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Improved dispersion of carbon nanotubes in aluminum nanocomposites

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ABSTRACT

In this study, we investigated the influence of the dispersion technique of carbon nanotubes (CNT) in the production of aluminum matrix nanocomposites. Three production routes using different dispersion techniques were tested: in R1 route the CNT were dispersed using an ultrasonic bath; in R2 route the dispersion was achieved by ultrasonication, while in R3 route the dispersion and mixing were performed by ultrasonication CNT and Al powders. Nanocomposites with several CNT contents (0.25–2.0 wt.%) were produced by conventional powder metallurgy procedures. Microstructural characterization by scanning and transmission electron microscopies revealed that the best dispersion of the CNT is obtained using R3 route. Nanocomposites with 0.75 wt.% of CNT exhibit well dispersed and embedded nanotubes and the highest hardness and tensile strength. The observed 200% increase in the tensile strength attested the strengthening effect of the CNT and the efficiency of the new dispersion treatment (R3 route).

1. Introduction

Metal matrix composites (MMC) demonstrate a range of fascinating properties, in particular high mechanical properties, including high strength and specific stiffness, desirable coefficient of thermal expansion and good damping properties. Several investigations have been conducted into the development of metal matrix composites with different types of reinforcement. The growing concerns about energy consumption and environmental protection require the development of high performance structural alloys. Aluminum alloys are in a position to meet these demands because of their resistance to corrosion, their low density and their high specific strength [1].

Carbon nanotubes' (CNT) reinforced metal matrix composites have generated a great deal of interest in recent years [2–12], due to the unique mechanical and physical properties of CNT, such as high stiffness (970 GPa), high strength (63 GPa) and high thermal conductivity, which, combined with their low weight, make them an ideal reinforcement material [13,14].

The successful development of a production process that promotes a uniform dispersion of CNT in the matrix without CNT damage, with high CNT density and effective load transfer between CNT and aluminum matrix, is essential for obtaining the expected strengthening of nanocomposites. Several techniques have been suggested as adequate for production of these nanocomposites; sintering followed by deformation processes or hot deformation processes alone appear to be the most promising techniques [6–12].

Processing of CNT/Al using only conventional sintering is reported in few studies. Pérez-Bustamante et al. [6] investigated nanocomposites of Al with multi-walled nanotubes (MWCNT) produced by uniaxial pressing at 950 MPa followed by conventional sintering at 550 °C for 3 h. The CNT and Al powders were mixed by mechanical milling in a high-energy shaker mill at inert atmosphere. Transmission electron microscopy (TEM) images of CNT show some amorphization due to the milling process. The formation of Al_4C_3 carbide, usually reported in these nanocomposites, was not detected. The hardness of the nanocomposites was higher than pure Al.

To improve the reinforcement effect of the CNT in the metal matrix, some studies have been conducted which combine sintering and deformation processes [8,9]. Kuzumaki et al. [8] were the first to fabricate CNT/Al nanocomposites by adopting hot pressing and hot extrusion processes with 5 and 10 vol.% CNT. Due to the non-uniform dispersion of CNT, the tensile strength was similar to the bulk Al. George et al. [9] produced nanocomposites with MWCNT and single-walled nanotubes (SWCNT) by sintering at 580 °C for 45 min followed by hot extrusion at 560 °C. The CNT were dispersed in ethanol by sonication for 20 min and mixed with Al powders through ball milling at 200 rpm for 5 min. The results have shown a tensile strength of 134 and 141 MPa with 0.5% of MWCNT and SWCNT respectively. The strengthening mechanisms were not identified.

Other authors suggest a production carried out only by hot deformation processes. Esawi et al. [10,11] produced Al-2 wt.% CNT nanocomposites using rolling or extrusion processes and obtained a 100% or 50% increase in the tensile strength, respectively. The mixing was performed using high energy ball milling

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for 30 min at 400 rpm. The formation of carbide (Al_4C_3) was observed. The authors also observed structural damage in CNT due to the ball milling process. Kwon and Leparoux [12] obtained a higher strength for CNT/Al nanocomposites produced by mechanical ball milling followed by a direct powder hot extrusion process. The nanocomposites with 1 vol.% of CNT exhibited a tensile strength three times greater than that of pure Al. A small quantity of nanometric Al_4C_3 was detected. The increase in mechanical properties of the CNT reinforced nanocomposites are more effective when hot deformation processes such as extrusion, rolling or pressing are used.

Despite scientific and technological advances that have resulted from these studies, there are some challenges that need to be overcome in the production of these nanocomposites. The dispersion of CNT in the metal matrix is the biggest challenge. It is necessary to develop new dispersion techniques to obtain a better dispersion of the CNT, undamaged, and increase the amount of CNT embedded in the matrix. Most of the studies have used the ball milling process, which apparently permits a good dispersion amongst the metal powders but causes damage to the CNT. Recently, some studies have reported new methods for a better dispersion of CNT in the metal matrix [15,16]. For example, Jiang et al. [15] used a flake powder metallurgy to achieve a uniform distribution of CNT and Noguchi et al. [16] developed a new mixing method called nanoscale dispersion, which disperses the CNT with a metal powder using natural rubber as a mixing medium. The results have shown that these processes improve the dispersion while causing minor structural damage to the CNT, thus increasing the mechanical properties.

