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# Non-monotonic Coercivity of Iron Filled Carbon Nanotube/Polystyrene Composites

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## Abstract

Multiwall carbon nanotubes (MWCNT) filled with magnetic nanoparticles constitute a fabulous magnetic system due to the fact that encapsulated magnetic nanoparticles remain protected from both aggregation and oxidation. Such a magnetic system has exotic properties, e.g. an enhanced magnetic coercivity. The coercivity is observed to get further enhanced if these magnetic nanoparticles filled MWCNT are homogeneously dispersed into a polymer matrix. We attempted to investigate the magnetic response of iron filled-multiwall carbon nanotubes (Fe-MWCNT) by dispersing them in polystyrene (PS) in different amounts. In this fashion we prepared Fe-MWCNT/PS composites of 0.1 to 7.0 wt%, and studied the variation in coercivity ($H_c$) of composites at 300, 150 and 10 K. An unexpected non-monotonic variation in the value of $H_c$ of Fe-MWCNT/PS composites as function of Fe-MWCNT loading is understood qualitatively by considering the effect of inter-particle separations on dominant magnetic interactions.

## Keywords

Fe-MWCNT, Polymer composite, Coercivity

Iron-filled multiwall carbon nanotubes (Fe-MWCNT) are fantastic filler materials for the fabrication of multifunctional composite materials as their inclusion to a non-conducting and diamagnetic polymer matrix transforms into a conducting and ferromagnetic composite materials, which can be used for applications such as electromagnetic shielding [1]. More importantly, encapsulation of Fe- nanoparticles (NP) within MWCNT prevents oxidation and aggregation of Fe-NP while composite processing [2]. We studied the variation in coercivity ($H_c$) of composites (with MWCNT loading from 0.1 to 7 wt%) at 300, 150 and 10 K; composites were prepared by dispersing Fe-MWCNT in poly-styrene (PS). Non-monotonicity of coercivity ($H_c$) in composite films has been observed as a function of MWCNT loading which is qualitatively explained on the basis of variation in the strength of magnetic interactions as a function of inter-particle distances.

Fe-MWCNT used for the fabrication of the polymer composites were synthesized by thermally assisted chemical vapor deposition of toluene/ferrocene (1 mL/100 mg) mixture; toluene acts as carbon source material and ferrocene provides the catalytic Fe nanoparticles. The details on the synthesis of Fe-MWCNT are given elsewhere [2]. The FEI Quanta 200 scanning electron microscope (SEM) and Technai F30 transmission electron microscope (TEM) were used for the detailed morphological and structural investigations. Free standing composite films (thickness of film ~50 $\mu$m) of Fe-MWCNT/PS are prepared by the solution processing casting technique. The temperature dependent magnetization data are recorded by using Quantum Design SQUID

magnetometer (MPMS XL). The data are acquired in steps of 400 Oe.

SEM & TEM micrographs of the as-synthesized Fe-MWCNT sample are presented in Figure 1. SEM micrograph (Figure 1A) shows reasonably good one dimensional morphological features of MWCNT, but it did not provide any clue of Fe within the tubes. The encapsulation of Fe nanoparticles within the tubes is established by TEM characterization along with the energy dispersive analysis of X-rays (EDAX) measurements (Figure 1B and 1C); TEM & high resolution TEM micrographs confirm the entrapment of Fe nanoparticles of rod-like shapes, with an average diameter and length of 15 nm and 80 nm, respectively. The electron microscopy studies of Fe-MWCNT/PS composites have shown that the nanotubes are quite well dispersed in PS [2].

