grain boundaries of the aluminum matrix alloy, particularly at elevated content of reinforcing phase, which can significantly degrade the mechanical properties of cast AMCs and restricts the content of the reinforcing phase [8].

In recent years, the special attention is paid to the development of aluminum matrix composites with nanoscale reinforcing phase TiC [9-12]. The nanocomposites are attractive because of the action of other mechanisms of strengthening than in the case of larger reinforcing particles of micron size, the nanocomposites can have a high level of strength properties at a much lower content of reinforcing phase, and as a result, high ductility [13, 14]. For example, nanocomposite (Al-5wt.%Cu)/0.5wt.%TiC showed in tension along with the high strength of 540 MPa the unique ductility δ =19%, which was almost 3 times higher than that of the original matrix alloy Al-5%Cu with indicators of 485 MPa and 6.6%, respectively [9]. This nanocomposite was fabricated ex-situ at 800°C by dissolution in molten alloy Al-5%Cu of master alloy Al-TiC, previously synthesized by SHS through combustion of a mixture of powders of Al and Ti with carbon nanotubes (CNTs) in vacuum. The same unique mechanical properties in tension at room temperature and creep resistance at 453-493 K were observed in nanocomposite (Al-5wt.%Cu)/0.3wt.%TiC fabricated in-situ when conducting SHS reactions in the mixtures of Al-Ti-CNTs in molten Al-5%Cu [10, 11]. Note, that the unique ductility was observed with a small content of nanoparticles (0.3 to 0.5) wt.%TiC in the cast composites; with the greater content, the ductility decreased due to the agglomeration of nanoparticles in the cast matrix. The nanocomposites of (Al-Zn-Mg-Cu)/TiC with high content and uniform distribution of reinforcing phase TiC (20, 25 and 30 vol.%) was successfully fabricated in-situ by SHS and hot press consolidation in Al-Ti-C/CNTs system [12]. Partial and complete replacement of carbon in the form of carbon black with carbon nanotubes (CNTs) in the starting mixture of powders leds to a significant decrease in the size of the synthesized TiC nanoparticles and a significant improvement in strength and ductility under compression and wear resistance of nanocomposites.

Along with these factors, a significant role in forming the properties of aluminum matrix composites and nanocomposites plays the alloying of aluminum matrix. One of the most frequently used alloying elements is copper with usually injected amount of 4.5-5.3 wt.% because this concentration may be fully dissolved in the aluminum matrix according to the phase diagram of Al-Cu [6, 9–11, 15]. Released during the cooling, the θ ' phase CuAl₂ leads to the precipitation strengthening of the alloy. In general, the alloying with copper is responsible for several mechanisms of strengthening, which would also include the solid solution strengthening and strengthening due to the grain refinement of matrix alloy [16]. In addition to copper, there is information about the successful

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alloying of aluminum matrix with other elements. When alloying with magnesium and zinc, the melting point of the aluminum alloy decreases, the synthesis of the reinforcing TiC particles occurs at a lower temperature that leads to smaller particle size [12]. An increase in the concentration of silicon in the composition of the aluminum base also decreases the size of grains of titanium carbide obtained by the SHS, and therefore, significantly increases the wear resistance of composites [17]. Positive results on the effect of alloying elements Mg, Zn and Sn on the microstructure and compressive properties of the composite Al/TiC with high TiC content (50 wt.%) are presented in [18]. Alloying with 1 wt.% molybdenum improves the wettability due to the formation of molybdenum sheath around the particles of titanium carbide, and the result is a more uniform distribution of particles in the matrix [19]. Thus, the efficiency of the alloying of aluminum matrix composites is quite obvious.

The aim of this study was to study the influence of alloying elements of copper and manganese on the structure and properties of promising composite Al/10%TiC (hereinafter wt. %) fabricated by SHS of reinforcing phase TiC in the aluminum melt. This study is a continuation of studies on the possibility of obtaining a cast nanocomposite of Al-10%TiC based on the melt of pure aluminum [3, 20].

