Fabrication and Investigation of Damping Properties of Nano Particulate Composites

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ABSTRACT

Nano particulate composites with Al-Si alloys (LM6 and LM25) as matrix and Multiwall Carbon Nanotube (MWNT) as reinforcement (0.25, 0.5, 0.75, 1.0 and 1.5 weight percentage) have been fabricated by powder metallurgy process. Damping specimens were prepared according to ASTM E 756-05 standard and the specimens were subjected to free vibration test to investigate the damping ratio and natural frequency. It is observed from the free vibration test data; both alloys (LM6 and LM25) have shown significant improvement in damping ratio, natural frequency and modulus when reinforced with 0.5 weight percentage of MWNT. Increase in weight percentage of MWNT beyond 0.5 leads to deterioration in damping ratio, natural frequency and modulus. This is due to agglomeration of reinforcement phase. The same has been observed in the morphological study using transmission electron microscopy (TEM). In this work an attempt has been made to also investigate the mechanical properties of the fabricated composites.

Keywords: *Multiwall Carbon Nanotube; LM6; LM25; Damping ratio; Natural frequency; powder metallurgy process.*

1. INTRODUCTION

In aviation, automobile and other structural applications, the demand for materials possessing superior properties like higher strength to weight ratio, high modulus and high temperature stability along with good damping ability is continuously increases. However, it is difficult to achieve all these properties in a single material. This is one of the driving force for the development of newer and newer composite materials. In this context many researchers all over the world investigating new composite materials using either polymer matrix or metal matrix with different reinforcements. For components used at higher operating temperatures, metal matrix composite materials with a good reinforcement are preferred.

In general, aluminium (Al) and its alloys have emerged as one of the most dominant metal matrix materials in the 21st century [1]. This is because of their attractive mechanical properties [2, 3]. Among the Al alloys, Aluminium-Silicon (Al-Si) alloys in particular have been utilized significantly for engineering applications due to their better mechanical and physical properties. They also possess good manufacturing ability and lower density than Al [4]. This reduces weight and results in energy savings in automotive and aerospace applications.

Many researchers have studied varieties of reinforcements with aluminum matrix to produce Metal Matrix Composites (MMCs) to achieve the required properties. Out of these reinforcements, Carbon Nano Tubes (CNT) is an ideal reinforcement since it possesses extremely high modulus of elasticity, strength, stiffness, low density and high specific surface area.

MMCs are generally fabricated through processes such as casting, extrusion and powder metallurgy (P/M). Out of these, P/M process is regarded as one of the important processing techniques on account of homogeneity in composition and microstructure, minimum scrap, consistent porosity and close dimensional tolerances. P/M process also gives ample scope to produce wide variety of alloying systems and metal matrix composites [5-8]. With this insight it has been reported that, Al based particle reinforced composites produced by P/M process exhibit attractive mechanical properties such as increased stiffness, low density, good specific strength and vibration damping [9-12]. However literature studies indicate that major works have been carried out on polymer and ceramic based CNT reinforced composites [13]. When appropriate quantity of nanotubes is mixed with polymers, they show improved mechanical properties without sacrificing any other base properties [14]. Many researchers have also produced and characterized the Carbon nanotube based Aluminium metal matrix composites by powder metallurgy process [15-17] and it has been reported that Powder metallurgy process is one of the better processing techniques for these kinds of metal matrix composites. In the present work MWNT reinforced Al-Si alloy (LM6 and LM25) based composites are produced by powder metallurgy process and their mechanical and damping properties have been investigated.

2. MATERIALS AND METHODS

2.1 Materials

Two types of nano particulate reinforced Al-Si composites were manufactured by using LM6 and LM25 powders of 200 mesh size as a matrix and MWNT as reinforcement. LM6 and LM25 were supplied by M/s Metal Powder. Co Ltd, Chennai, and the research grade MWNT was supplied by M/s Sigma Aldrich, Bangalore. The properties of as supplied MWNTs are given in Table.1.

