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Development of a novel MWCNT reinforced iron matrix nanocomposite through powder metallurgy route

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ABSTRACT

Iron-Multiwalled Carbon Nanotubes (iron-MWCNT) were synthesized using varied durations of optimized high-energy ball milling (HEBM) of powder mix. Characterization and furthermore structural stability of these nanotubes were ascertained using state-of-the-art techniques such as Scanning and high resolution Transmission Electron Microscopy, X-ray Diffraction (XRD), X-ray Photoelectron Spectroscopy (XPS), Fourier Transform Infrared Spectroscopy (FTIR), Raman Spectroscopy, and Differential Scanning Calorimetry (DSC). Additionally, magnetic properties were measured by Vibrating Sample Magnetometer (VSM). Our results indicate that structural integrity and therefore characteristic properties of MWCNTs such as (magnetic, electrical and mechanical properties) are retained up to 60 min post ball milling. Expectedly, HEBM led to partial destruction of C—C bonds, however, the amount of carbon atoms released by bond disruption is insignificant and heavy super saturation of iron with carbon and extensive precipitation of carbides during heating is untenable. Optimal ball milling ensures the formation of smooth and continuous interfaces between the matrix therefore reinforcing the MWCNTs. The excellent interfacial structure so achieved has significantly improved the magnetic properties of iron-MWCNT nanocomposites synthesized in this study.

1. Introduction

In recent years, use of carbon nanotubes (CNTs) as reinforcing materials in development of functional nanocomposites has garnered widespread attention due to unique physical and mechanical properties of CNTs. Following success in development of useful polymer matrix-CNT composites, a great deal of research efforts has been exerted to harness the potential of CNTs as reinforcements in metal matrices [1–9]. Unlike polymer-CNT composites, integration of CNTs in a metal matrix is quite complex and therefore, fabrication and processing of metal matrix-CNT composites has been a matter of substantial concern. High aspect ratios and Van der Waals forces present in thin long CNTs are responsible for their susceptibility towards agglomeration; therefore, securing a uniform dispersion of CNTs within metal matrices appears to be a challenging task. Liquid metallurgy and stir casting routes have not been successful owing to poor wettability of reinforcements [10]. In liquid metallurgical processes, differences in densities between multiwall carbon nanotubes (MWCNTs) and metal matrix poses additional difficulties in achieving homogeneous dispersions of MWCNTs [10]. Mechanical alloying route, which is essentially a high-energy ball milling (HEBM) process, has been successful in achieving more or less uniform dispersions of CNTs in Al-matrix [5,7,11–14]. This has popularized HEBM as a preferred technique for synthesizing metal matrix-CNT composites [15,16].

It is well documented that MWCNTs undergo considerable damage during HEBM [2]. Extensive ball milling may lead to shortening and even amorphization of MWCNTs [9,17,18]. The defects created during milling of MWCNTs deteriorate properties, and therefore continue to limit its application until the evolution of structural degradation during the course of ball milling of MWCNTs is adequately characterized. Moreover, there are reports of accelerated damage of MWCNTs when milled with metal powders such as iron [19,20].

Desired properties of nanocomposites such as (mechanical property) are not attained when metal matrix nanocomposites are synthesized using mechanical alloying. A major factor which prohibits the attainment of such properties in metal matrix nanocomposites is ascribed to poor bonding between CNTs and matrix metal [4]. Theoretical studies with the aid of density function theory (DFT) have been conducted to demonstrate interfacial bonding characteristics of metal particles with CNTs [21,22] plausible solutions to overcome the challenges emanating from poor MWCNT-metal bonding is a subject of extensive research. Interestingly, transition metals with vacant 3d orbital (such as iron) may be expected to hybridize with p-orbital of CNTs and in turn, a superior interfacial bonding may be obtained [4]. Reports of studies on magnetic behavior of Fe-nanoparticles filled CNTs

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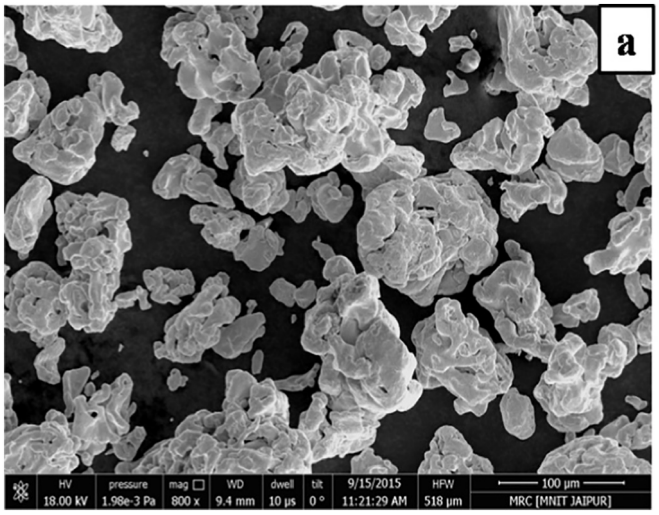

Fig. 1. FEG-SEM micrograph of Fe powder.

or iron nanoparticles layered MWCNTs are also available in literature [23–31]. However, there is no recorded attempt to disperse MWCNT in an iron matrix for augmenting its physical properties.