In order understand the results clearly, the parameters describing the magnetic response of these composites are extracted from M-H data and are tabulated in Table 1. The $H_c$ and squareness ratio ($S$=$M_r$/$M_s$) are non-monotonic while the saturation magnetization ($M_s$) shows monotonic variation with Fe-MWCNT concentration within composites; monotonicity of $M_s$ is quite understandable as value of $M_s$ depends on the Fe content which increases with Fe-MWCNT loading. However, non-monotonic variation of $H_c$ and $S$ as a function of Fe-MWCNT concentration appears to be an interesting observation; both $H_c$ and $S$ follow similar trend. It is to be noted that the $H_c$ is maximum for 1 wt% composite and its value is close to 2.7 kOe (at 10 K) which is an unexpected result, and this enhancement is mainly due to the dominance of dipolar interactions among Fe-NP. It is worth noting that the maximum value of $H_c$ for bulk Fe-MWCNT is around 2.3 kOe (at 10 K) [2]. The occurrence of a maximum $H_c$ of 1 wt% can be understood using Figure 2. It is known that $H_c$ of dispersed ferromagnetic nanoparticles (NP) system varies as a function of the inter-particle separation and distribution; NP dispersed in a non-magnetic matrix interacts either via long range dipolar interactions (characterized by $H_{ij}^{dipole} = [\mu_i.\mu_j - 3(\mu_i.\hat{r}_{ij})\ (\mu j.\hat{r}_{ij})]/r_{ij}^3)$ or short range exchange interactions, which in turn depends on the inter-particle distances [3, 4]. The exchange interactions dominate when the NP are agglomerated while the dipolar interactions will be more effective when the ferromagnetic NP are uniformly dispersed. The dominance of the dipolar interactions tends to enhance the $H_c$. In the composite with an intermediate (medium) loading of Fe-MWCNT (say $p$) the inter-particle separation (or critical separation) between Fe-NP is such that the dipolar interactions are most effective, and the $H_c$ is maximum. In case of composites with low Fe-MWCNT loading (less than $p$) the inter-particle separations are larger than the critical separation (see Figure 2), that weakens the effectiveness of dipolar interactions which results in the decrease in the value of $H_c$. For composites fabricated with high Fe-MWCNT loading (more than $p$) the Fe-NP come closer such that the separations between Fe-NP are less than the critical value, and hence the exchange interactions dominate over dipolar interactions, resulting once again in the lowering of $H_c$. Thus the observation of a maximum in $H_c$ vs. Fe-MWCNT loading for composite of 1 wt% suggests that the inter-particle separations between the encapsulated Fe-NP lie in the critical range, and the value of $p$ happened to be 1 wt%.

