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Multi Walled Carbon Nanotube Composites with Mumetal for Electromagnetic Interference shielding

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Abstract
Electromagnetic Interference (EMI) Shielding material containing a blend of multi walled carbon nano tube (MWCNT) and mumetal has been prepared and their electromagnetic shielding capabilities were characterised. The mumetal (alloy of Ni, Fe, Cu and Cr) in nano form has been synthesised by milling the respective elements in different compositions. The MWCNT has been prepared by CVD technique. After the purification of CNT, EMI shielding composites were prepared in polyvinylidenefluoride (PVdF). The structural features of the various composites were employed through XRD, SEM, EDAX, etc. The Shielding effectiveness (SE) of the prepared composites 3 wt % MWCNT & 3 wt % mu metal, 3 wt % MWCNT & 10 wt % mu metal, 3 wt % MWCNT & 15 wt % mu metal and 7 wt % MWCNT & 30 wt % mu metal were 8.38 dB, 9.62 dB, 8.5 dB,15.9 dB and 17.2 dB respectively.

Index Terms- EMI, Shielding Effectiveness, Nanocomposites, Polymer

I.INTRODUCTION
Electromagnetic Interference (EMI) is a well known problem in commercial and scientific electromagnetic instruments, antenna systems and military electronic devices; there is a critical need for developing effective and practical EMI shielding materials and their potential applications. The Electro Magnetic Radiations (EMR) from one device may interfere with other devices causing severe problems [1]..Electro Magnetic Interference (EMI) shielding refers to the blocking of electromagnetic radiation so that the radiation cannot pass through the shield. Light weight EMI shielding is needed to protect the workspace and environment from radiation coming from computers and telecommunication equipment as well as to protect sensitive circuits [2]. The EMI shielding effectiveness (SE) of a composite depends on many factors such as the conductivity of the intrinsic fillers, dielectric constant, aspect ratio etc., [3]. Materials with lower surface electrical resistivity have higher SE. Depending upon the SE at different frequency ranges, the materials can be used for the encapsulation of different microelectronic devices, computer housings, switches, connector gaskets etc., [4].
The present work deals with the preparation of MWCNT/mu-metal/Polyvinylidene fluoride (PVdF) nanocomposites for effective EMI shielding. Due to their multifunctional properties, CNT / mu-metal / polymer composites are mainly used for shielding applications. Highly conducting MWCNTs enhances the conductivity of the composites and thereby improving the SE by reflecting the EM radiations. The drawback of increasing the % of CNT would reduces the mechanical properties of the composites like strength, ductibility etc. However, it is difficult to achieve the maximum enhancement of physical properties through CNTs without homogeneous dispersion and strong interfacial adhesion between CNTs and polymer matrix [5]. Magnetically permeable mu-metal acts as magnetic filler, it is effective for the attenuation of electromagnetic radiation by absorption.

II.MATERIALS AND METHODS
Synthesis of mumetal alloy:
Mu-metal is an alloy of Nickel, Iron, Copper and Chromium with very high magnetic permeability. The composition of mu-metal is 77 wt % of nickel, 6 wt % of iron, 5 wt % of copper and 2 wt % of chromium. Ni (3.87299 g), Fe (0.76566 g), Cu (0.27225 g) and Cr (0.0891g) were weighed with particle sizes of + 200 mesh, 5 μ, + 325 meshes, ≤ 325 mesh respectively. It is then grinded using mortar and pestle. The grinded powders were milled in a high energy planetary ball mill with ball to powder ratio as 10:1 at the rotation speed of 200 rpm. In order to minimize oxidation, the entire operation was performed in an argon atmosphere. After 12 hours of milling, the fine powders of mu-metal alloy has been collected and stored in vacuum.

Synthesis of MWCNT / mu-metal / PVdF composites:
The MWCNT / mu metal / PVdF composite were fabricated in N-N, Dimethyl formamide (DMF) as a solvent. 30 mg of f-MWCNT was dispersed in 40 ml of DMF and sonicated for 30 minutes. 30 mg of mu metal was dispersed in 30 ml of DMF and sonicated for 30 minutes separately. Polyvinyliedene fluoride (PVdF) solution was obtained by dissolving 940 mg of PVdF in 40 ml of DMF. All the three solutions were mixed well and again sonicated for 1 hour. After that, the suspension was agitated well in the mixer at 3000 rpm for 6-7 hours. Finally, it is transferred into the petri-dish and kept in oven at 100˚C overnight. Based on this procedure, different compositions of the composites were prepared. Various composites prepared are represented as **A :** 3 wt % f-MWCNT and 3 wt % mu metal, **B :** 3 wt % f-

