



Novel aluminum-graphene and aluminum-graphite metallic composite materials: Synthesis and properties



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ARTICLE INFO

Article history:

Received 30 August 2015

Received in revised form

3 November 2015

Accepted 10 December 2015

Available online 13 December 2015

Keywords:

Graphene

Aluminum

Metal-matrix composites

Mechanical properties

Corrosion

ABSTRACT

A novel method of creating new lightweight, aluminum-metallic, composite materials under halides melt at temperatures 973–1073 K under air atmosphere is proposed. The method for synthesizing aluminum-based metallic composite materials, containing up to 2 wt. % graphene sheets uniformly distributed in a metal matrix, is entirely new, having no analogies in current science and practice. The synthesis of graphene nanosheets in a metal matrix is one-step, simultaneous process, taking place directly in molten aluminum under alkali halides melt without the necessity of a separate stage of synthesis and introduction of graphene. This has the potential to facilitate the inexpensive synthesis of aluminum-graphene composites with a high concentration of graphene.

The aluminum-graphene composites formed according to this method are characterized by a high uniformity of graphene films with linear dimensions from 100 nm to 50 μm and a thickness from one to three nm in the metal bulk.

No aluminum carbide forms under synthesis; the aluminum-graphene and aluminum-graphite composites are resistant to corrosion in NaCl solution.

The hardness, strength and ductility of aluminum-graphene composites are at least 2–3 times higher than the initial aluminum material, proportional to the concentration of graphene.

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1. Introduction

The production of novel aluminum alloys and composites with a decreased specific density and improved mechanical properties is one of the most significant goals of contemporary material science investigations. The creation of new aluminum metallic composites with a high content of nano- and microparticles of carbon in different allotrope modifications, or with ceramic nano- and microparticles such as aluminum oxide and silicon carbide, presents both a scientific challenge and an industrial necessity for numerous applications in automotive, aircraft or battery technologies.

Studies of interactions between aluminum and carbon have been carried out both by experimental method and computer simulation [1,2]. According to Schunk's data [3], the solubility of carbon in liquid aluminum is about 0.03 at.%. The carbon (graphite)

does not allow wetting by liquid aluminum at temperatures below 1773 K; this makes graphite the most usable materials for anodes in aluminum production. Consequently, the synthesis of aluminum composite materials with carbon concentrations above the equilibrium value presents a particularly difficult challenge.

Currently, the most commonly utilized processes of production of aluminum-carbon metallic composites are the sintering of aluminum into carbon fiber matrices [4], or the impregnation of carbon fibers with liquid aluminum [5]. The carbon fibers are typically covered by thin layers of silver, copper, nickel 0.4–1.0 μm thick for the enhancing of carbon fiber wetting [6]. Other existing methods include “mixing in the melt” of particles, wicks and short fibers of carbon (up to 50 wt. %) with aluminum melt under standard casting technology [7].

Due to a significant difference in the densities of carbon and aluminum, and the weak wettability of carbon by molten aluminum, it is difficult to obtain a homogeneous material; consequently, aluminum-carbon composite materials produced by traditional methods tend to have poor operational and technological characteristics. The fibrous, uniaxial composite materials are

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characterized by anisotropy of mechanical properties along and across the fibers, consequently exhibiting a low-resistance inter-layer shift and tendency to transversal breakage.

In addition, an increase of the process temperature up to 1373 K for wetting by aluminum of carbon particles and fibers improvement leads to a dissolution of the carbon with the formation of significant quantities of aluminum carbide. The aluminum carbide fully hydrolyzes in aqueous media with aluminum hydroxide and gaseous hydrocarbons such as methane or acetylene. This leads to a deterioration in the corrosion resistance of the composite material and is a common contributory factor in mechanical failure.

Metallic materials with degrees of reinforcement up to 50% can also be produced by means of powder metallurgy [8,9]. Typically, the degree of reinforcement does not exceed 25% because fracture toughness is reduced at further increases of carbon content. The aluminum-carbon composites produced by powder metallurgy with carbon content up to 25 wt. % are characterized by high porosity and poor sintering due to the aluminum oxide layer on the surface of the aluminum powder.

Some alternative, high-energy methods for the creation of aluminum-carbon metallic composites have been proposed in recent years. Thus, in the course of the implantation of carbon ions into an aluminum target [10–12], carbon embeds to a depth of 150 nm with formation of aluminum carbide. The accurate, prolonged ball milling of carbon nanoparticles or carbon nanotubes (CNT) with aluminum nanoparticles [13,14] – or the combination of different above-mentioned synthesis methods [15] – allows various aluminum-carbon composite materials to be obtained.

A method for synthesizing aluminum-carbon composite materials with weak mechanical anisotropy, low porosity and good sintering is described [16]. This method is based on the interaction of molten aluminum with graphite in K_2TiF_6 and/or cryolite melt media. Reinforcing titanium/aluminum carbide and titanium aluminide particles are formed at high-temperature interaction. The fact that the dispersion of carbon and titanium carbide particles is non-uniform greatly limits the applicability of this method.

