Characterization of ball-milled carbon nanotube dispersed aluminum mixed powders

M A Maleque, U Abdullah, I Yaacob and Y Ali
Department of Manufacturing and Materials Engineering-Kulliyyah of Engineering, International Islamic University Malaysia, Kuala Lumpur- Malaysia
E-mail: maleque@iium.edu.my

Abstract. Currently, carbon nanotube (CNT) is attracting much interest as fibrous materials for reinforcing aluminum matrix composites due to unique properties, such as high strength, elastic modulus, flexibility and high aspect ratios. However, the quality of the dispersion is the major concerning factor which determines the homogeneity of the enhanced mechanical and tribological properties of the composite. This work study and characterized carbon nanotube dispersion in ballmilled CNT-aluminum mixed powders with four different formulations such as 1, 1.5, 2 and 2.5 wt% CNT under high energy planetary ball milling operations. The ball milling was performed for two hours at constant milling speed of 250 rpm under controlled atmosphere. The characterization is performed using FESEM and EDX analyzer for mapping, elemental and line analysis. The experimental results showed homogeneous dispersion of CNTs in aluminum matrix. The composite mixture showed similar pattern from mapping, elemental and line analysis. Identification of only two peaks proved that controlled atmosphere during milling prevented the formation of inter metallic compounds such as aluminum carbide in the composite mixture. Therefore, this CNT-Al composite powder mixture can be used for new nano-composite development without any agglomeration problem.

1. Introduction
Al matrix composites have wide prospects of application in aviation, spaceflight and automobile industries because of lower density (a requirement necessary for the weight reduction for many components thereby saving fuels and hence energy). Research in the field of carbon was revolutionized by the discovery of carbon nanotubes (CNTs) by Iijima in 1991 [1]. Although CNTs might have been synthesised in 1960 by Bacon, it took the genius of Iijima to realise that they are tubes made by rolling a graphene sheet onto itself. A multiwalled carbon nanotube (MWCNT) is made up of many single walled carbon nanotubes (SWCNT) arranged in a concentric manner. Experiments and simulations showed that CNTs have extraordinary mechanical properties over carbon fibers, e.g. stiffness up to 1000 GPa, strength of the order of 100 GPa [2], and thermal conductivity of up to 6000 W mK. Carbon nanotube reinforced metal matrix composites (CNT-MMC) are prepared using different type of processing techniques. Powder metallurgy (PM) is the most popular and widely used route by the researchers to synthesize CNT-MMC materials. Carbon nanotube reinforced aluminium (CNT-Al) composites are mainly produced using powder metallurgy method. Ball milling is the easier and cheaper means of synthesisation of composite materials using powder metallurgy route. Nano composite materials are multiphase materials obtained through the artificial combination of different materials either ceramic, polymeric and metallic matrices with carbon nanotubes to attain properties that the individual components by themselves cannot attain. It is believed that graphitic type of carbon

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fibre enhances the thermal conductivity, mechanical and tribological property of the material significantly.

However, successful dispersion of CNTs in the metallic matrices is of paramount important before achieving any sort of significant benefits in terms of the mentioned property of the composite [3]. Some work covered ball milling of a mixture of CNT and aluminum powder to break down the CNT clusters and the authors limited the milling speed to 200 rpm and the milling time to 5 min. Morsi and Esawi [4, 5], investigated the effect of CNT wt% (2 and 5 wt%) and the influence of milling time and process control agents on the size and morphology of the mechanically alloyed powder. The results show that particle size and morphology vary with milling time and CNT content and also the addition of methanol as process control agents can aid in controlling the powder characteristics. This paper studied and analyzed carbon nanotube reinforced aluminum nano-composite prepared using powder metallurgy route under high energy planetary ball milling operations. The CNTs dispersion in aluminum matrix were analyzed by energy dispersion spectroscopy (EDX) using energy dispersive X-ray analyzer.