MWCNT and 10 wt % mu metal, **C :** 3 wt % f-MWCNT and 15 wt % mu metal, **D :** 7 wt % f-MWCNT and 15 wt % mu metal, **E:** 7 wt % f- MWCNT and 30 wt % mu metal

III.RESULTS AND DISCUSSION

Characterization of the mu metal using XRD:

The XRD patterns were recorded for the mu-metal mixture (before milling) and mu- metal alloy (after milling). The extent of alloy formation has been confirmed from the XRD pattern. Fig. (1 a) represents the XRD pattern of mumetal mixture and fig. (1 b) represents the mu-metal alloy.

From fig.(1a), it is observed that the individual peaks for nickel (Ni), iron (Fe), copper (Cu) and chromium (Cr) are found. The peak at 43.435 degree shows the presence of Cr. The peaks corresponding to 44.494, 51.8474 and 76.378 degree confirm the presence of Ni and the peak at 74.2746 shows the presence of copper. Iron peak is merged with Cr and Ni peak and it shows a peak at 44 degree.

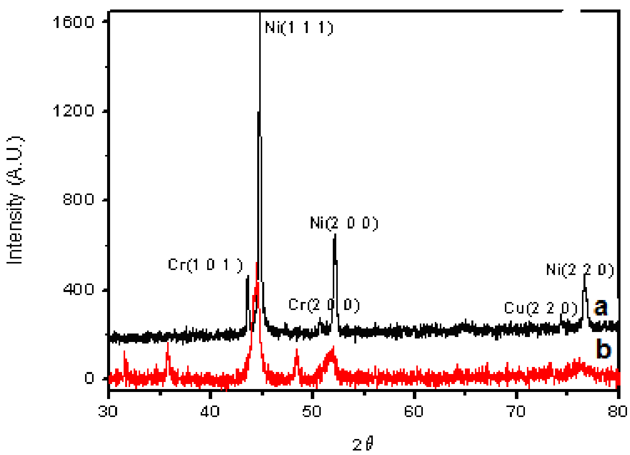


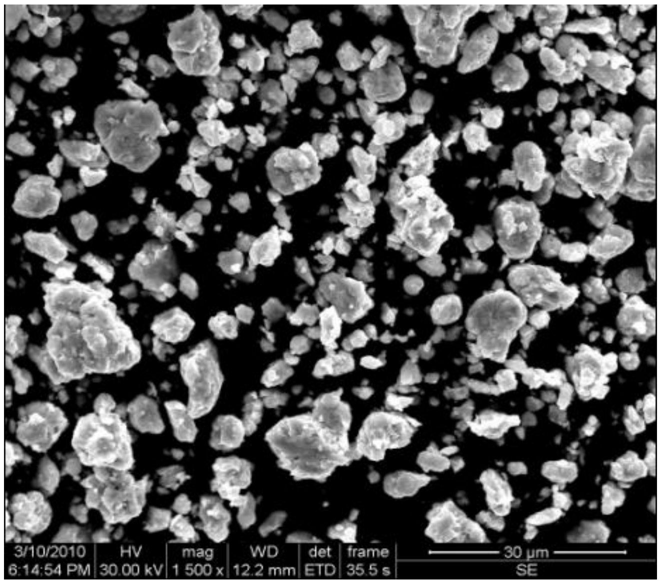
Fig. 1 XRD patterns of mu- metal (a) before and (b) after milling

From fig.(1b), it is clear that the individual characteristic peaks for the pure elements are disappeared and a new peak with line broadening effect confirms the formation of mumetal alloy in nano form [6].