A significant number of papers on synthesis methods and mechanical properties of aluminum-graphene composites have been published in recent years [17–23]. Typical synthesis processes as prolonged milling and annealing in an inert atmosphere have preliminary synthesized graphene or graphene oxide with powdered aluminum; this is also the case with application of CNT as a reinforcing additive.

Nearly all researchers have noted the graphene hardening effect on an aluminum matrix with aluminum carbide, which formation was confirmed by X-Ray investigations. The formation of a thin layer of aluminum carbide at the ND–Al interface may be beneficial to the overall performance of the metal-matrix composites as it improves the bonding strength between the reinforcement agent and the metal matrix [24].

In contrast, even small aluminum carbide additions are damaging to aluminum-graphene composite materials in aggressive environments – and even in conditions of atmospheric corrosion – because of non-uniform, pitting corrosion. This limits the practical use of thus obtained aluminum-graphene composite materials.

Thus, we can conclude that there are currently no reliable and productive methods of aluminum-carbon synthesis with high concentration and uniform distribution of carbon as graphite or graphene in aluminum matrix without the unwanted production of aluminum carbide. Even in impurity amounts, the presence of aluminum carbide negatively impacts on the corrosion-resistant properties of metallic composite materials.

The subject of this study is to obtain aluminum-graphene and aluminum-graphite metallic composites with carbon concentration

up to 2 wt. % having an improved structure and physical/chemical properties without aluminum carbide formation at temperatures under 1073 K by direct chemical interaction of carbon-containing substances “in situ” with molten aluminum under a layer of molten alkali halides in air.

2. Experimental

The interaction between the molten aluminum and the carbon-containing component was performed in molten alkali halide media using an alumina crucible. The aluminum sample with 0.65 wt. % impurities (Si – up to 0.62 wt. %; Fe, Cu, Mn, Mg – up to 0.05 wt. % in total; Zn – up to 0.1 wt. %) comprises a disc 3 cm in diameter and 1 cm in height. The aluminum sample size was equal to the inner crucible diameter.

Salt mixtures of alkali chlorides such as LiCl, NaCl, KCl, CsCl and $CaCl_2$ with melting points of less than 700°C were used as base electrolyte. The small fluoride additions of NaF or AlF_3 (less than 2 wt. %) formed part of the salt flux electrolyte. Metal or non-metal carbide powders such as WC, TiC, ZrC, Mo_2C , SiC or B_4C were used as the carbon-containing additive.

A pre-melted salt electrolyte was thoroughly mixed with a specific amount (not more than 0.2 to 6.5 wt. % of the total salt weight) of carbide powder that had an average grain size of 100–200 μm . The mixture of alkali-halide metals with the carbide addition was placed on the bottom of the crucible.

The aluminum was loaded into part of the salt mixture on the bottom of the crucible. The mixture of alkali-halide metals with the carbide addition was also placed on the top of the aluminum in order to eliminate the oxidation of molten aluminum through contact with air oxygen. The alumina crucible was covered with a cap and then placed into the vertical heating furnace. The temperature interval for the experiments was 973–1073 K. The interaction temperature was chosen under the melting point of both the aluminum (935 K) and the salt mixture. These temperatures are lower than that of aluminum carbide synthesis. The exposure time varied from 0.5 to 5 h. After the high temperature exposure, the liquid aluminum globule was poured into the cold alumina crucible with a controlled cooling period. After the salt mixture had fully solidified, it was dissolved and the salts were washed off the surface of the aluminum globule. The metal drop was weighed and its specific density defined according to its chemical composition and structure.

The chemical composition of aluminum carbon-containing composites was determined by chemical X-Ray fluorescent analysis using an XRF-1800 X-Ray fluorescent spectrometer by Shimadzu, Japan and CS-230 infrared analyzer of carbon and sulfur absorption by LECO (USA).

The aluminum-carbon composite was then divided into two halves. The first half was poured by current-carrying gum and then polished by six different silicon solutions using a Struers disc-finishing machine (Austria). The specimen was then examined as follows: scanning electron microscopy (SEM) using JEOL 5900LV and Zeiss Sigma VP microscopes; Raman spectroscopy, using a Renishaw-1000 Raman microscope-spectrometer with a green 514 nm argon laser; servo-hydraulic testing machine INSTRON 8801 and Neophot 32 optical microscope, micro hardness testing, using the system for micro hardness testing WIN-HCU FISCHER-SCOPE HM 2000 XYm which designed to measure Martens test under a load in accordance with standard ISO 14577. Elastic modulus was determined by unloading curves by standard Oliver - Pharr method. The Oliver-Pharr method [25] is the most frequently adopted method in instrumented indentation testing to probe the elastic modulus of materials. Electron backscatter diffraction (EBSD) patterns were obtained using HKL NORDLYSS

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