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Fabrication and Characterization of Magnesium Matrix Nanocomposites Reinforced with Multiwall Carbon Nanotubes

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Abstract

Multiwall Carbon Nanotubes (MWCNTs)-reinforced magnesium nanocomposites were fabricated through powder metallurgy route and then hot extruded as secondary process. CNTs were added by 0.5, 1 and 1.5 wt% with magnesium powders and ball milled to obtain a homogeneous mixture. The effect on mechanical properties of Mg nanocomposites due to the reinforcement of MWCNTs was investigated. The effect of mixing through high-energy ball milling, sintering temperature and extrusion process on the mechanical properties is explored. Mechanical property characterization reveals an improvement in 0.2% YTS, UTS, hardness with higher weight percentages of CNTs incorporated in the Mg matrix without affecting ductility.

Keywords: *magnesium matrix nanocomposites (MMNCs), multiwall carbon nanotubes (MWCNTs), high energy ball milling (HEBM), microwave sintering, hot extrusion*

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INTRODUCTION

Metal Matrix Composites (MMCs) are the better choice to get relatively superior mechanical properties, wear resistance, high elastic modulus and yield strength, and good damping properties compared to monolithic metals. Particulate-reinforced MMCs are considered as better than the fiber reinforced ones because of their lower fabrication cost [1, 2]. Magnesium-based MMCs are best suitable among other MMCs which are widely used in various applications in aerospace, automobiles, and sports equipment industries because of their low density and better mechanical properties [3].

Ceramic powder such as micron-size SiC and Al_2O_3 are commonly used as the reinforcement in Mg because of the low cost and easy availability. However, with the use of micron-size SiC and Al_2O_3, there is no difficulty in homogeneous disbursement of particles in the Mg matrix but the tensile strength and ductility of the composites are usually sacrificed relative to the monolithic Mg. The resultant lower strength or ductility is due to addition of micron-size ceramic reinforcement, which is due to the problems of particle fracture and particle/matrix interfacial failure. To overcome these problems and to look for further improvement in properties, nanosize reinforcements were tried through ball milling process and powder metallurgy process and the effects were investigated.

Carbon Nanotubes (CNTs) are the most exciting nanostructured materials of the twentieth century with superior mechanical, thermal and electrical properties discovered by IIjima [4]. CNTs are discovered to have Young's modulus and tensile strength of 3 TPa and 2 GPa respectively and density of 2.0 g/cm^3 [5–8]. Looking to these properties, CNTs could be an ideal reinforcement for magnesium and its alloys as a matrix material. Recent research producing Mg matrix composites reinforced with CNTs has been limited due to the problem of agglomeration of the CNTs due to Wander wall forces but was largely focused on polymer matrix composites [9–12]. But a few authors have tried with other reinforcements like Al_2O_3, S_iC and TiO_2 and found reasonable improvement in the mechanical properties [13–15].

The main objective of this research was to fabricate Mg-based nanocomposites reinforced with MWCNTs through powder metallurgy process. The nanocomposites were compacted initially with 0.5, 1, and 1.5 wt% of MWCNTs and sintered as primary process and then hot extruded as secondary process. These nanocomposites were characterized for the mechanical properties. The effect of increasing weight fraction, mixing medium, sintering temperature and extrusion parameters were correlated with the various properties characterized.

EXPERIMENTAL DETAILS

Magnesium (Mg) powder with 99.5% purity supplied by Neeraj Industries, Rohtak, Haryana, India, was used as the matrix material and multi-walled carbon nanotubes (MWCNT) produced by Nano Shell (USA), supplied by Intelligent Materials (P) Ltd, Chandigarh, as shown in Figures 1–3 with an average diameter of 20 to 50 nm were used as the reinforcement material.