2. Materials and methods of research

Components used for the study are: pig aluminum of A7 brand (purity of 99.7%), titanium powder of TPP-7 brand (97.95%; average particle size 230 μ m), copper powder of PMS-1 brand (99.5%; average particle size 100 μ m), manganese powder Mn-95 (95.0%; 1-3 mm), carbon black P-701 (99.7%; average particle size 70 nm; average size of agglomerates 1 μ m), halide salt Na₂TiF₆ (99.0%; 15 μ m).

The charge for SHS, consisting of powders of titanium and carbon in stoichiometric ratio, was subjected to preliminary drying (2–3 hours) and mechanical activation in drum ball mill (1 h). The salt Na_2TiF_6 was added to the charge in amounts of 5–10% by weight of the charge. The charge portions of mass about 6-8 grams were wrapped in aluminum foil with a thickness of 50-100 μ m.

For the formation of matrix alloy of aluminum-copper, the copper powder in amount of 5% mass of the melt was first introduced into the aluminum melt at 800°C, followed by aging for 30 min. Next, the melt was heated up to 900°C, and the charge portions were introduced alternately in the melt. For the formation of the matrix alloy of aluminum-copper-manganese, the copper powder in an amount of 5% mass of the melt was first introduced into the aluminum melt at 800°C, followed by aging for 30 min, then the melt was heated up to 850°C, and manganese in amount of 2% mass of the melt was introduced into the melt. This was followed by heating the melt up to 900°C and finally the charge portions were introduced alternately in the melt. Each charge portion was kept under the melt level to the beginning of the active SHS reaction attended with spark – and gas evolution. During the reaction the melt was stirred thoroughly. Upon completion of the reaction, the melt was kept for 5 minutes, stirred and poured into a steel chill with a hole diameter of 20 mm to obtain a cast sample of the composite in the form of a rod with a diameter of 20 mm and a length of 110 mm.

The obtained samples of cast AMCs were notched on the side and ruptured by impact bending to determine a mode of fracture and a visual appearance of fracture surface. A brittle fracture with homogeneous gray fracture surface testified that the SHS process was fully completed and a desired structure of the composite was formed. A ductile fracture and heterogeneous "dirty" structure, with inclusions of unreacted charge, testified that the SHS reaction was not fully.

The samples with homogeneous structure were used to make sections with the polishing machine POLILAB. To reveal the microstructure, these samples were etched with a solution of acids (50% HF+50%HNO $_3$) for 10-15 seconds. Metallographic analysis was performed on scanning electron microscope Jeol JSM-6390A. The phase composition was analyzed by the method of X-ray diffraction (XRD). Shooting X-ray spectra was performed on an automated diffractometer ARL X'trA (Thermo Scientific) using Cu-radiation with continuous scanning in the range of angles 20 from 20 to 80 degrees with a speed of 2 deg/min. Hardness of the obtained experimental samples were determined by Brinell hardness tester TSH-2M. Tensile tests were carried out on a tensile testing machine Inspekt 200.

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3. Results and discussion

Figure 1 shows the microstructure of the sample obtained on the basis of Al-5% Cu with addition of 10%Na₂TiF₆ to the SHS charge.

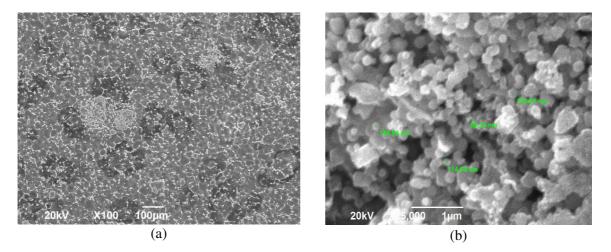


Figure 1. SEM image of (Al-5%Cu)/10%TiC sample.

After the occurrence of the intense SHS reaction, the sample with a uniform gray fracture was fabricated, testifying to the fullness of the occurrence of SHS reactions of formation of TiC. Nano- and ultrafine particles of titanium carbide (40–550 nm) are distributed in small clusters throughout the body of the alloy. (Note that in the absence of alloying, in the matrix of pure aluminum, large clusters of TiC particles along the grain boundaries of aluminum were observed [20].) X-ray phase analysis showed the presence of TiC and $CuAl_2$ phases and the absence of undesirable embrittling phases Al_3Ti and Al_4C_3 (figure 2).

