



Original Research Paper

Effect of coarse grain content on microstructure, cold workability and strain hardening behavior of trimodal AA 6061 nanocomposites reinforced with multi-walled carbon nanotubes

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ABSTRACT

Trimodal nanocomposites of AA 6061 nanocrystalline (NC) matrix reinforced with 2 weight (wt.%) multi-walled carbon nanotubes (MWCNTs) blended with different wt.% (0, 5, 10, 15, 20 and 25) of coarse grain (CG) elemental powders related to AA 6061 alloy were successfully produced by 30 h of mechanical alloying (MA). MWCNTs were added at the end of 28th h of MA to avoid structural damage of MWCNTs. The study of improvement of compressive ductility while maintaining high strength and toughness for NC materials was carried out by incremental cold upsetting of bulk trimodal composite. The 25% CG trimodal composite was observed to be the good one as it exhibited a better strain hardening behavior and high percentage of cold workability while maintaining considerable strength.

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1. Introduction

Major interest in composite materials started in the middle of the last century, mainly to meet the demanded materials by the automotive and aeronautic industries where light weight, good strength, corrosion resistive and wear resistive components are desired [1,2]. Since then, important progress in the development of composite materials has been achieved. In the field of composite materials, aluminum-based composites is an important area due to their low density and good workability, such properties are attractive for diverse industrial applications. Thus, in turn nanostructured aluminum matrix composites (AMCs) have gained considerable interest due to their excellent properties (i.e., high strength, low density, good corrosion resistance), and their technical and economical ease of manufacturing [3]. Extensive research has been focused on the production of AMCs. AMCs reinforced with multi-wall carbon nanotubes (MWCNTs) have been particularly attractive due to their superior mechanical and physical properties for aerospace, automotive, defence and structural applications [4–6].

MWCNTs exhibit relatively low density of 1.8 g/cm³ [7,8], and subsequently have high strength of 63 GPa, high stiffness of

970 GPa [9], high Young's modulus of the order of 1.8 TPa and thermal conductivity up to 3000 W m⁻¹ K⁻¹ [10]. These excellent mechanical properties, concomitant with their chemical stability, suggest that MWCNTs might be suitable as a novel reinforcement material for AMCs. Hence, these unique mechanical properties of MWCNTs make them promising reinforcements for synthesizing light weight, high strength metal matrix structural composites.

Composites have been prepared with metal/alloy matrices of Al [11–13], Cu [14–17], Ni [18,19], Mg [20,21] and metallic glasses [22] reinforced with MWCNTs for the purpose of light weight, high-strength structural materials. Several fabrication routes namely powder metallurgy (P/M), ball milling, extrusion, hot pressing, equal-channel angular pressing, spark plasma sintering, electro-deposition, electro-less deposition and thermal spraying have been used to synthesize metal matrix composites with MWCNTs. Noguchi et al. [11] have reported a sevenfold increase in compressive yield strength in 1.6 vol.% CNT reinforced Al composites prepared by a nano-scale dispersion method. Zhou and co-workers [12] have shown a 30% decrease in the wear rate and friction coefficient of Al-CNT alloys by addition of 20 vol.% of CNTs. The previous studies indicate that there is lot of scope for strengthening and enhancement of properties of AMCs by addition of MWCNTs. Furthermore, the use of mechanical milling to produce CNT reinforcing AMCs is still very limited [23–25]. In this regard, the work described herein deals with the production of novel Al alloy based nanocomposites by mechanical milling. Synthesis,

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consolidation behavior and mechanical properties of AA 6061-MWCNTs nanocomposites have been investigated in our previous study [26].

Mechanical alloying (MA) is a novel technique that produces bulk ultra-fine/nanostructure materials in large quantities. A strong metallurgical bond between reinforcement particles and matrix particles can be obtained via MA. MA is a solid-state powder processing technique involving repeated welding, fracturing, and rewelding of powder particles in a high-energy ball mill [27]. One of the greatest advantages of MA is in the synthesis of novel alloys that are not possible by any other technique, such as alloying of normally immiscible elements. Many parameters influence the stages of milling, such as the BPR (ball-to-powder ratio), speed, milling atmosphere, temperature, process control agent, particle size, volume fraction and type of reinforcement [27]. The decreasing of particle size with milling time was studied by Samal et al. [28] during production of Al–Cu alloy by MA (0, 10, 25, 35 and 50 h). It was found that the average particle size has been reduced from 21 μm to 3 μm after 50 h of milling.

Various authors have studied the mechanical behavior of nanostructure materials in terms of stress–strain curves through uniaxial tensile and simple compression tests [29–31]. In fact, the uniaxial tensile test would not sustain a uniform tensile deformation at ambient temperature for more than a couple of percent of plastic strain, especially in refined grain materials. Hence, compression tests (like the cold-upsetting, here) are needed to provide a direct evaluation of the deformation behavior as the function of true effective strain because the compressive behavior is not strongly influenced by superfluous factors such as surface or internal blemish [32].

