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Fabrication and consolidation behavior of Al 6061 nanocomposite powders reinforced by multi-walled carbon nanotubes

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ABSTRACT

The composite behavior of an Al 6061 nanocrystalline matrix reinforced with different weight percentages (0, 0.5, 1.0, 1.5 and 2.0 wt.%) of multi-walled carbon nanotubes (MWCNTs) produced by mechanical alloying (MA) with a milling time of 30 h was investigated. MWCNTs were added during the last 2 h of milling to avoid structural damage during MA. The milled powders were consolidated by cold uni-axial compaction followed by sintering at different temperatures (450 °C, 525 °C and 600 °C) under a reducing atmosphere (N_2) to evaluate the materials' sinterability. The structure of the MWCNTs was evaluated by Raman spectroscopy of as-received pure MWCNTs, Al 6061-2 wt.% MWCNT nanocomposite powder after 30 h MA and sintered samples of Al 6061-1 wt.% MWCNTs & Al 6061-2 wt.% MWCNTs. The crystallite size and lattice parameter of the composites were examined. The relative density, compressibility, green compressive strength, sinterability and Vickers hardness of the composites were also examined. The effects of MWCNTs as reinforcement on the consolidation behavior of the nanocrystalline matrix were studied using the Heckel, Panelli and Ambrosio Filho and Ge equations. The fabricated composites exhibited high Vickers hardness of 76 HV (818 MPa), which is approximately three times higher than that of microcrystalline Al 6061. However, because of MWCNTs that were added during the last 2 h of milling, higher hardness values were obtained in this investigation.

1. Introduction

Recently, in the development of metal matrix composites (MMCs), the size of reinforcements was decreased from the macro- to the nano-scale. Aluminum-based MMCs with small amounts of nanometer-sized reinforcements have attracted considerable research interest in recent years due to the potential for the development of novel composites with unique mechanical and physical properties [1]. Carbon nanotubes (CNTs) are nanoscale reinforcement structures that exhibit a large aspect ratio and good mechanical, electrical and thermal properties [2–4]. Several methods have been proposed and implemented to synthesize nanocrystalline matrix reinforced with multi-walled carbon nanotubes (MWCNTs), such as thermal spraying [5,6], powder metallurgy [7–9], mechanical alloying (MA) [10–15], semi-solid powder processing [16,17], spark plasma sintering [18,19], friction stir processing [20,21], flake powder metallurgy [22], spark plasma extrusion [23] and nano-scale dispersion [24]. However, the use of MWCNTs as reinforcement in Al MMCs is a great challenge due to their agglomeration and poor dispersion. Among the aforementioned methods, MA appears to be best for inducing the dispersion of nanoparticles within metal matrices. A number of research groups have investigated the use of ball milling for the production of Al-MWCNT nanocomposites [8–15]. However, only limited work is related to the densification that occurs upon the MA of Al 6061-MWCNT nanocomposites.

The present work investigated the synthesis, characterization, compressibility and sinterability of both Al 6061 alloy and a MWCNTs-reinforced Al 6061 nanocrystalline matrix through high-energy ball milling. Crystallite size, green compressive strength, compressibility and sinterability with respect to various weight percentages of MWCNTs were investigated and reported.

2. Experimental

2.1. Materials

The chemical composition required producing the Al 6061 nanocrystalline alloy powder used is shown in Table 1. Pure Al powder was used as the main matrix material, and other pure elemental powders, including silicon, iron, copper, manganese, magnesium, chromium, zinc and titanium, were used as solute materials with an average

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Table 1
Chemical composition to make the Al 6061 alloy powder.

Name of elements	Silicon, Si	Iron, Fe	Copper, Cu	Manganese, Mn	Magnesium, Mg	Chromium, Cr	Zinc, Zn	Titanium, Ti	Aluminum, Al
Element concentrations (gravimetric, wt.%)	0.600	0.700	0.275	0.150	1.000	0.195	0.250	0.150	Bal

All powders are more than 99% purity and mesh size of −325.

particle size of less than 45 μm (Alfa Aesar, 99% purity). Fig. 1(a) shows the morphology of as-received Al powder particles using secondary electron image of scanning electron micrograph (SEM). From Fig. 1(a), it can be observed that the Al matrix powder particles were irregular-flake like shape. The upper right inset of Fig. 1(a) shows the corresponding X-ray diffraction pattern which conformed the well crystalline nature of the powder. Further, the lower left inset of Fig. 1(a) shows the energy dispersive spectroscopy (EDS) of as-received Al matrix powder which conformed no impurities of as-received Al matrix. MWCNTs of 97% purity with an inner diameter of 20 nm, outer diameter of 40 nm and length of 50 μm (Redex Nano Lab, India) were used. The as-received morphology of MWCNTs was analyzed using field emission transmission electron microscope (FETEM). From Fig. 1(b) of bright field image, it was conformed the above mentioned specification of as-received MWCNTs. Toluene supplied by Ranbaxy, India, was used as a process control agent (PCA).