With the understanding that MWCNTs subjected to HEBM with metal powders are susceptible to structural damage, it may be surmised that a controlled damage of MWCNT, secured by optimal ball milling with iron may insure excellent interfacial bonding while embedding MWCNTs into metal matrices.

Therefore, the current body of work, attempts to probe into the feasibility of achieving superior interfacial bonding between reinforced CNTs and iron metal matrix by way of tailored damage of outer layers of MWCNTs, yet retaining its structural integrity; this insures harnessing the excellent potential of MWCNTs to enhance physical properties of nanocomposites. To this end, high energy ball milling of iron-MWCNT mixture was carried out for various milling times and the degree of damage in CNTs was monitored. Extensive characterization was carried out to optimize process parameters and enhance magnetic properties of the synthesized nanocomposites.

2. Experimental

Raw materials used for the present investigation consist of Fe-powder and multiwall carbon nanotubes (MWCNTs). Irregular shaped iron powder of size 325 mesh (purity > 99%) was procured from Sigma Aldrich Chemicals Pvt. Ltd. (Bangalore, India). MWCNTs were purchased from Nano Shell (3422 Old Capitol Suit 1305, Wilmington, DE 19808, United States). MWCNT (purity > 98%) with diameters ~ 10–20 nm and length ranges 3–8 μm were used for this study. The shape, size, and morphology of iron powder were observed using Field Emission Scanning Electron Microscopy (FESEM) and are shown in Fig. 1.

Transmission electron micrographs (TEM) of MWCNT used for the present investigation are shown in Fig. 2(a) & (b).

It is seen from Fig. 2(a) that the long thin and tubular CNT's are clustered and also bent at some places; the walls of MWCNT is clearly discerned in Fig. 2(b) and the total numbers of walls of MWCNT is found to be around 10–12.

High energy ball milling of a mixture of 30-gram iron powder and 2 wt% MWCNT has been carried out in a Planetary Ball Mill. Thus, in Fe-2 wt% MWCNT, 29.4-gram iron and 0.6 g MWCNT were taken in 250 ml tungsten carbide vial. The air inside the vial was flushed out by passing pure argon gas. Tungsten carbide balls (ball:powder = 6:1) were used for milling the mixture at a vial speed of 200 rpm for various periods of time. In order to avoid sticking and agglomeration of iron powder, 1 wt% stearic acid has been used as the process control agent (PCA). Samples milled under argon atmosphere were collected at regular intervals of milling viz. 10, 20, 30, 40, 60 & 90 min for characterizing the Fe-based MWCNT composites. Evolution of structure of nanocomposite with progress in milling time has been studied by field emission scanning electron microscope of model, NOVA NANOSEM 450, at an accelerating voltage of 15 kV. X-ray Diffraction study was carried out in PANALYTICAL P6 model and the study (Xpert-Pro Pan Analytical) was conducted within 2θ range of 20–100° for powder samples and at scan speed of 2°/min, step size 0.04° and counting time 30 s. The slit size used for the present experiments has been 10 mm × 15 mm and the penetration depth of X-ray was maintained at about 15 μm. The detection limit of minor phase is <1%. The XRD experiment has been carried out for long enough time to secure a high signal to noise ratio which has enabled to achieve a detection limit <1 wt% even for MWCNT of low atomic scattering factor. Microstructural characterization of the nanocomposites after different milling times has been carried out in high resolution transmission electron microscope (Tecnai $G^2$20 FEI S Twin). Powder samples, each of 1 mg was dispersed in absolute alcohol and then subjected to ultrasonication for 30 min; about 20 μl of ultrasonicated suspension was taken from the top layer and dropped on carbon coated grid. The sample became ready for HRTEM observation after complete evaporation of Ethanol.

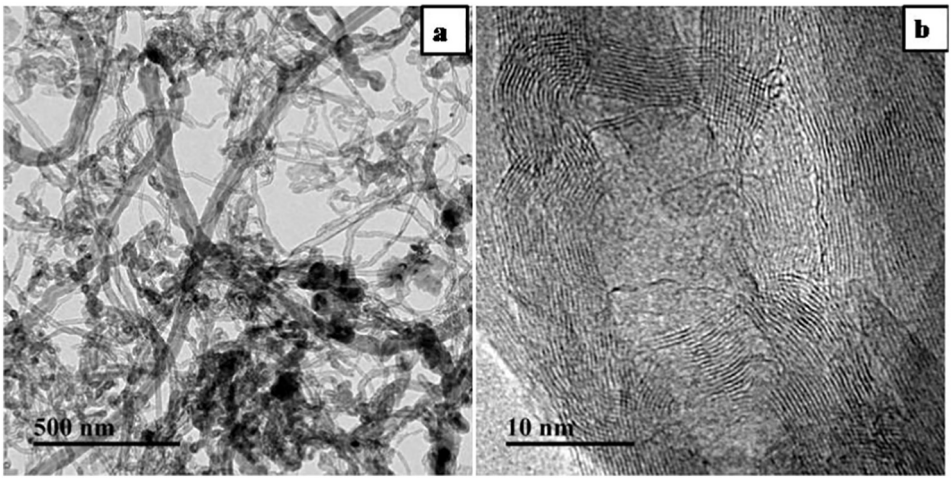

Fig. 2. (a): TEM image of MWCNT and (b) HRTEM image of MWCNT.