Mechanical properties of bulk composites based on aluminum alloy reinforced with CNT with ex-situ TiC layer

A.V. Aborkin, D.M. Babin, A.I. Zalesnov, D.V. Bokaryov, A.I. Elkin
Vladimir State University, 87 Gorky str., Vladimir, 600000, Russia
aborkin@vlsu.ru

Abstract. The composite powders AlMg6 + 0.3 wt.% CNT and AlMg6 + 0.3 wt.% TiC/CNT were synthesized by the method of high-energy ball milling. Characterization of powders was carried out by methods of scanning electron microscopy, granulometric, and X-ray phase analysis. The powders were consolidated by hot pressing. The structural-phase composition and mechanical properties of bulk composites have been studied. The positive effect of the ex-situ TiC layer on the CNT surface on the mechanical properties of composites based on the AlMg6 aluminum alloy is shown.

1. Introduction
Composite materials based on aluminum and its alloys, meeting the growing requirements for high weight efficiency, have great potential for application in the automotive industry [1]. High strength and Young's modulus [2] give grounds to consider carbon nanotubes (CNTs) as one of the most promising types of strengthening additives for creating composite materials.

For the production of Al/CNT composites, powder metallurgy is widely used, in particular, high-energy ball milling (HEBM) [3,4]. In this case, the consolidation of composite powders to obtain bulk materials is carried out using various technological methods or their combinations [1, 5-12]. However, the achieved level of physical and mechanical properties of the obtained composites is much lower than expected [5]. To make full use of the properties of CNT, it is necessary to take into account many aspects related not only to the technology of synthesis of the composite material but also to the compatibility of the matrix and reinforcement [13]. The relationship between the matrix material and the relatively “inert” CNTs is critical to the formation of the properties of the composite. The matrix material must have good adhesion to the CNT surface so that the load can be transferred to the CNT and prevent the two contacting surfaces from sliding together [14]. Therefore, the task of forming a stable and durable interface is of particular importance.

Increasing the strength of composites based on aluminum and its alloys by improving the interphase load transfer is possible through the formation of an in-situ Al4C3 interface layer at the matrix-reinforcement interface [15-18]. However, the problem of this approach is associated with the difficulty of controlling the degree of reaction of the formation of the Al4C3 phase, as well as its tendency to hydrolysis [19].

Another option is the pretreatment of CNTs to create ex-situ ceramic nanoparticles or coatings on their surface [20, 21], which can be used as a boundary phase with a developed surface to improve the interaction with the matrix material [22].
This work is aimed at studying the effect of an ex-situ TiC layer on the CNT surface on the mechanical properties of composites based on the AlMg6 aluminum alloy.

2. Methods and equipment
For comparison, two types of carbon nanostructures were used as strengthening additives: multi-walled carbon nanotubes in the initial state after synthesis and multi-walled carbon nanotubes (CNT) coated with a titanium carbide layer (TiC/CNT). The AlMg6 aluminum matrix alloy was mixed with reinforcing additives by high energy ball milling (HEBM) using a planetary mill (PULVERISETTE 6, Fritsch, Germany). The mass fraction of reinforcing additives was 0.3 wt. %. For milling, steel balls with a diameter of 8 mm were used with a ratio of the mass of the loaded components to the mass of grinding bodies of 1:15. The treatment was carried out at a rotational speed of 600 rpm for 6 hours. The milled powders were subjected to uniaxial cold pressing on a hydraulic press to obtain briquettes with a diameter of 16 mm. The resulting briquettes were subjected to hot pressing on the same press in a special steel mold heated to 450 °C at a pressure of 400 MPa to obtain compacts with a diameter of 17 mm and a height of 12 mm.

The structure and properties of powder and bulk composites were characterized by scanning electron microscopy (ZEISS ULTRA PLUS), particle size distribution (MICROSIZER 201), and X-ray phase analysis (BRUKER D8 ADVANCE), as well as mechanical compression tests (TIME GROUP WDW-100E).

3. Results and discussion
Figure 1 shows typical SEM images of composite powders reinforced with CNT and TiC/CNT structures. Powder particles have a rounded shape with a developed surface. The average powder particle size determined by DLS, regardless of the type and mass fraction of the reinforcement, changes insignificantly and amounted to ~ 48.5 μm.

Fig. 1. SEM images of composite powders reinforced by CNT (a, c) and CNT with an ex-situ TiC layer (b, d).
The results of X-ray phase analysis of the synthesized composite powders and bulk composites are shown in Fig. 2. Also, Fig. 2 shows XRD curves of samples and matrix material processed along the same route. Comparative analysis of XRD results of composites and samples from matrix material shows the absence of the formation of new phases during mechanical processing in a planetary mill and subsequent consolidation. In this case, only the X-ray diffraction peaks of the matrix material were recorded on the X-ray diffraction patterns of composite powders. Also worth noting the decrease in the FWHM of the Bragg diffraction peaks of the matrix material. This indicates an increase in grain size during isothermal holding at consolidation. Isothermal holding at 450 °C leads to an increase in the crystallite size of the matrix material, which at least partially neutralizes the effect of severe plastic deformation during ball milling.

![Fig. 2. XRD of powder (a) and bulk (b) samples.](image)

In the course of compression tests, it was found that the obtained bulk composites have conditional yield stress significantly higher than that of specimens without strengthening additives (Fig. 3).

![Fig. 3. Deformation curves of bulk specimens in compression tests.](image)

In this case, the type of reinforcing additives does not significantly affect the conventional yield stress of composite materials, which is ~ 740 MPa and ~ 725 MPa. At the same time, the ultimate strength in compression of composites reinforced with TiC/CNT is ~ 60 MPa higher than for composites containing CNT. It should be remembered that the presence of a TiC layer on the CNT surface leads to a decrease in the volume fraction of strengthening particles since the density of TiC is more than two times higher than that of CNT. Thus, despite the equal weight fraction of the strengthening additives, the volume fraction in the case of reinforcing with CNT-hybrid structures will be somewhat less. This
also confirms the positive effect of the ex-situ TiC layer on the CNT surface on the mechanical properties of the composites.

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