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Microarticle

Fabrication of multi-walled carbon nanotubes–aluminum matrix composite by powder metallurgy technique

N.A. Bunakov [a,*], D.V. Kozlov [a], V.N. Golovanov [a], E.S. Klimov [b], E.E. Grebchuk [c], M.S. Efimov [a], B.B. Kostishko [a]

[a] *Ulyanovsk State University, Russia*
[b] *Ulyanovsk State Technical University, Russia*
[c] *ANO «Centre for Development of Nuclear Innovation Cluster of Dimitrovgrad, Ulyanovsk region», Russia*

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We report on fabrication of an aluminum matrix composite containing multi-walled carbon nanotubes (MWCNTs) produced by MOCVD method and functionalized via acid treatment by a H_2SO_4/HNO_3 mixture.

Specimens were prepared by spark plasma sintering (SPS) of the aluminum powder with different amounts of functionalized MWCNTs (FMWCNTs) in the range of 0.1–1 wt.%. We studied the effect of FMWCNTs amount on microstructure and mechanical properties of composites. It is shown that functionalization allows homogeneous dispersing of the MWCNTs in Al powder. The maximal increase in micro-hardness and tensile strength is registered at 0.1 wt.%.

Introduction

Outstanding mechanical properties of multi-walled carbon nanotubes make them a promising reinforcement component [1,2].

The tendency of the CNTs to agglomeration is among the main difficulties in fabrication of MWCNTs-metal matrix composites since highly homogeneous dispersing is required for high performance functionality.

Addressing this challenge, powder metallurgy is commonly used to produce homogeneously dispersed composite. Micron-scale metal particles mixed with MWCNTs are sintered to fabricate compact specimens employed for studies of structural properties.

Pristine MWCNTs obtained by CVD, MOCVD and other methods are usually employed in fabrication of metal matrix composites. Some papers report using functionalization of MWCNTs to attach the polar groups (–OH, –C=O, –COOH etc.), to the surface that allows obtaining a more stable dispersion in polar solvents enhancing the chemical compatibility with the metal matrix.

Pristine MWCNTs and functionalized MWCNTs (FMWCNTs) were used in our experiments. One should note that FMWCNTs provide a more homogeneous dispersing of the MWCNTs in the Al powder.

For this study, the nanotubes amount below 1 wt.% was chosen according to the best strength enhancement of metal matrices reported for these concentrations in Refs. [5–8]. We study the effect of FMWCNTs amount on microstructure and mechanical properties of composites.

Materials and methods

The aluminum powder with 99.9% purity and an average particle size of about 6 μm and pristine MWCNTs having 2% of amorphous carbon and graphite produced by MOCVD method (Metal Organic Chemical Vapor Deposition) [3] were employed in this study.

At the first stage, the process of functionalization was performed using a concentrated mixture of sulfuric and nitric acid (3:1 v/v) at 90 °C for 90 min [4].

The second stage is a separate ultrasonic processing of the FMWCNTs and Al powder in ethanol followed by a joint ultrasonic treatment and intensive stirring to provide a uniform FMWCNTs dispersion in the Al powder. The specimens were dried at 100 °C for 3 h. The following dried suspensions were prepared: the aluminum powders with 0 wt.% (test specimen), 0.1 wt.%, 0.25 wt.%, 0.5 wt.%, 1 wt.% FMWCNTs.

The third stage is the spark plasma sintering (SPS) of powders at 600 °C under 50 MPa applied stress for 20 min in a vacuum. Finally, cylindrical specimens, Ø20 × 10 mm and Ø10 × 20 mm in size, were fabricated.

Phase analysis of the initial components was carried out using diffractometer Bruker D2 PHASER.

* Corresponding author.

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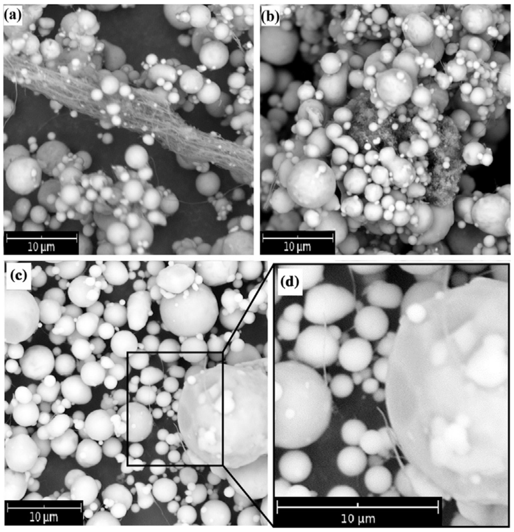

Fig. 1. SEM images of composites: (a, b) 0.5 wt.% pristine MWCNTs; (c, d) 0.5 wt.% FMWCNTs.

