Magnetic property measurements on single wall carbon nanotube-polyimide composites

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[Abstract]

Temperature and magnetic field dependent magnetization measurements were performed on polyimide nanocomposite samples, synthesized with various weight percentages of single wall carbon nanotubes. It was found that the magnetization of the composite, normalized to the mass of nanotube material in the sample, decreased with increasing weight percentage of nanotubes. It is possible that the interfacial coupling between the carbon nanotube (CNT) fillers and the polyimide matrix promotes the diamagnetic response from CNTs and reduces the total magnetization of the composite. The coercivity of the samples, believed to originate from the residual magnetic catalyst particles, was enhanced and had a stronger temperature dependence as a result of the composite synthesis. These changes in magnetic properties can form the basis of a new approach to investigate the interfacial properties in the CNT nanocomposites through magnetic property measurements.

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Introduction

Multifunctional nanocomposites prepared from single wall carbon nanotubes (SWCNTs) and polyimides are anticipated to have many aerospace applications. Nanocomposite films containing small amounts of well dispersed SWCNTs have been shown to possess enhanced electrical conductivity, mechanical strength and thermal stability [1]. Theoretically, it has been derived that external magnetic fields have a strong effect on the electronic structure and bulk properties of carbon nanotubes (CNTs) [2-3]. A SWCNT can have either a paramagnetic or diamagnetic response to an applied magnetic field depending on the tube's chirality, Fermi energy level, and the direction of the magnetic field relative to the tube axis. A SWCNT-polymer composite thus may have magnetic field sensing functions if a proper amount of SWCNTs are embedded in the polymer and the intrinsic properties of CNTs, which are sensitive to the applied magnetic field, can be measured. In addition to controllable dispersion, alignment of SWCNTs in a polymer matrix is a primary factor [4] for maximizing the use of CNTs' exceptional mechanical properties in the reinforcement of a structure. Films of strong magnetic field aligned SWCNTs and SWCNT ropes have been produced [5-6] and anisotropic behavior in some of their physical properties have been characterized [7]. Both in the magnetic alignment of SWCNTs for fabrication of a rope and in the magnetic alignment of SWCNTs and SWCNT ropes in polymers, the magnetic susceptibility of CNTs is one of the crucial parameters to determine the effectiveness of the applied magnetic field.

Raman spectroscopy has been used in the investigation of the elastic properties of SWCNTs in polymer composites [4, 8] and has been proposed as a new approach to determine the residual strains and load transfer in the composites [4]. A direct coupling of SWCNT to the polymer matrix, instead of a van der Waals type mechanism, was suggested as the cause of the frequency shift of certain Raman modes. It is possible that the magnetic property of SWCNTs would also be affected as a result of the same type of coupling in a composite and become another approach to study the interfacial properties of a nanocomposite nondestructively. Our measurements on SWCNT nanocomposite films investigate the magnetic properties of the composites and the changes in these properties with SWCNT concentration. It is hoped that the obtained results could be informative in the development of magnetic sensing capabilities within nanocomposites, for a feasibility study of in-situ alignment of nanotubes while synthesizing composites, and in the refinement of SWCNT- polyimide nanocomposite fabrication techniques.

Experimental

Using a superconducting quantum interference device (Quantum Design, SQUID-MPMS system) magnetometer, magnetization measurements as a function of temperature at constant magnetic field and as a function of magnetic field at constant temperature were performed on single wall carbon nanotube polyimide composite films. Most of the measured samples differed in weight percentage of SWCNTs. Composites with 0.1%, 0.2%, 1%, 5%, 10%, and 20% SWCNTs were studied. The weights of the composite film samples were in the range of 3 to 50 mg, and thicknesses were between 23 and 81 μm. Magnetic properties of pure polyimide and pristine SWCNT samples were also measured to provide background information. Measurements were performed over temperatures in the range of 5 K to 400 K and at magnetic fields up to 1.5 Tesla.

Data reproducibility was checked by either repeating measurements on the same sample or conducting the same measurements on different samples from the same batch of SWCNTs. Data consistency was also obtained by comparing the results of temperature dependence measurements with those of magnetic field dependence. There was very good agreement between the two sets of data in our measurements; experimental uncertainties were better than 1%. With the exception of the 0.1% nanocomposite sample which had very small magnitude of magnetization (in the order of 10^{-5} emu), magnetization measurements had less than 0.5% experimental error.