Table.1: Properties of *MWNT*

Properties	Values
Purity	Carbon > 90% (trace metal basis)
OD × ID × L	10-15 nm × 2-6 nm × 0.1-10 μm
Total Impurities	Amorphous carbon, none detected by transmission electron microscope (TEM))
Melting Point	3652-3697 °C
Density	~2.1 g/ml at 25 °C

2.2 Methods

2.2.1 Establishment of compaction load

In order to achieve optimal compaction density, trial compactions were done at different loads for both LM6 and LM25 powders. A cylindrical die of 25.4 mm diameter was used to obtain cylindrical specimen as per ASTM B-925. A coating of zinc stearate was applied to the die and punch to minimize friction during the compaction process. 35 grams of metal powder was taken in the die and load was applied through a hydraulic press of 40 Ton capacity at the rate of 2 Ton/min. After compaction, the specimen was ejected and its volume was measured. The density was determined by mass-volume relation. The variation of density versus load for LM6 and LM25 are shown in Figure 1. From the densification trend it is observed that optimum density obtained at a compaction load of around 160 KN and 140 KN for LM6 and LM25 respectively.
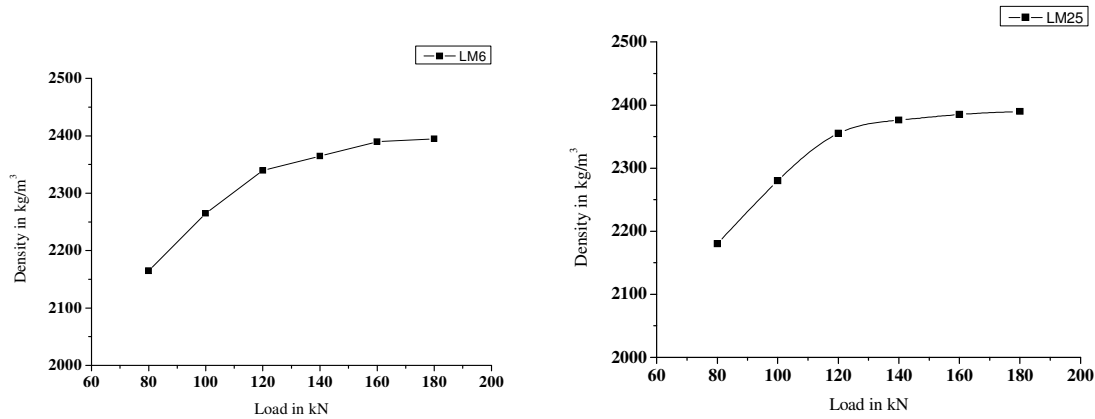
Figure 1. Variation of density with compaction load.

2.2.2 Preparation of composite

The CNT powder was initially purified by mixing it in concentrated Nitric acid, filtering and washing with de-ionized water and drying at 120° C. This is done to remove the impurities such as graphitic particles, amorphous carbon or any other impurities present. MWNT of 0.25, 0.5, 0.75, 1.0 and 1.5 weight percentage was mixed with LM6 powder in ethanol solution. The mixing was done in a ultrasonic shaker for 30 min. Finally, the mixed powders were dried at 120°C in vacuum (less than 10^{-2} Pa) and further ball milled using Retsch PM100 high speed planetary ball mill apparatus. The process of mixing is continued for duration of 10 min at 200 rpm in order to get uniform mixing. The mixture of a particular weight percentage of MWNT and LM6 was compacted in the die assembly using a 40 Ton capacity universal testing machine. The standardized load (160 KN) was applied at the rate of 2 Ton/min as per ASTM B-925. After ejecting, the green specimen was sintered in nitrogen atmosphere for 1 hour at 490° C. The sintered specimen was subjected to hot extrusion at a temperature of 350 ° C to produce the rectangular strips. Figure 2 & Figure 3 shows the photographs of specimen after compaction and extrusion respectively.

The same procedure was repeated to produce specimens of rectangular extruded strips of different weight percent content of MWNT with LM25 matrix. While compacting LM25 matrix based composite, a compaction load of 140KN and a sintering temperature of 515° C were used.