2. Materials and method

Pure Al (99.7%), with particle size of 78 μm which has nearly spherical shape with some satellite subparticles was used as a matrix material. The image of the aluminium powder obtained using scanning electron microscopy (SEM) is showed in Fig.1a. The multi walled carbon nano tubes (MWCNTs) with a nominal diameter of 10 nm, length of 5-15 μm, and surface area of 40-300 m2g-1 was used as a reinforcement. The image of the MWCNTs obtained through field emission scanning electron microscopy (FESEM) is shown in Fig. 1b. Three compositions such as 1.5, 2 and 2.5 wt% CNT with the balance in each case being Al were studied in this investigation. Each composition was place in a tube together with a stainless steel ball of 10 mm diameter to make the ratio of ball-to-powder ratio of 5:1 for preliminary mixing via manual shaking for about 10-15 minutes. The preliminary mixture was then placed in 250 mL stainless steel mixing jars containing stainless steel milling balls of 10 mm diameter, the initial ball-to-powder weight ratio (BPR) is 10:1. The jar was filled with argon gas and the ball mill operations started using planetary mill (FRITSCH pulverisette 5 05.5000/00409) at constant speed of 250 rpm for tree different milling time of 1, 2 and 3hours. The CNTs dispersion were analysed by mapping, elemental and line analysis using EDX analyzer.

![Figure 1. Image of the as-received raw materials: (a) aluminum powder and (b) CNT](image)

3. Result and Discussion

The investigation on the uniform and homogeneous dispersion of CNT into the Al matrix was conducted by elemental mapping using FESEM-EDX analyser. Fig. 2 presented mapping results of 1 and 1.5 wt% CNT and Fig. 3 for 2 and 2.5 wt% CNT in CNT-Al mixed powders. The data obtained from mapping result followed the same trend for all the CNT compositions and showed the presence of CNT over the entire surface of Al particles. The scanned image was mapped to study the possible elemental composition of the scanned portion.
Figure 2. Mapping and elemental analysis of: (a) 1 wt% and (b) 1.5 wt% CNT-Al mixed powders.

Ball milling parameters: 250 rpm speed and 2 hrs milling time

From the mapping results, two elements were observed such as aluminium (Al) and CNT but CNT is represented by the symbol (C) and the map proved that there is no formation of compound such as carbides, due to the fact that only two elements were observed and the reinforcement material CNT were well dispersed in the aluminium matrix. Two different maps were shown with two different colours for easy identification of the found materials which was indicated at the extreme right of each map. The analysis graph was within the energy range of 0-20 keV.
Figure 3. Mapping and elemental analysis of: (a) 2 wt% and (b) 2.5 wt% CNT-Al mixed powders.
Ball milling parameters: 250 rpm speed and 2 hrs milling time

Line analysis of the CNT-Al mixed powders is presented in Fig. 4 and showed the effective dispersion of CNTs into the Al matrix during milling operation of mixed and blended CNT-Al powder at a speed of 250 rpm for 2 hrs of milling. The line analysis results were observed at the line intensity of between 0-45 and the distance within the range of 0-1.33 mm.
For all CNT compositions, the result showed similar trend as was obtained from mapping and line analysis also indicated almost similar quality of dispersion of CNT in the aluminum matrix. The line analysis of the mixed CNT and Al powders showed that 2 hrs milling time was sufficient to disperse CNT into the Al matrix at 250 rpm milling speed. This indicates the mutual coexistence of CNT and Al which has been shown in two different colours such as blue and green for aluminum and carbon respectively (Fig. 4). The lines indicated that Al and CNT go along and followed exactly the same trends which indicated the uniform distribution of CNT in the aluminum matrix for all the composition. It can be said that the data obtained from mapping, elemental and line analysis plots for...
all the CNT compositions indicated that CNT is uniformly and homogeneously dispersed in aluminum matrix without any formation of the significant carbides due to controlled atmosphere of milling. However, the involvement of argon gas during milling operation have eliminated unwanted reactions that are likely to occur during the process, and the use of suitable milling speed and time also help in preventing the contamination. The use of argon gas during milling operation was very vital as it control surface and interface contamination which are likely to occur during the synthesization of any composite mixture via high energy ball milling Esawi et al. [6].

4. Conclusion
The data obtained from mapping, elemental and line analysis plots for all CNT compositions indicated that CNT is uniformly and homogeneously dispersed in aluminium matrix without any formation of carbides due to controlled atmosphere of milling without formation of significant carbide. Therefore, this CNT-Al composite powder mixture can be used for new nano-composite development without any agglomeration problem.

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