Workability is a convoluted technological concept that depends not only on the material but also the various process parameters such as stress, strain rate, temperature and friction. Workability criterion of P/M compacts was discussed by Abdel-Rahman and El-Sheikh [33]. They investigated that the effect of relative density on the forming limit of P/M compacts in upsetting. The cold workability and the strain hardening behavior of porous P/M composites under uniaxial, plane and triaxial conditions have been elaborately analyzed and discussed in the previous works by Narayanasamy et al. [34–40]. Their results have shown that the workability behavior of metals/alloys/composites of P/M components depends on the relative density, the aspect ratio, the preform geometry, the particle size and the percentage of reinforcement for the composites, the die geometry, the lubricants, and the compacting load.

Through the present work, the science and technology is being developed for the society as different nanocomposite materials with different condition has been used, developed and investigated. This will be very useful for the scientific community. In order to obtain light weight structures, the low elastic modulus of AMCs, compared to other MMCs is the most important problem to overcome in the substitution of AMCs. Thus, many researchers have attempted to improve the strength of AMCs by reinforcing CNTs [41].

Nanocrystalline materials generally possess insufficient ductility and a reduced toughness compared to their coarse-grained materials. Telkamp et al. [42] and Legros et al. [43] observed that incorporation of coarse grains improved the ductility of nanocrystalline Al 5053 and Cu respectively. The presence of coarse grains within the nanocrystalline matrix may enhance the ductility of nanocrystalline materials [44–46]. The introduction of coarse-grained Al into the nanocrystalline Al, have been evaluated to improve the ductility for the nanocrystalline metals [47–50].

The effect of coarse grain matrix phase in AA 6061-TiO₂ and AA 6061-TiC nanocomposites on microstructure, cold workability and strain hardening behavior have been investigated our previous

studies [51,52]. However, the effect of MWCNTs addition on AA 6061 alloy by mechanical alloying, the addition of CG matrix phase on AA 6061-MWCNTs nanocomposite and its deformation ability were not carried out so far. Further, there is no work related to correlating coarse grain content on AA 6061-MWCNTs nanocomposites.

Therefore, the main aim of the present research work is to study and investigate the effect of coarse grain (CG) content in AA 6061-MWCNTs nanocomposites structure on cold workability and strain hardening behavior at room temperature.

2. Experimental procedure

As-received pure aluminum powder with an average particle size of 40 μm was used as the major matrix material, and other pure elemental powders, including titanium, zinc, magnesium, manganese, iron, chromium, silicon and copper were used as solute materials with an average particle size of less than 45 μm (99% purity). All powders were supplied by Alfa Aesar, USA. The selected reinforcement of 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. Toluene, C₆H₅CH₃, (sulfur-free) supplied by Ranbaxy, India was used as a process control agent (PCA). The MA was carried out up to 30 h using stainless steel medium in toluene with ball-to-powder ratio (BPR) of 10:1 at 280 rpm. MWCNTs were added during the last 2 h of milling to avoid structural damage during MA. The MA operation yields agglomerates of NC-Al grains that contain a uniform dispersion of solidly bonded, sub-micron MWCNTs particles. In our previous work [26] the details about the synthesis and characterization of the produced nanocomposites were investigated.

The MWCNTs/NC-Al agglomerates were mechanically blended with 0, 5, 10, 15, 20 and 25 wt.% CG elemental powders related to AA 6061 alloy matrix in the same planetary ball mill with BPR of 1:1 at 120 rpm for 2 h. The trimodal composite powders were dried and stress recovered at 343 K under N₂ atmosphere. The milled powders were consolidated into cylindrical pellets of 30 mm diameter with aspect ratio of 0.375 as applicable to powder metallurgy industries using a double action uni-axial compaction dies in a hydraulic press (Insmart systems, Hyderabad, India) with a capacity of 40 tons at a compaction pressure of 500 MPa. The green compacts were degassed and then sintered at 798 K for 6 h under N₂ atmosphere. The sintered preforms were tested under non-heat treated condition. Zinc-stearate was used to lubricate the punches and die wall before compaction to reduce the die wall frictional effects. Fabricated trimodal AA 6061-MWCNTs nanocomposites powders were characterized extensively with optical microscopy (OM). Phase identification of the as-milled trimodal composites were characterized by XRD using Cu K α radiation (1.5406 Å) in a D/MAX ULTIMA III diffractometer (Rigaku Corporation, Japan) operating at 40 kV and 30 mA. The OM was used to investigate the hierarchal microstructure (e.g., the size and distribution of CG-Al domains and NC-Al/MWCNTs agglomerates).

The resulting post-sintered trimodal composites were subjected to incremental compressive load (cold-upsetting) of 5 kN between two surface ground flat open dies on a hydraulic press (100 tons capacity). The initial diameter (D_0), height (h_0) and density (ρ_0) were measured and recorded. The deformation was carried out until the appearance of the first visible crack on the free surface. The dimensional changes in the specimen such as height after deformation (h_f), top contact diameter (D_{TC}), bottom contact diameter (D_{BC}), bulged diameter (D_B) and density of the preform (ρ_f) were measured after every interval of loading. At least five readings were obtained and the average was used for investigation. The density (average of five readings) of cold-upset preforms after

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