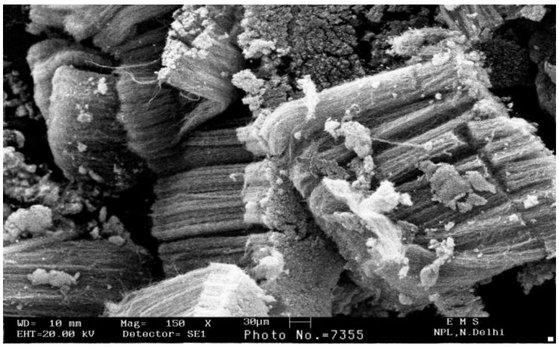

Fig. 1: *SEM Image of As-Produced MWCNT Aligned Bundles.*

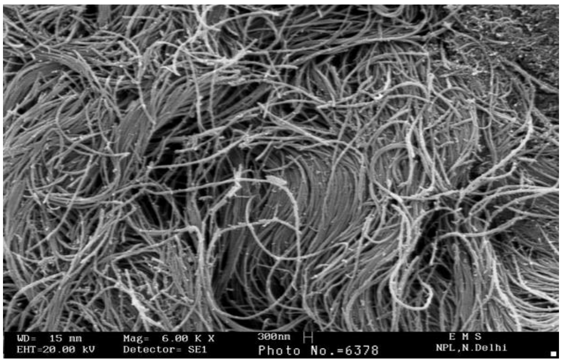

Fig. 2: *SEM Image of As-Produced MWCNT Long Length.*

Powder metallurgy route was used to synthesize both monolithic magnesium (Mg) and magnesium nanocomposite (Mg-CNT). The Mg powders were mixed in a high-energy planetary ball mill at CEMAJOR Lab of Annamalai University, Chidambaram, Tamilnadu, with 0.5, 1, and 1.5 wt% CNTs and ball milled for 2 h which helped CNTs to get dispersed evenly.

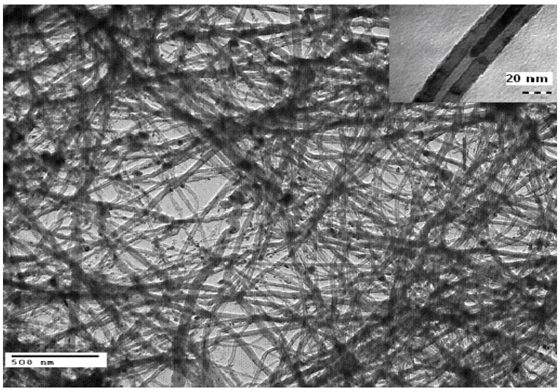

Fig. 3: *TEM Image of As-Produced MWCNT.*

The homogenized powder in different variations of CNTs is compacted into cylindrical billets of 30 mm diameter and 40 mm height in a die at a pressure of 25 tons using a 100-ton hydraulic press under ambient conditions. Monolithic Mg billets were also fabricated directly by compacting pure Mg powders without CNTs. The monolithic Mg and Mg-CNT billets were sintered in a microwave sintering furnace at 500 ^{o}C for 2 h.

Finally, the sintered specimens are extruded in a die with 45^{o} die angle at 350 ^{o}C temperature to 12 mm diameter using a hydraulic press attached with heating arrangements. The after-processed specimens of monolithic Mg and Mg-CNT were investigated for mechanical properties and compared.

Archimedes principle was used to measure the density of Mg and Mg-CNT nanocomposites. Vickers hardness measurements were conducted on the extruded specimens of Mg and Mg-CNT nanocomposites. The 0.2% YTS, UTS of the extruded Mg and Mg nanocomposites were determined according to ASTM standards.

RESULTS AND DISCUSSIONS

The density measurement and hardness results of Mg and Mg-CNT nanocomposites are shown in Table 1. From the table it is observed that the density of the nanocomposites decreases with increase in the weight percentages of CNTs. The macrohardness results of Mg and Mg-CNT nanocomposites are observed and it is found that there is an increase in the macrohardness of Mg-CNT nanocomposites with the addition of CNTs.

Table 1: *Density Measurement and Hardness Results of Mg and Mg-CNT Nanocomposites.*

Description	Density (gm/cm^3)	Hardness (Hv)
Mg	1.740	62 ± 1
Mg + 0.5 wt% CNTs	1.738	72 ± 1
Mg + 1.0 wt% CNTs	1.736	76 ± 2
Mg + 1.5 wt% CNTs	1.716	80 ± 2

Tensile behavior of Mg and Mg-CNT nanocomposites is shown in Table 2. It is observed that there is an increase in 0.2% YTS and UTS with addition of CNTs into the Mg matrix. The ductility increases with 0.5 and 1 wt% CNT and decreases with increase in the amount of CNT to 1.5 wt% with the level similar to pure Mg. It is also observed that density which decreases with 0.5 and 1 wt% CNT and significantly decreases with 1.5 wt% CNT shows the uniform distribution of CNTs in Mg matrix.

The mechanical characterization of Mg-CNT nanocomposites reveals that the powder metallurgy technique can be adopted for their manufacture followed by high-energy ball milling to disburse the MWCNTs effectively into the Mg matrix. The results show the increase in tensile strength and modulus with increase in CNT without compromising ductility.