The fabrication methodology of the composites was described and discussed in a previous report [1]. For this study, batches of SWCNTs from various sources were used for fabricating the nanocomposites. The 0.1% nanocomposites employed batch A SWCNTs, the 0.2%, 1%, 5% and 10% composites used batch B SWCNTs, another 10% sample was fabricated with batch C, and the 20% composite used batch D SWCNTs. Different batches were either obtained from different sources or prepared with a slightly different process than other batches from a single source. The names and characteristics of the samples are summarized in Table 1. Two additional nanotube sources, batch E and batch F, were also examined, although limited quantities of these sources precluded composite material fabrication. It should be noted that batches A-E are HiPCO carbon nanotubes. Iron particles were employed as the catalyst in the process for the growth of these carbon tubes [9-11]. Batch F carbon nanotubes were made by a laser ablation method. Cobalt and Nickel were used as catalysts. Although all samples were subjected to a purification process, low weight percentages of magnetic particles still remained in the samples. Variations in size and concentration of the remaining iron particles were found to result in a large batch to batch variation in the measured magnetic properties of the composite samples. Changes in the magnetic properties of a single nanotube batch as a function of doping concentration in the composite where also observed and may yield important information on the coupling of the tube to the matrix.

Results and Discussion

A. Nanocomposites:

In the following figures and discussions, unless it is indicated otherwise, emu/g represents the magnetization per gram of the SWCNT batch, which includes the mass of SWCNTs and magnetic residues. Fig. 1 shows magnetization curves of the 10% batch C nanocomposite film sample (C1) at temperatures of 50, 100, 200, 300, and 400 K. All of the curves show the typical hysteresis loop of a ferromagnetic material. Both the saturation magnetization and the coercivity increased as temperature decreased. At a magnetic field of 8 kOe, the composite had a magnetization of 0.356 emu per gram of the composite at 400 K; increasing to 0.513 emu per gram at 50 K. Coercivity of the sample was quite significant up to 400 K, measured as 95 and 1055 Oe at temperatures of 400 K and 50 K respectively. The coercivity of batch C bare nanotubes was slightly over a hundred Oersteds at 100 K and had much smaller temperature dependence than that found for the composite sample C1 (912 Oe at 100 K). This increase in coercivity of the SWCNTs upon incorporation into the composite may well be the result of interfacial couplings between the SWCNTs and polymers. The 20% batch D composite sample D1 had similar characteristics to sample C1. Magnetization data of this sample showed an increase in coercivity from approximately 137 Oe at 400 K, up to 1001 Oe at 50 K and 1290 Oe at 10 K.

Fig. 2 shows the field dependent magnetization curves of the B3 composite sample at different temperatures. Contrary to samples C1 and D1, the coercivity of sample B3 (5% SWCNT) was small and the hysteresis loop almost closed up when the sample temperature was at or above 100 K. Taking the measurement uncertainty into account, the coercivity was essentially zero at these temperatures. While the saturation magnetization increased with decreasing temperatures from 400 K to 10 K, measurable coercivity was not present until the sample temperature was lowered below 100 K. Furthermore, a plot of the magnetization vs. magnetic field /temperature for sample B3 (Fig. 3) exhibits overlapping curves, similar to what occurs to a ferromagnet when it is in the superparamagnetic state [12]. Magnetic particles of very fine size are known to exhibit superparamagnetism. In this state, no hysteresis and no coercivity would be observed. Measurements of pristine batch B nanotubes and composite samples of other weight percentage of batch B nanotubes showed a similar magnetic field dependence. The much smaller coercivity of batch B composite samples as compared to samples C1 and D1 is therefore most likely due to a smaller residual catalyst particle size in the batch B nanotubes as compared to batches C and D. As we proceed to analyze the coercivity of other samples, this argument will gain more evidence.

Magnetization of pure polyimide was also measured. Pure polyimide displayed a diamagnetic response at all temperatures. Fig. 4 illustrates the magnetization variation as a function of temperature at a magnetic field of 1 Tesla for sample B2 (1%) and a pure polyimide film. The curve at the bottom is for the pure polyimide film. Subtraction of the magnetization of pure polyimide from the composite sample data (curve in the middle) resulted in magnetization expressed by the curve at the top, which is assumed to be contributed by the batch B SWCNTs embedded in the polyimide. As can be seen, the magnetization increases with decreasing temperature. Unlike the typical saturation magnetization curve of a ferromagnet which follows the molecular field theory [13] and would flatten out as temperature approaches zero degree Kelvin, the magnetization of sample B2 kept increasing. This unusual behavior was observed for all the other nanocomposite samples. Although oxygen gas undergoes a magnetic phase transition [14] from diamagnetic to paramagnetic at 57 K, the transition alone does not account for the increasing trend of the magnetization with decreasing temperature as seen in Fig. 4.