The increase in mechanical properties by carbon nanotube reinforcement is very promising, but the implementation of these composites on an industrial scale is only possible if they are produced in a simple and cost-effective way, if possible using conventional procedures. At this point it is possible to obtain high properties, but this always involves a combination of many processes, especially extrusion. Our objective is to obtain equivalent properties with powder metallurgical routes (pressing and sintering), allowing the production of more complex components. To achieve this, the present study concentrates on the development of a new dispersion technique of CNT in the metal matrix. The main objective is, while minimizing the structural damage, to obtain a good dispersion of the CNT in nanocomposites produced by conventional powder metallurgy routes.

The knowledge of the relationship between the microstructure and mechanical properties of these nanocomposites is a fundamental step towards understanding the strengthening mechanisms of CNT reinforced aluminum matrix nanocomposites. A strong CNT/Al interface is crucial to an effective load transfer, which according to some authors [12] is improved by the formation of Al_4C_3 carbide. In this study, the microstructure was characterized by electron microscopy techniques and mechanical properties were evaluated by tensile and hardness tests in order to validate the dispersion process of the CNT in the metal matrix and correlate the mechanical properties and microstructural features.

2. Experimental procedure

CNT used in this work (from Fibermax Composites) are mostly multi-walled nanotubes, MWCNT. Aluminum powders utilized (from Goodfellow) present a maximum particle size of 22 ± 11 µm and a purity of 99.5%. The characterization of these materials was performed by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and high resolution transmission electron microscopy (HRTEM). A high resolution FEI QUANTA 400 FEG SEM and a JEOL 2010F were used for this purpose.

Three production routes were used in our research. The schematic flowchart of the different stages of CNT/Al nanocomposites production can be seen in Fig. 1. In the first one (the R1 route), the CNT were dispersed in ethanol or isopropanol using an ultrasonic bath (Bandelin, Sonorex RX106, 35 kHz) for a period of 15–90 min. SEM and TEM observations investigated the evaluation of the most suitable conditions for obtaining untangled and dispersed CNT. The CNT were mixed with Al powders in a Turbula for 60–600 min. The nanocomposites were produced with several amounts of CNT: 0, 0.25, 0.50, 0.75, 1.0 and 2.0 wt.%. The materials were uniaxially pressed with 100–300 MPa and sintered at 500–640 °C for 30–90 min in a vertical furnace under a vacuum of 10^{-2} Pa.

To improve the dispersion, a modification of the procedure was investigated. In this route (R2) only the dispersion technique was modified; the CNT were dispersed in isopropanol by ultrasonication (IKA T25D ultrasonicator) for 15–60 min at 20,400 kHz.

A third route (R3) was also investigated. In this case, the dispersion and mixing were performed in just one step. The CNT and Al powders were ultrasonicated in isopropanol for 15–60 min at 20,400 kHz. The morphology and distribution of the CNT in Al powders were characterized by SEM.

To evaluate whether the production technique causes damage and/or defects in the CNT, Raman spectroscopy experiments were conducted with a wavelength of 532 nm. Microstructural characterization of CNT/Al nanocomposites was performed by SEM, electron backscattered diffraction (EBSD) and HRTEM. Cross-sections of the nanocomposites were prepared using standard metallographic techniques. An electron transparent cross-section of nano-

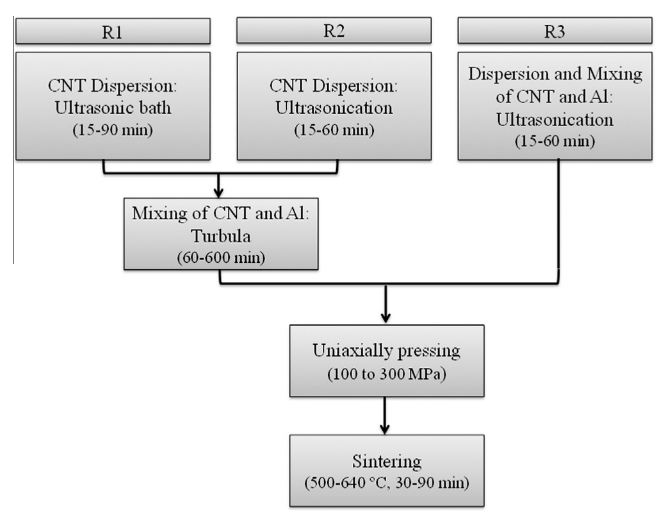

Fig. 1. Schematic flowchart of the different stages of CNT/Al nanocomposites production.

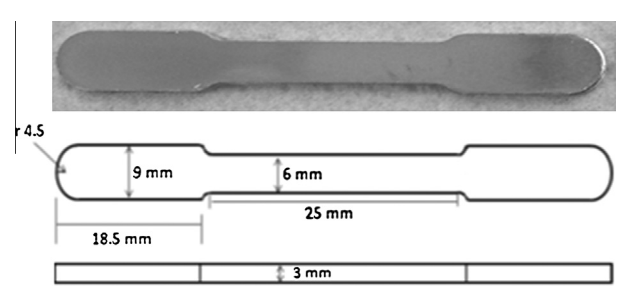

Fig. 2. Tensile specimen and a schematic showing the geometry and the dimensions of the tensile test specimen.