3. EXPERIMENTATION RESULTS AND DISCUSSIONS

3.1 Mechanical Properties

3.1.1 Hardness

Figure 2. Cylindrical Specimen after compaction.   Figure 3. Specimen c/s after extrusion.

Hardness of specimen of base alloys (LM6 and LM25) and MWNT reinforced composites were determined by using Rockwell Hardness Testing apparatus as per   ASTM B-925. The results are tabulated in Table 2. It is found that both for LM6 and LM25 based composites, the hardness increases with the addition of MWNT upto 0.5 wt % of MWNT and then the hardness decreases beyond 0.5 wt%. However, the hardness of LM6 composites is slightly on higher side compared to LM25 composites. This is due to the contribution of metallurgical composition and structure of base material LM6.

Table 2. Hardness values of green and sintered compacts.

Material	Load (Ton)	Hardness (RHN) Before sintering	Hardness (RHN) After sintering	For 0.25 wt % MWNT	For 0.50 wt % MWNT	For 0.75 wt % MWNT
LM6	160	18	33	39	48	44
LM25	140	17	30	35	43	39

3.1.2. Density

Density of the LM6 and LM25 base alloys and their composite before and after sintering were computed by mass-volume relation and plotted against wt% of CNT. The variation in density is shown in Figure 4. The density decreases with an increase in weight percentage of MWNT in the composites, both before and after sintering. It has also been observed that, the density decreased remarkably in both LM6 and LM25 based composites at above 1.0 weight percent of reinforcement. This is due to agglomeration of MWNT in the matrix. The agglomeration of CNT is well supported by the TEM images, shown in Figure 5.
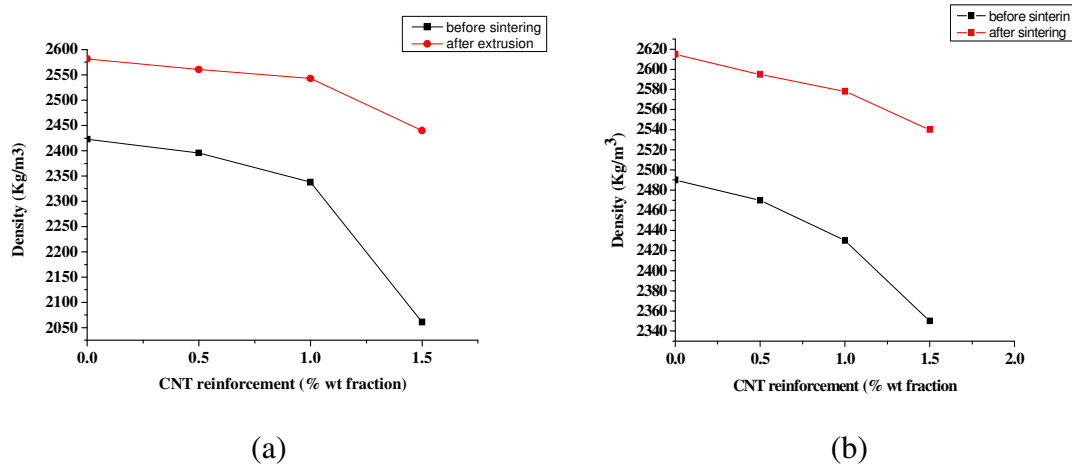
(a)   (b)