In performing this analysis, a careful consideration of the properties of the initial nanotube batch must be taken into account. Fig. 5 shows the measured magnetization as a function of magnetic field at 100 K for four of the bare SWCNT samples from four different batches. As can be seen, saturation magnetization and coercivity of the CNTs are batch dependent. A CNT nanocomposite synthesized with SWCNTs with large coercivity would be expected to have a greater coercivity than a nanocomposite synthesized from a lower coercivity SWCNT batch. For composites made with SWCNTs from the same batch, however, coercivity exhibits a variation with nanotube weight percentage, but on a much smaller scale. Fig. 6 displays the temperature dependent coercivity of samples B1, B2, B3, B4 and C1. The coercivity of sample C1 decreases almost linearly with increasing temperature and is quite separated from the other four curves. As displayed in Fig. 5, the batch C nanotubes have a larger coercivity than batch B. The batch B nanotubes and their nanocomposites show zero or close to zero coercivity at high temperatures and turn sharply to higher values at low temperatures. Furthermore, for these batch B composites at low temperatures, the coercivity is slightly higher in samples with larger SWCNT weight percentages. One possibility is the occurrence of an aggregation of nanotubes. Uniform dispersion of carbon nanotubes in polymers becomes more difficult as the weight percentage increases. It is possible that the long-range interactions among magnetic particles in the aggregate SWCNTs become effective enough to restrain the motions of the magnetic moments of the particles.

There was iron residue remaining within the SWCNTs in the composites we measured, as verified by electron spin resonance [15] and TGA (thermogravimetric analysis). Assuming that the saturation magnetization of the nanocomposites is contributed only by the iron particles, it is estimated that there are 1.5- 4.1% iron particles in the batches A - D of SWCNTs, based on the SQUID results. Since there is no expectation that carbon nanotubes would have magnetic domain structures, it is plausible to assume the iron particles are responsible for the occurrence of magnetic hysteresis and, thus, the coercivity. However, the coercivity of the studied nanocomposites was found to have a much more significant temperature dependence than that of the bare SWCNTs. Changes in stress conditions on the surface of the iron particles, due to the synthesis with the polyimide, could have resulted in the increased coercivity and the stronger temperature dependence. Reports [4, 8] that describe stress induced band shifts in the

Raman spectrum of SWCNTs in carbon nanotubes composites help to substantiate this theory.

Fine and single-domain magnetic particles are known to possess superparamagnetic behavior above certain temperatures [12]. When the magnetization and/or anisotropic energy of these particles are of the same order of magnitude as the thermal fluctuation energy, magnetic moments become randomly oriented when the external magnetic field is removed. The magnetic remanence is insignificant and no coercivity is observed. When the temperature is lowered thermal energies are reduced and internal magnetic interactions, such as magnetocrystalline interactions, become predominant. As a result, magnetic order will be retained in the sample as the external magnetic field is removed. This effect is most likely what we observed for the batch B SWCNT composites. The samples were in the superparamagnetic state at temperatures above 100 K. There have been discussions on particle size of iron during SWCNT growth [16] and of iron oxide in polymer composites [17]. For the batch B SWCNT composite films, it was difficult to accurately determine the size of the iron particles embedded in the composite. High resolution scanning electron microscopy of the batch B bare SWCNTs, however, did reveal that the iron residues were less than 5 nm in diameter. The magnetic anisotropy energy, E_a, of the iron particles can then be estimated from equation [12],

$$E_a = 25K_BT_B/ \langle V \rangle,$$

where K_B is Boltzmann's constant, T_B is blocking temperature of the superparamagnetism (assumed to be 100 K for the batch B composite samples), and <V> is the volume of the particle. Using 5 nm as the maximum diameter of the iron particles in the polyimides results in an estimate of $E_a \geq 5.27 \times 10^6$ erg/cm^3 and is an order of magnitude larger than that of an isolated iron particle [13]. The anisotropic energy of a nano-size magnetic particle can be contributed by the magnetocrystalline interactions, surface stress of the particle, shape of the particle, and dipole-dipole magnetic interactions between the particles. For the low weight percentage of SWCNT-polyimide samples with spherical magnetic particles, it is believed that the anisotropy energy arises mainly from the magnetocrystalline interactions and is enhanced by the surface stress between particles and polymers [17].