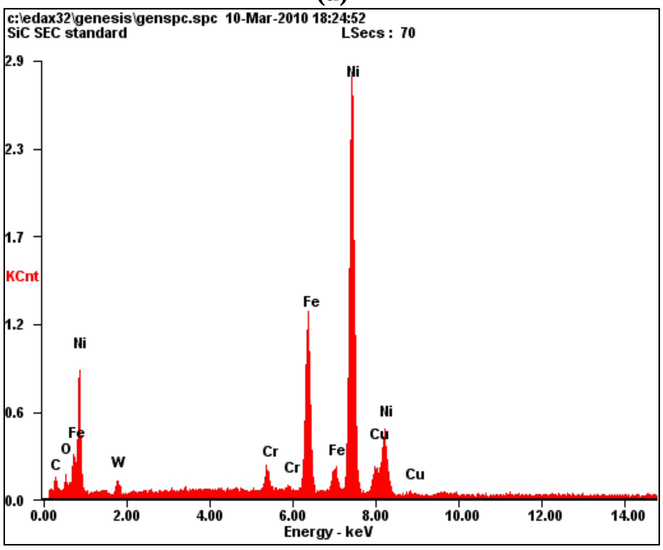
Characterization of mu metal using SEM with EDAX:

Figure (2, a) presents the SEM image of the mu metal alloy. It is observed that the diameter of the particle size reduces and it is in spherical shape. This spherical shape is resulted from continuous refinement of particle size with increase in milling time. The particle size distribution is also narrower [6].

From the EDAX (figure 2, b) spectrum, the composition of the alloy is determined. It is found that the alloy contains Ni - 62.46 wt %, Fe - 16.57 wt %, Cu - 5.46 wt % and Cr - 1.90 wt % respectively. The results of EDAX are well consistent with the analysis by XRD patterns.



(a)

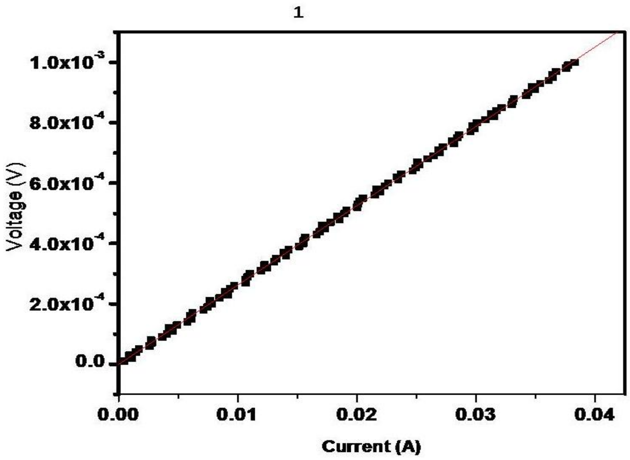


(b)

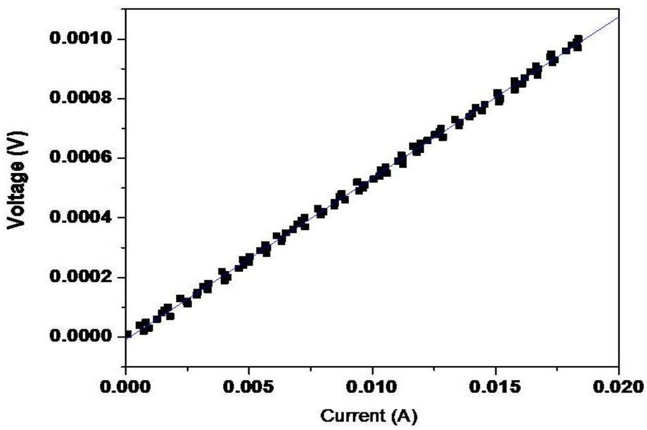
Fig. 2 a, SEM image of the mu- metal b, EDAX spectrum of mu- metal

DC Electrical resistivity:

Electrical resistivities of the various polymeric composites (A to D) have been measured using four probe method. Figure (3) represents the V-I characteristics of the different composites from **A** to **D**. The resistance values of the respective composites have been evaluated by taking the slope as R=V/I. The composites **A, B, C** and **D** have resistance values of 0.05415 V/A, 0.0263 V/A, 0.01591 V/A and 0.00986 V/A respectively. The composite 'A' has very high resistance value on comparison with other composites and possesses low conductivity. Thus the resistivity values greatly influence the SE of the composite materials. Those composites having lower resistivity will have higher SE. As per these data, the composite 'D' shows higher SE than other composites. Shielding Effectiveness studies also confirm these results.