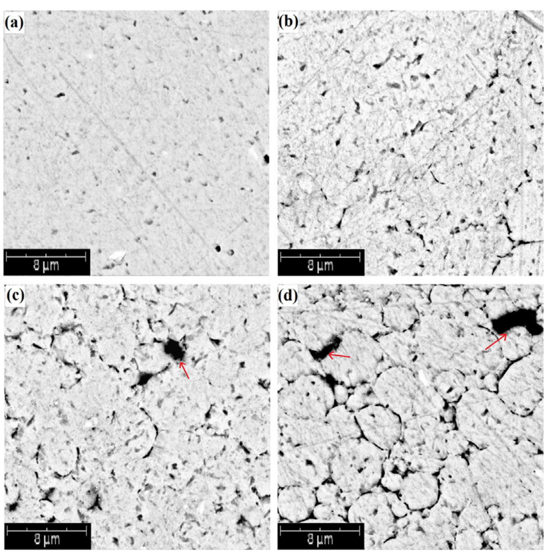

Fig. 2. Cross-sectional SEM images of specimens after SPS at 600 °C for 20 min: (a) Al/0 wt.% FMWCNTs (test specimen); (b) Al/0.1 wt.% FMWCNTs; (c) Al/0.5 wt.% FMWCNTs; (d) Al/1 wt.% FMWCNTs.

Structure of the FMWCNTs-aluminum matrix composite powders was studied using scanning electron microscope PHENOM Pro X.

Micro-hardness of the FMWCNTs-Al matrix composite bulks was measured by Vickers hardness tester (Qness Q10M).

The tensile tests were carried out using testing machine Walter +Bai LFM 125 kN.

Dispersion of FMWCNTs in the Al-powder

SEM images of the FMWCNTs dispersion in composites are demonstrated in Fig. 1. Fig. 1(a, b) shows large agglomerations of 20–50 µm in the specimen with the pristine MWCNTs. In the case of FMWCNTs, there are individual nanotubes of 4–8 µm uniformly dispersed within the Al powder (Fig. 1(c, d)). Smaller agglomerations of 2–4 µm are registered at 0.5 and 1 wt.% FMWCNTs (red arrows in Fig. 2(c, d)).

Al/FMWCNTs microstructure after SPS

As one can see in Fig. 2(a), there are small pores of 300–700 nm and no definite boundaries between the particles in the Al/0 wt.% FMWCNTs specimen. An increase in the FMWCNTs amount leads more distinct and longer boundaries between particles (Fig. 2(b, c, d)) indicating a significant contribution of nanotubes to the Al powder sintering process. The thickness of the boundaries is 100–400 nm.

Table 1
Mechanical properties of the SPSed FMWCNTs-Al matrix composites.

Specimen	Property			
	Micro-hardness (MPa) (±1–2 MPa)	Tensile strength (MPa)	Yield strength (MPa)	Strain (%)
Al + 0%FMWCNTs (test specimen)	43	126	97	24
Al + 0.1% FMWCNTs	50	163	101	13
Al + 0.25% FMWCNTs	47	151	85	6
Al + 0.5% FMWCNTs	45	146	100	11
Al + 1% FMWCNTs	46	132	87	4

Mechanical properties

Mechanical properties of the composites are listed in Table 1. A cross correlation is observed between micro-hardness and tensile strength values. Their maximal increase is registered for 0.1 wt.% FMWCNTs amount, at which micro-hardness and tensile strength demonstrate an increase of 16% and 30%, respectively, relative to Al/0% wt.% FMWCNTs specimen. Strain of specimens reduces with an increasing of FMWCNTs amount that is most likely due to enlargement in size and number of nanotube agglomerations. The specimen with the best tensile strength maintains high enough strain of about ~13%. Yield strength is within 85–101 MPa.

Conclusion

An effect of nanotubes amount on the SPS process of Al powder has been demonstrated. It is shown that addition of 0.1 wt.% FMWCNTs can lead to an increase of 16% and 30% of micro-hardness and tensile strength, respectively. Further SEM studies on composite microstructure and FMWCNTs/Al-matrix contact are expected. Also, additional treatments (e.g. hot extrusion etc.) are planned to study their contribution to enhancement of mechanical properties of composites.

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