Figure 4. Density trend of (a) MWNT-LM6 and (b) MWNT-LM25
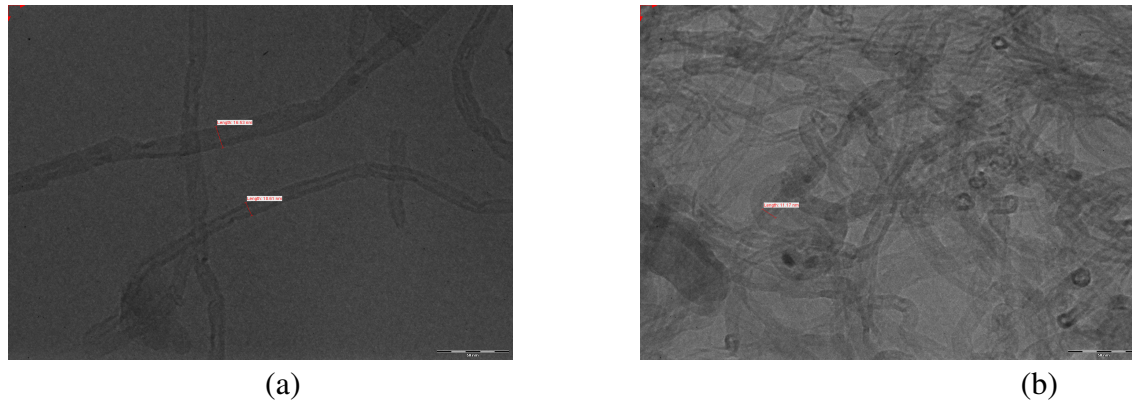Before and after sintering

(a)   (b)

Figure 5. TEM image: (a) LM6 - 0.5 wt.% MWNT composite and (b) LM6 -1.5wt.% MWNT
composite

3.1.3 Young's modulus (E)

The Young's modulus of the LM6 and LM25 alloys without MWNT and with MWNT reinforced were examined as per ASTM standard E-8 by using computer interfaced electronic load cell controlled Lloyds universal testing machine. The young's moduli of composites are given in Table 3. It is observed that LM6 composite containing 0.5 wt percentage MWNT exhibits highest (69.33GPa) value of E followed by LM25 composite containing 0.5 wt percentage MWNT (64.2 GPa). Further, increase in MWNT in both the composites is not beneficial from the point of view of modulus. This reduction in modulus is again attributed to the agglomeration of MWNT in the matrix at higher content.

Table 3. Young's modulus of LM6 and LM25 composites

Material combination	E (GPa)	Material combination	E (GPa)
LM6	64.2	LM25	59.8
LM6- 0.25 wt % MWNT	60.99	LM25-0.25 wt %MWNT	57.8
LM6-0.5 wt % MW NT	69.33	LM25-0.5 wt % MWNT	64.2
LM6-0.75 wt % MWNT	62.63	LM25-0.75 wt % MWNT	58.1
LM6-1.0 wt % MWNT	60.34	LM25-1.0 wt % MWNT	55.6
LM6-1.5 wt % MWNT	56.49	LM25-1.5 wt % MWNT	51.2

3.1.4 SEM and EDS of composites.

The EDS of 0.5 weight percentage reinforced LM6 and LM25 composites is shown in Figure 6. is performed to ascertain the correctness of composition and fabrication steps. The EDS image is shown particularly for 0.5 wt% of MWNT, as the best properties were obtained at this wt% for both LM6 & LM25. The composition obtained from EDS results are tabulated in Table 4.

Table 4. Constituents of *MWNT-LM6* and *MWNT- LM25*

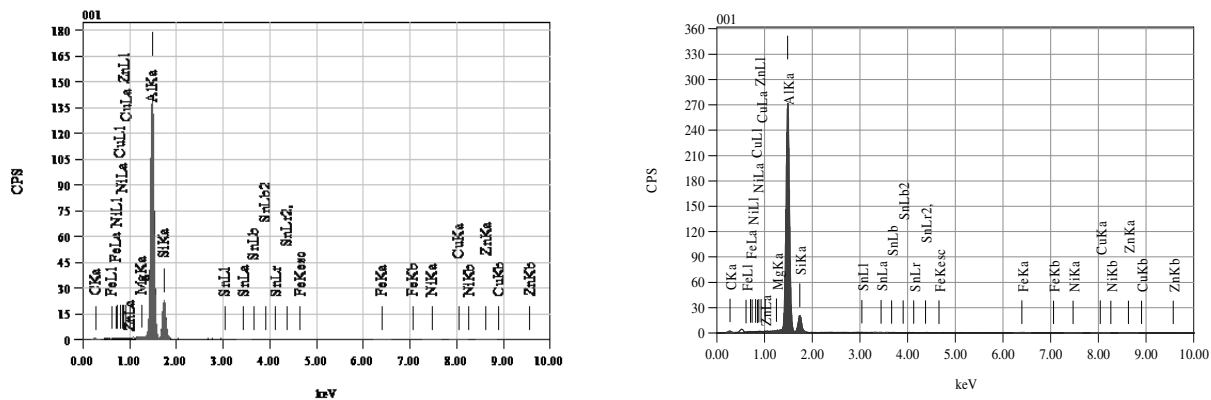| Material	Constituents	Si	Cu	Mg	Fe	Zn	Pb	Sn	C	Al
LM6	%	12.1	0.1	0.1	0.6	0.1	0.1	0.05	0.48	Remaining
LM25	%	6.9	0.1	0.3	0.5	0.1	0.1	0.05	0.47	Remaining