(A)



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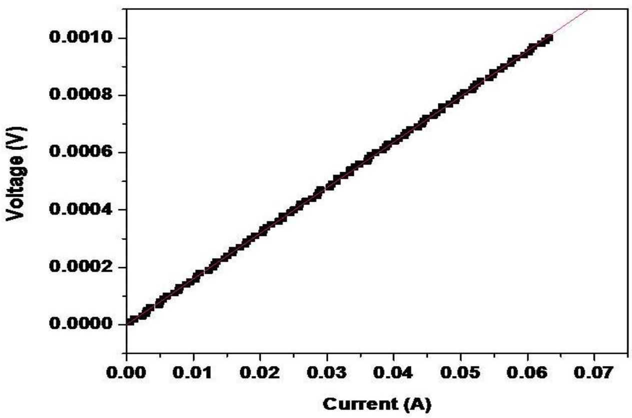
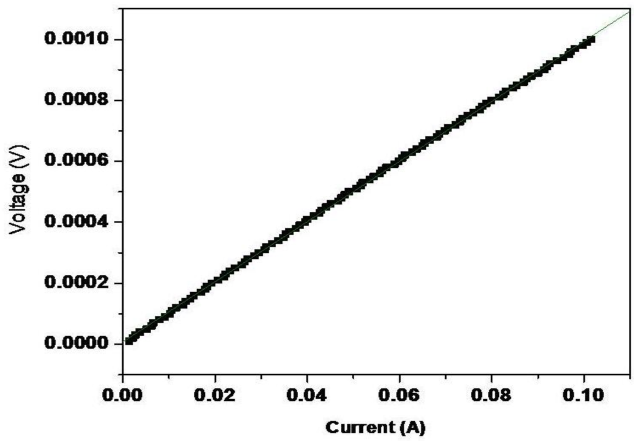


(C)



(D)

Fig. 3 V-I Characteristics of the MWCNT / Mu metal / PVdF composite film A to D

EMI Shielding Effectiveness (SE) Characteristics:

Fig (4) represents the EMI shielding effectiveness (SE) of various composites (A to E) over the frequency range of 8-12 GHz consisting of various amounts of magnetic and conductive filler. The EMR is attenuated by reflection as well as absorption. For reflecting the radiation, the shield should be conducting in nature. For the absorbing the radiation, the shields should be of highly magnetic. The prepared nano composites consists both type of materials. So the EMI-SE could take place by reflecting and also by absorbing the EM radiations.

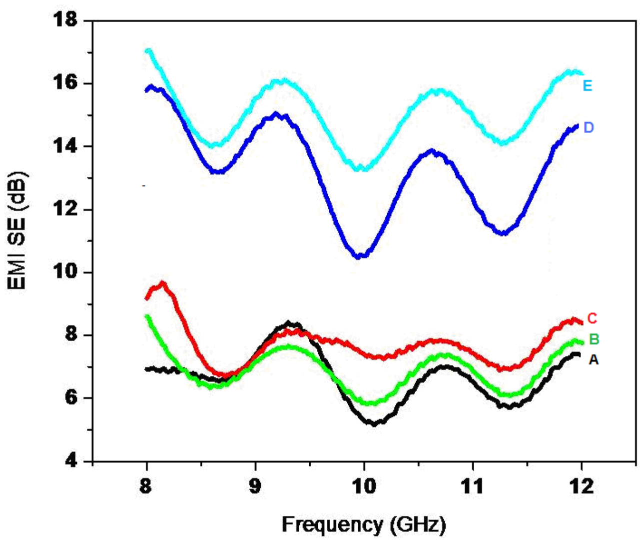


Fig. 4 EMI shielding effectiveness of various composites A to E

From the graph, Fig 4., the SE of the composites have been calculated for **A** to **E** and found that 8.38 dB, 9.62 dB, 8.5 dB, 15.9 dB and 17.2 dB respectively. The composite **B** has higher SE when compared with **A**. This is because of increase in magnetic filler content. Since the mumetal is highly permeable, it attenuates the radiation by absorption. The composite **C** has lower SE value than the composite **B.** Even though it possess higher % of mumetal, the SE value gets reduced. Such higher % of mumetal suppresses the conducting nature of the composite. The higher SE of the composites **D** and **E** is due to increase in magnetic filler as well as conducting filler. Higher % of MWCNT and mu-metal alloy enhances the SE values simultaneously by reflecting and absorbing the EM radiations.

Electrical conductivity is the most important property for effective EMI shielding for a material. As such, PVdF is an insulating material (10^{-16}S/cm). In the case of MWNTs and in presence of mumetal the electrical conductivity increases. In the case of MWNTs the conductivity may be due to percolation behaviour whereas in the case of mumetal the conductivity may be due to the presence of free electrons from the metal which may help in tunnelling inside MWNTs. High aspect of ratio and good dispersion of MWNTs may be responsible factor for the conductivity in PVdF.