Assuming the magnetization of samples B1, B2, B3, and B4 was saturated at 6 kOe, the saturation magnetization of the samples was obtained after the background magnetization was subtracted. The weight percentages of polyimide in the nanocomposites were known and the diamagnetic susceptibility of the pure polyimide was measured such that the magnetization contributed by the nanotubes/iron particles could be calculated. Fig. 7 has the calculated magnetization in units of emu per gram of the batch B SWCNTs and their nanocomposites as a function of temperature. For each sample, magnetization increased with decreasing temperature of the sample. The rate of increase was smaller for the samples with higher loading of SWCNTs. In addition, at each temperature the magnetization decreased as the weight percentage of SWCNTs increased. This decrease in magnetization of SWCNTs as a result of being synthesized with polyimides was also observed for samples of A1, C1 and D1. Coupling between the nanotubes and polymer was expected [18], and it appears that the coupling not only increased surface stress on iron particles but also affected the magnetic properties of the CNTs. It is possible that the interfacial coupling between carbon nanotubes and polyimides promotes the diamagnetic response from CNTs and reduces the total magnetization of the composite. The reason for the systematic decrease of magnetization with increasing weight percentage, however, is not yet completely clear.

B. Magnetic responses of SWCNTs

SWCNTs can have large paramagnetic or diamagnetic responses to an applied magnetic field [2-3]. The predominant role of the residual iron particles made it difficult to directly measure the magnetic response from the nanotubes in the batches A-D of SWCNTs and their composite film samples. For the SWCNT samples with very low percentages of magnetic particles, however, temperature dependent magnetization did exhibit some intriguing results.

Magnetization of a sample from batch F SWCNTs became saturated and stayed at a constant value when the magnetic field was higher than 6 kOe and the sample temperature was at 300 K. The weight percentage of magnetic residues calculated from the saturation measurements was estimated to be lower than 0.1%. As for the temperature dependent results, Fig. 8 displays the magnetization of the sample at 6 kOe, 8 kOe, and 1 Tesla. Similar to the nanocomposites, magnetization continued to increase as sample temperature was lowered. For each field, magnetization data were collected in a thermal cycle from 320 K to 10 K and back to 320 K. There was thermal hysteresis for the magnetization at temperatures between 200 K and 70K with a smaller magnetization while the sample was being cooled. At room temperature, magnetization is mostly due to the saturated magnetic moments of iron particles and had magnitudes quite close to each other for the three fields. Below 200K, the three sets of curves started to diverge from each other. Measurements had been repeated on samples taken from the same batch and showed the same behavior. The calculated magnetic susceptibilities from the temperature dependent data are quite consistent with those obtained from the magnetic field dependent magnetization results. Similar results were also obtained with the same measurements conducted on another batch (batch E) of SWCNTs, which had approximately 0.3 wt% of iron residues and a much larger coercivity (300 Oe at room temperature). The two batches were in powder form and each had a mass of less than 10 mg. As calculated from the data, the paramagnetic susceptibility increased with decreasing temperatures. For the samples from batch F, the averaged susceptibilities were $3\text{x}10^{-7}$, $1.37\text{x}10^{-5}$, and $5.24\text{x}10^{-5}$ emu/g at 300 K, 100 K, and 10 K, respectively. For Batch E, a much larger susceptibility ($1.3\text{x}10^{-5}$ emu/g) was obtained at 300K, probably due to the larger size of the ferromagnetic particles in this batch. Susceptibilities at low temperatures are close to the values of the batch F SWCNT samples.

Continuous increases in magnetization at high magnetic fields and in magnetic susceptibility when sample temperature is decreased are not usually observed for ferromagnetic particles. Magnetization of the metallic carbon nanotubes induced by the applied magnetic field could potentially contribute additional magnetization to the already saturated magnetization of ferromagnetic catalyst particles. Evidence of such a magnetic response of carbon nanotubes to an applied field has been documented in the literature. Recently, there was a report [19] on the induced magnetism in carbon nanotubes by contacting the nanotubes with magnetic materials. A visualization of dispersed SWCNTs in a polymer matrix via magnetic force microscopy (MFM) has also been reported [20]. Finally, it has been found in the fabrication of CNT electronic devices that the magnetic susceptibility of CNTs can be increased through the use of adhered ferromagnetic nanoparticles [21].