Figure 6. EDS image of (a) LM6-0.5 wt % MW NT and (b) LM25-0.5 wt % MWNT

3.2. Investigation of Damping Ratio

Internal damping results from mechanical energy dissipation within the material due to various microscopic and macroscopic processes. Internal damping of materials originates from the energy dissipation associated with microstructure defects such as grain boundaries and impurities, thermo-elastic effects caused by local temperature gradients, non-uniform stresses as in vibrating beams, eddy current effects in ferromagnetic materials, dislocation motion in metals and chain motion in polymers. It is observed that the material damping is the energy dissipated within the materials of construction and is due to internal hysteresis in materials arising from non-linear stress-strain behaviour, intergranular friction and thermo-elasticity [18].

In this research, specimens size of 150 mm X 15 mm X 5 mm were prepared from LM6 and LM25 based MWNT composites. The specimen were subjected to free vibration according the standard test methods for measuring Vibration-Damping properties of materials ASTM E 756-05. The specimens were treated as self supporting materials with cantilever beam configuration. The experiment was conducted on the specimen considering the dimensions of 100mm X 15mm X 5mm. This allows sufficient length for fixing the beam as cantilever. The experimental setup consists of an accelerometer A&D 3101 with sensitivity 9.8mV/g which is used to measure the beam response. Data acquisition is through National Instruments eight channel industrial platform sound and vibration measurement module having 24 bit resolution and acquisition rate capability of 1024 kS/s. Signal conditioning and analysis is done through National Instruments LabVIEW 8.6 software. The Experiment setup and LabVIEW block diagram for vibration measurement is shown in Figure 7.
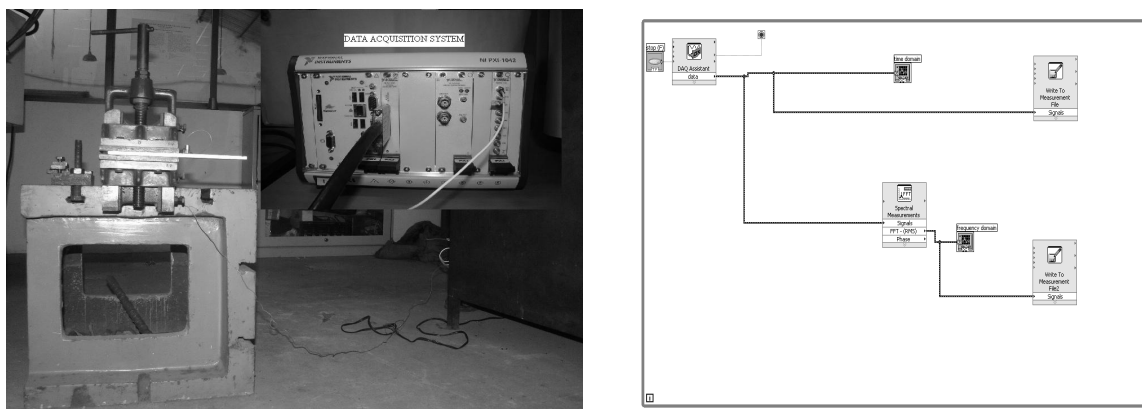

Figure 7. Free vibration test setup and LabVIEW block diagram

Three sample specimens were subjected to testing in each composite and average value was taken to compute damping ratio and the natural frequency. The damping ratio is calculated and tabulated in Table 5.