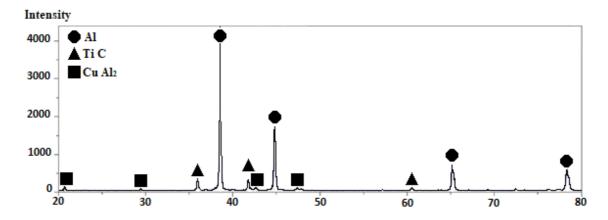


Figure 2. XRD pattern of (Al-5%Cu)/10%TiC sample.

Because the presence of 10% Na₂TiF₆ in the charge intensifies significantly the combustion process and raises the temperature of the system, that leads to a "sintering" the particles of titanium carbide and the formation of agglomerates, further experiments were carried out with the addition of a smaller amount of 5% Na₂TiF₆.

Manganese belongs to the group of transition metals, the cooling of the alloy Al-Mn is associated with precipitation of the second intermetallic phase MnAl₆, whose presence, along with CuAl₂ and

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TiC phases, also leads to the dispersion hardening. Figure 3 shows the microstructure of (Al-5%Cu-2%Mn)/10%TiC sample fabricated with addition of 5% Na₂TiF₆ to the SHS charge.

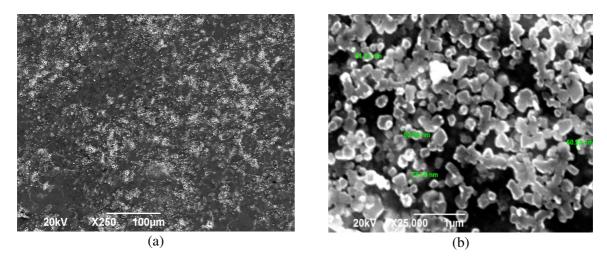


Figure 3. SEM image of (Al-5%Cu-2%Mn)/10%TiC sample.

The sample has the homogeneous gray fracture and uniform, without agglomerates, the distribution of nano- and ultrafine particles of titanium carbide (70-500 nm). X-ray analysis confirmed the presence of TiC and CuAl₂ phases (figure 4).

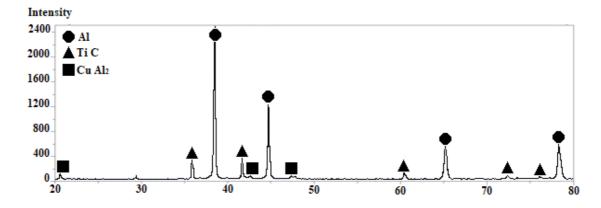


Figure 4. XRD pattern of (Al-5%Cu-2%Mn)/10%TiC sample.

Phase MnAl₆ was not detected due to small quantity. The mechanical characteristics of the alloyed composites is given in table 1.

Table1. Mechanical properties of aluminum and composites on its basis.

	Yield	Tensile	Ductility	Ductility	Hardness
	Strength	Strength	(%EL)	(%RA)	(HB)
	(MPa)	(MPa)			
Al (A7)	-	60	20	-	20
Al/10%TiC	82	110	12	23	35
(Al-5%Cu)/10%TiC	74	196	8	11	63
(Al-5%Cu-2%Mn)/10%TiC	114	208	6	7	97

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4. Conclusion

- 1) Introduction into the aluminum melt of alloying additive of 5%Cu and addition of 10%Na₂TiF₆ salt to the SHS charge (Ti+C) allows obtaining nano - and ultrafine particles of titanium carbide with a more uniform distribution throughout the body of Al-5%Cu matrix than in the case of unalloyed matrix. The structure of the cast composite (Al-5%Cu)/10%TiC is characterized by low TiC particle agglomeration along the grain boundaries.
- 2) Sequential introduction into Al melt of alloying additives of 5%Cu and 2%Mn, and addition of 5%Na₂TiF₆ to the SHS charge leads to the formation of nano- and ultrafine particles of TiC which are uniformly distributed over the body of (Al-5%Cu-2%Mn)/10%TiC composite.
- 3) The alloying of aluminum matrix composites Al/10%TiC with copper (5%) and manganese (2%) allowed us to achieve almost double strengthening while maintaining a sufficiently high level of ductility that confirms the prospects of using alloyed aluminum matrix composites of Al/TiC system as advanced engineering materials with improved mechanical properties.

Acknowledgements

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