2.2. Blending of microcrystalline powder and synthesis of nanocrystalline powder

The elemental powders required to produce a microcrystalline Al 6061 matrix were blended in a two-station (Insmart Systems, Hyderabad, India) planetary ball mill at 280 rpm for 2 h. Neither balls nor a PCA was used during the blending process. Powders of mechanically milled for 0 h were considered blended powders.

Nanostructured Al 6061 powder was first synthesized through 28 h of MA using all elemental powders (Table 1). The milling was performed with the following parameters: ball-to-powder ratio: 10:1 (weight); powder mass: 30 g; mass of balls: 301.5 g; ball diameter: 20 mm; number of balls: 9; ball and vial material: hardened stainless steel; plate speed: 100 rpm; vial speed: 280 rpm. Toluene was used as a PCA. Various weight percentages (0.5, 1.0, 1.5 and 2 wt.%) of MWCNTs were added at the end of the 28th h of milling. The constituent powders were milled for up to 30 h. The blended powders were designated as Al 6061 microcrystalline powder, and powders prepared by 30 h of MA without reinforcement were designated as Al 6061 nanocrystalline powder.

2.3. Powder consolidation and mechanical testing

Cylindrical specimens measuring 10 mm and 10 mm in height (1.73 g of powder) were mechanically pressed with a compaction pressure of 500 MPa using a double-action compaction die in a hydraulic press (Insmart Systems, Hyderabad, India) with a capacity of 40 tons to study the compaction characteristics with zinc stearate ($Zn(C_{18}H_{35}O_2)_2$) as a lubricant [25,26]. The green cylindrical specimens were degassed at 350 °C for 1 h and then sintered for 2 h at 450 °C, 525 °C and 600 °C under a reducing atmosphere (i.e. nitrogen, N_2). In order to examine and investigate the densification behavior of Al 6061-x wt.% MWCNTs (x = 0, 0.5, 1.0, 1.5 and 2.0 wt.%) nanocomposites with respect to temperature, the consolidated green compacts by cold uni-axial compaction were sintered over the temperature range of 450–600 °C with a step size of 75 °C. The densities of the green and sintered cylindrical specimens were estimated using Archimedes's principle. The results were averaged over three independent measurements. Green mechanical strength or crushing strength was estimated from green cylindrical samples measuring 10 mm in diameter and 10 mm in length by using a cold uni-axial tester. The hardness of the green and sintered cylindrical specimens was measured by the Vickers microhardness method using a 500-g load on polished samples. Green mechanical strength is one of the most important properties of green pressed bodies in order to evaluate the mechanical and thermal stability as the green compacts go through the pre-firing and sintering stages. All tests were repeated at least five times, and the average values were reported.

2.4. Powder characterization

X-ray diffraction (XRD) analysis of the powders was performed with a D/Max Ultima III XRD machine (Rigaku Corporation, Japan). CuKα radiation (λ = 1.5406 Å) at a scanning speed of 2° per minute operating

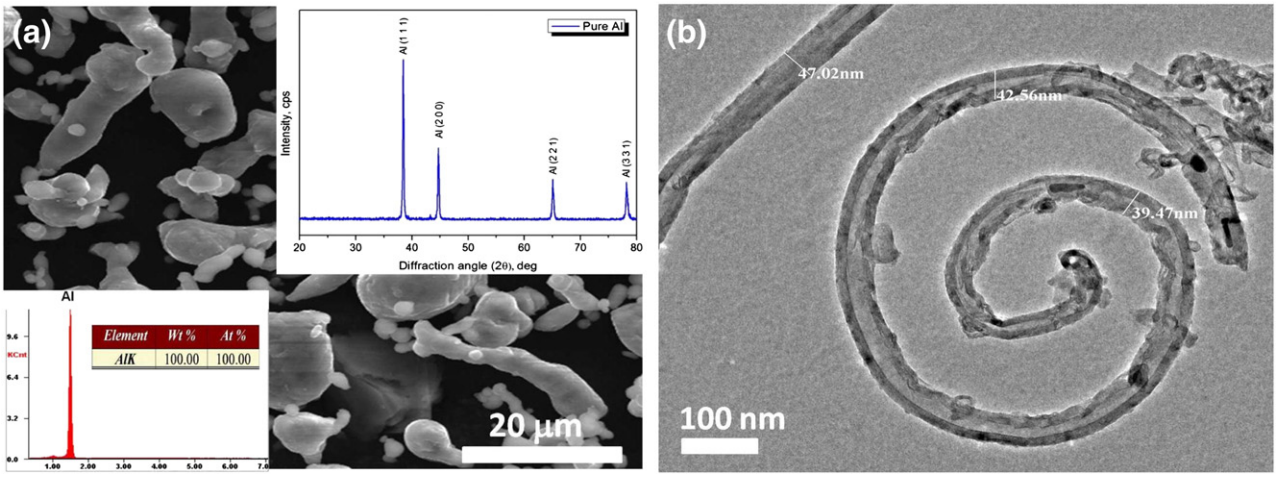

Fig. 1. (a) The SEM morphology of as-received Al matrix powder (Upper right inset shows corresponding X-ray diffraction, lower left inset shows corresponding EDS); (b) The bright field image of FETEM morphology of as-received MWCNT powders.