A typical free vibration response for a Single degree freedom system is shown in Figure 8. The Logarithmic decrement is computed from the amplitudes using the following Equation 3.1.

$$\text{Logarithmic decrement, } \delta = \left(\frac{X_1}{X_2} \right) = \frac{1}{n}\left(\frac{X_1}{X_{n-1}} \right) \tag{3.1}$$

Where, X1 and X2 are first two successive amplitudes and n is the number of cycles. The damping ratio is computed by using the Equation 3.2.

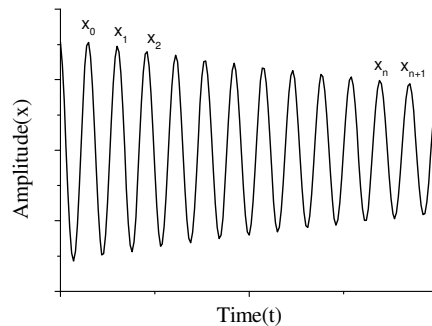$$\zeta = \frac{\delta}{\sqrt{4\pi^2 + \delta^2}} \tag{3.2}$$

Figure 8. Free vibration response of a single degree freedom system.

From the experimental data mentioned in Table 5, it is evident that 0.5 weight percent MWNT reinforced LM6 and LM25 exhibit excellent damping and natural frequencies in comparison with other weight percentages. 0.5 weight percent MWNT composite of both LM6 and LM25 also exhibit high natural frequency (416 Hz and 406 Hz). This is due to composites possessing high stiffness on account of high modulus of MWNT and its uniform distribution. The improvements in damping ratio for LM6 and LM25 with 0.5 weight percent MWNT with respect to base alloys are 50.9% and 38.1% respectively. At lower weight percentage of MWNT in the composites, the improvement in the damping ratio and natural frequency is very marginal. Higher weight percent of MWNT in the composites leads to decrease in damping ratio and natural frequency. Clustering of MWNT at higher weight percentage causes the decrease in natural frequency and damping ratio. At 0.5 weight percentage, the MWNT is uniformly distributed. This results in good bonding between the reinforcement and the matrix. This provides large interfacial area

between matrix and MWNT. This increases the modulus value as well as energy dissipation at interface.

Table 5. Natural frequencies and damping ratios of *LM6* and *LM25* based *MWNT* reinforced composites.

Material combination	Natural Frequency (Hz)	Damping Ratio	Material combination	Natural Frequency (Hz)	Damping Ratio
LM6	405	0.0055	LM25	399	0.0042
LM6- 0.25 wt % MWNT	402	0.0056	LM25-0.25 wt %MWNT	397	0.0045
LM6-0.5 wt % MW NT	416	0.0083	LM25-0.5 wt % MWNT	406	0.0058
LM6-0.75 wt % MWNT	406	0.0052	LM25-0.75 wt % MWNT	400	0.0043
LM6-1.0 wt % MWNT	395	0.0043	LM25-1.0 wt % MWNT	386	0.0040
LM6-1.5 wt % MWNT	380	0.0041	LM25-1.5 wt % MWNT	374	0.0038

4. CONCLUSION

LM6 and LM25 composites with MWNT in weight percentages of 0.25, 0.5, 0.75, 1.0 and 1.5 as reinforcement were produced through powder metallurgy route. The extruded specimens were subjected to free vibration test to evaluate the damping ability and natural frequency (stiffness). From the investigation, following points are concluded.

➢ LM6 has better damping ability than LM25.
➢ Both alloys show marked improvement in damping ratio when reinforced with 0.5 weight percentage of MWNT.
➢ More than 0.5 weight percentage of MWNT leads to clustering and thus the damping and stiffness properties are reduced remarkably.
➢ From the experimental observation, it is concluded that the mixing and compaction parameters for densification need to be standardized for each base material and MWNT weight percentage in order to further realize improvement in damping and stiffness properties.

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