Electromagnetic interference shielding effectiveness can be better understood from the study of various mass fractions of MWNTs. EMI shielding effectiveness of pure PVdF reported in the aforementioned ref is 0.3 dB. This is due to the insulating nature of PVdF. When 1 wt% of MWNT s is added the EMI shielding is reported to be increased to 2.27dB. This increase may be due to the semiconducting property of MWNTs in PVdF matrix. Hence for an increment in EMI SE, it is necessary to have conducting path and connective network in an insulating material. In the present study the conducting path is provided by mu-metals and the connective network is provided by MWNTs / PVdF composites. In the present case maximum of 18dB has been achieved by the use of Mu metal. Thus the present study results suggest that the MWNTs with mumetal can be used for electrostatic discharge applications. In these cases the most prominent EMI shielding mechanism may be due to reflection. This may be due to the presence of conjugated π electrons on the surface of MWNTs from mumetal. It may be due to the absorption radiation which may be due to electric dipoles that are present between the metal and MWNTs in PVdF matrix. It is reported that MWCNTs and PVdF composites exhibit more absorption than reflection.

The electrical conductivity of the composites prepared from A to E increases. This aspect is really beneficial to EMI SE since the conductivity increase by the use of mumetal will be a better technique to avoid skin effect and nano fibers filled PVdF coating materials with different milling conditions.

Target value of EMI S.E is 20 dB for commercial applications and in the present case it has been achieved almost to this value (18dB) for E, 7 wt % MWCNT metal and 30 wt % mumetal. The dominating shielding mechanism in this case may be due to reflection of electromagnetic radiation. In the present case EMI shielding effectiveness may be due to the composite material and this is mainly due to the conducting mechanism that has been achieved by the high % of mumetal. MWCNT presence helps to fill the void space between carbon nanofibers within PVdF polymer matrix.

EMI SE (dB) is calculated using the expression

$$\text{EMI S.E} = 20 \log (1+ 0.5\ \sigma\ d\ Z_0)$$

Where, σ is the conductivity, d is the thickness and Z_0 is the free space impedance which in the present case, the value is taken as 377 Ohms. The theoretical values are calculated for the thickness of the membrane as indicated in the graph and the experimental values are obtained for the thickness of the membrane to the values scaled up by 10 as indicated in the graph and almost it agrees well with the experimental values for the mu metal MWCNT in PVdF matrix. The thickness is large compared to the theoretical thickness and this is in view of the lower conductivity values of the polymer matrix. It is found that the presence of chlorine in polymer wastes has enhanced their capability to absorb the low energies [7]. Due to their unique geometry and the chirality, carbon nanotubes were supposed to have unique electrical and optical properties that could be used in nanoelectronics [8].

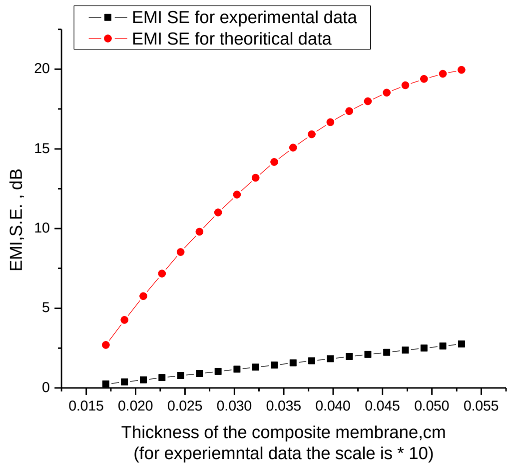


Fig. 5 Shielding Effectiveness curve for experimental & theoretical data

IV.Conclusion

EMI shielding material called mu metal has been synthesized by grinding the Ni, Fe, Cu and Cr in proper wt % in ball mill. The structural features has been established through XRD, SEM, EDAX etc., Highly conducting filler material used in the composite is MWCNT. The MWCNT has been prepared in CVD technique. After the purification, EMI shielding composite were prepared in PVdF. The shielding effectiveness (SE) of the prepared composites was measured using vector network analyzer in X-band frequency range. From this result, it is concluded that the highly conducting composite with higher % of mu-metal possess high shielding effectiveness.

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