Table 2: *Tensile Behavior of Mg and Mg-CNT Nanocomposites.*

Description	0.2% YTS (MPa)	UTS (MPa)	Ductility (%)
Mg	135 ± 5	204 ± 4	10 ± 2
Mg + 0.5wt% CNTs	138 ± 4	202 ± 2	12 ± 1
Mg + 1.0wt% CNTs	142 ± 2	207 ± 4	11 ± 1
Mg + 1.5wt% CNTs	146 ± 5	210 ± 6	10 ± 1

The reason for increase in the improved mechanical behavior of Mg-CNT nanocomposite is discussed below:

The yield strength of a reinforced matrix is given by the following equation given by Dai *et al.* [16].

$$\sigma_{my} = \sigma_{m0} + \Delta\sigma \quad (1)$$

where σ_{my} and σ_{m0} are the yield strength of the reinforced and the unreinforced matrix respectively. $\Delta\sigma$, which represents the total increment in yield stress of the reinforced matrix, can be estimated by [17]:

$$\Delta\sigma = \sqrt{}(\Delta\sigma_{EM})^2 \pm (\Delta\sigma_{CTE})^2 \quad (2)$$

where $\Delta\sigma_{EM}$ and $\Delta\sigma_{CTE}$ are the stress increments due to elastic modulus and coefficient of thermal expansion mismatch between the matrix and the CNTs.

These, as determined by Taylor dislocation strengthening mechanism, can be expressed as:

$$\Delta\sigma_{EM} = \sqrt{3}\alpha\mu b\sqrt{\rho_G}^{EM} \quad (3)$$

and

$$\Delta\sigma_{CTE} = \sqrt{3}\beta\mu_m b\sqrt{\rho_G}^{CTE} \quad (4)$$

where μ_m is the shear modulus of the matrix, b is Burgers vector, and α and β are the strengthening coefficients.

The geometrically necessary dislocations are stored near the surfaces of the CNTs for accommodation of deformation caused by elastic modulus and CTE mismatch between the matrix and the carbon nanotubes.

The geometrically necessary dislocation density due to elastic modulus mismatch [18] is given by:

$$\rho_G^{EM} = \nu^m/b\lambda \quad (5)$$

where γ^m is the shear strain in the matrix, and λ is the local length scale of the deformation field, which can be interpreted as the distance whereby dislocations generated at the reinforcements are restrained from movement. λ is affected by fine matrix grain size as well as reinforcement spacing [19]. Rod-shaped reinforcements such as CNTs are deduced to strengthen the matrix more effectively than spherical reinforcements due to resultant shorter inter-reinforcement spacing. According to a study by Kelly [20], rod-shaped particles resulted in approximately twice as much strengthening as spherical particles of the same volume fraction.

Arsenault *et al.* [21] has observed that during thermal cycling, i.e., during the sintering and cooling processes, the distribution of dislocations within the matrix of the composites was not uniform and there was a higher density near the reinforcing particles. These geometrically necessary dislocations generated in the matrix around the reinforcements due to difference in coefficients of thermal expansion between the matrix and CNTs can be estimated by the following equation, which has been previously derived for rod-shaped reinforcements:

$$\rho_G^{CTE} = \frac{10 f_{CNT}{}^{\epsilon}}{b(1-f_{CNT}) d_{CNT}} \quad (6)$$

where f_{CNT} is the volume fraction of the CNTs, ε is the misfit strain due to the different CTE of Mg and CNTs, and d_{CNT} is the diameter of the CNT. From Eq. (6), it can be seen that with increasing volume fraction of CNTs and decreasing diameter of the CNT, a higher dislocation density due to CTE mismatch can be generated, and hence higher yield strength can be obtained. This equation fits the results obtained in this experiment, where an increasing amount of CNTs incorporated has resulted in increasing yield strength.

UTS results of the Mg nanocomposites remain relatively unchanged with increasing weight percent of CNTs. The fluctuations in the strength are still within the standard deviation. No obvious strain-hardening behavior was observed in the Mg nanocomposites as compared to monolithic Mg. When micron-sized ceramic or metallic particles are added to Mg as reinforcements, the UTS of the resultant composites will usually drop due to particle fracture or particle/matrix interfacial failure. CNTs in Mg remain intact during tensile deformation due to their high tensile strength which could be as high as 30 GPa [22], the excellent mechanical property of CNTs effectively eliminates the possibility of reinforcement fracture during tensile deformation which could contribute to a decrease in the UTS of the nanocomposites.

An increase in ductility has been observed in Mg reinforced with up to 1.5 wt.% of CNTs. Increased ductility in Mg composites was previously found in Mg reinforced with micron-sized Ti particles [23] and Al_2O_3 nanoparticles [24] respectively. One of the explanations given was the change in fracture mode from a brittle to a ductile one. However, this phenomenon was not observed in the present study.

CONCLUSIONS

(i) Powder metallurgy process was successfully applied to synthesize the Mg-CNT nanocomposites and was found cost effective.
(ii) The micro hardness and tensile tests have revealed enhanced mechanical properties of Mg-MWCNT composites.
(iii) The results of the mechanical behavior revealed that an increasing volume fraction of CNTs in the Mg matrix leads to an improvement in 0.2% YTS without losing ductility.

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