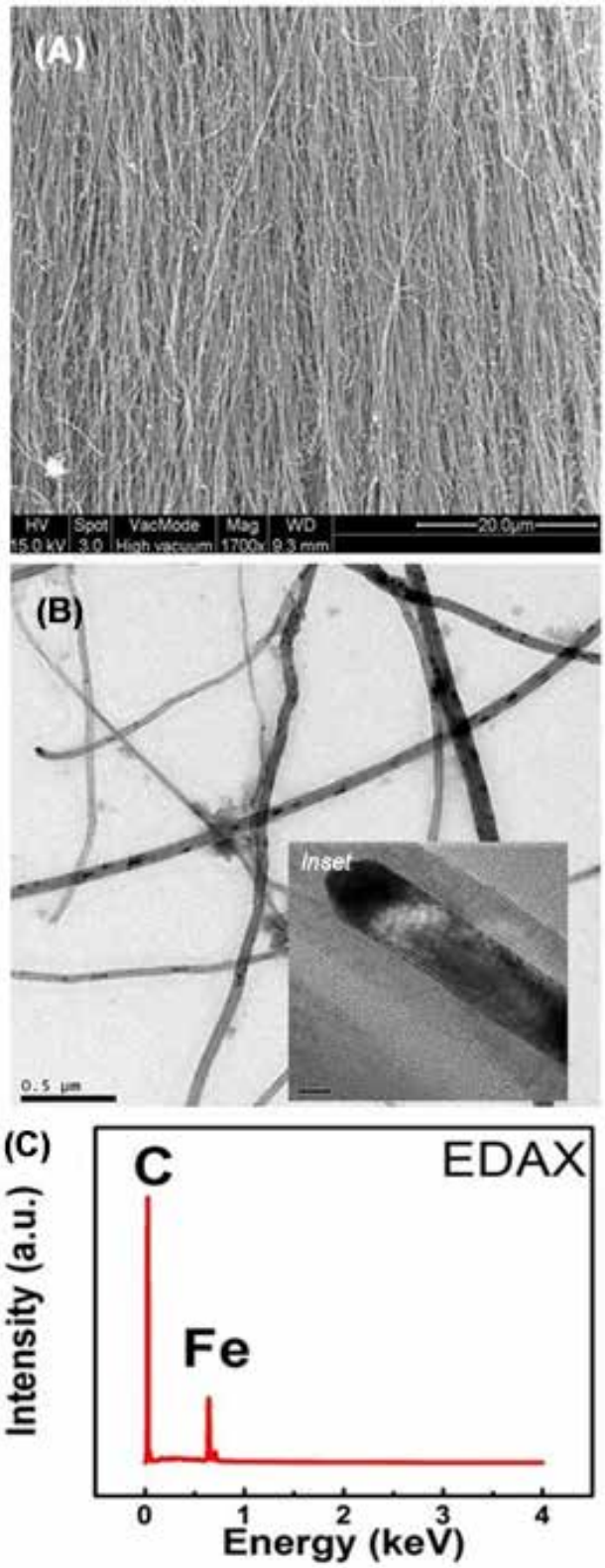


**Figure 1: (A)** SEM micrograph of MWCNT. **(B)** TEM micrograph of MWCNT. **Inset**: High resolution TEM micrograph displaying the encapsulated Fe-NP. **(C)** EDAX pattern of MWCNT.

**Table 1:** Values of coercivity ($H_c$), saturation magnetization ($M_s$) and squareness ratio (S) of Fe-MWCNT and Fe-MWCNT/ PS samples at various temperatures.

| | $H_c$(Oe) | | | $M_s$(emu/g) | | | $S=M_r/M_s$ | | |
|---|---|---|---|---|---|---|---|---|---|
| T (K) → Sample ↓ | 10 | 150 | 300 | 10 | 150 | 300 | 10 | 150 | 300 |
| 0.1 wt% | 508 | 329 | 197 | 0.008 | 0.006 | 0.006 | 0.267 | 0.533 | 0.383 |
| 0.2 wt% | 1385 | 1032 | 459 | 0.013 | 0.010 | 0.009 | 0.433 | 0.565 | 0.384 |
| 0.3 wt% | 1577 | 1036 | 402 | 0.019 | 0.014 | 0.013 | 0.495 | 0.563 | 0.385 |
| 0.5 wt% | 1750 | 1034 | 416 | 0.034 | 0.028 | 0.025 | 0.412 | 0.401 | 0.264 |
| **1 wt%** | **2658** | **1564** | **583** | 0.080 | 0.074 | 0.071 | **0.745** | **0.609** | **0.249** |
| 7 wt% | 2302 | 1310 | 505 | 0.923 | 0.909 | 0.817 | 0.594 | 0.499 | 0.321 |
| **Fe-MWCNT** | 2289 | 1301 | 488 | **4.810** | **4.360** | **3.820** | 0.495 | 0.467 | 0.346 |

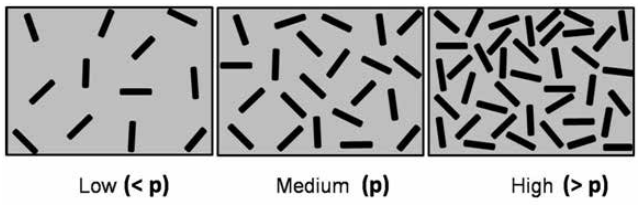


**Figure 2:** Schematic diagram showing the distribution of Fe-NP in polymer matrix with low, medium and high loading.

To conclude, non-monotonicity in $H_c$ is due to the interplay between the exchange and dipolar interactions among Fe-NP; exchange interactions dominate for high loading composites and the dipolar interactions are maximized for composite with intermediate loading, at a critical separation of NP. This suggests that factors like aggregation and inter-particle separation play major roles in observing the maximum in $H_c$. In composites above 1 wt%, the inter-particle separations are reduced so that the dipolar interactions are nearly saturated, and the enhanced exchange interactions could lower the values of $H_c$ as well. This explains the observed maximum in $H_c$ at 1 wt% composite. However, a theoretical mathematical model needs to be sought for quantitative understanding of this peculiar experimental observation.

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