Summary

Magnetization measurements were performed on composite films of various weight percentages of SWCNTs in polyimides. The composites, except those showing superparamagnetic behavior, had enhanced coercivity and stronger temperature dependence of coercivity than the constituent SWCNTs. It was also observed that the saturation magnetization of the composites decreased with increasing weight percentage of SWCNTs. Magnetic properties of the composites were largely affected by magnetic particles which were used as catalysts for the growth of CNTs and remained with the CNTs during processing and synthesis of the nanocomposites. An increase in coercivity of composites over constituent SWCNTs was measured and is believed to be induced by the surface stress in the coupling between the iron particles and the polymer molecules [4, 17]. The same type of coupling between the carbon nanotubes and polymer is believed to be responsible for the increased diamagnetic effects from SWCNTs and resulted in a decrease of magnetization per gram of the SWCNTs with increasing SWCNT weight percentage. Coupling between the carbon nanotubes and the polyimides play an important role in determining the mechanical strength of a nanocomposite. Investigating changes in the magnetic properties of SWCNTs in the polymer matrix may provide a unique tool for the study and characterization of interfacial interactions between the component materials in a nanocomposite. SWCNT batches with low percentage of iron residues showed increasing magnetic susceptibility with decreasing temperature, which may be caused by the contribution from magnetized iron residues coupling with paramagnetic SWCNTs. For the development of magnetic field sensing and magnetic alignment, a greater susceptibility certainly would provide a more effective magnetic response to an applied external magnetic field.

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Table 1. Weight percentage of SWCNTs in the measured polyimide composite samples.

Nanocomposite Sample Name	Weight Percentage of SWCNTs (%)	Source of SWCNTs (HiPCO)	Sample Thickness (μm)
A1	0.1	batch A	58
B1	0.2	batch B	44
B2	1	batch B	69
B3	5	batch B	81
B4	10	batch B	23
C1	10	batch C	46
D1	20	batch D	38

Captions:

FIG. 1. Magnetic field dependent magnetization curves of sample C1 at temperatures of 50 K, 100 K, 200 K, 300 K, and 400 K. The curves exhibit the typical hysteresis behavior of a ferromagnet. The coercivity is still significant up to 400 K.

FIG. 2. Magnetization as a function of magnetic field of sample B3 at constant temperature. Saturation magnetization increases with the decrease of sample temperature. No coercivity is observed at temperatures above 100 K.

FIG. 3. Magnetization as a function of magnetic field/temperature for the sample B3 at constant temperatures. Overlapping of curves could imply that magnetic particles, which are responsible for the magnetization, are of very fine size and have single magnetic domain.

FIG. 4. Temperature dependent magnetization of the 1 wt.% Batch B composite sample (sample B2) at magnetic field of 1 T. The curve (with "♦") at bottom shows the diamagnetic response of pure polyimide. Subtracting the magnetization of the polyimide from the raw data (curve shown in the middle) results in the magnetization (curve at the top with "+") response from the Batch B SWCNTs in the composite.

FIG. 5. Magnetization of different batches of SWCNTs at 100K. Not only the saturation magnetization but also the coercivity varies from batch to batch.

FIG. 6. Temperature dependence of coercivity of samples B1, B2, B3, B4, and C1 (from bottom to top). Magnetic properties of the nanocomposites are mostly determined by their component SWCNTs/iron particles.

FIG. 7. Temperature dependent magnetization of batch B SWCNTs and batch B nanocomposites. Curves from top to the bottom are the magnetization at 6 kOe for the batch B nanotubes (curve with ♦), and composite samples B1 (0.2 wt-■), B2 (1 wt%-x), B3 (5 wt %-▲) and B4 (10 wt-∗) respectively. Contribution of magnetization by per gram of SWCNT batch decreases with increasing weight percentage.

Fig. 8. Temperature dependent magnetization of batch F SWCNTs at fields of 6 kOe, 8 kOe, and 1T. The increase of magnetization at low temperatures could be attributed to the paramagnetism of the SWCNTs.

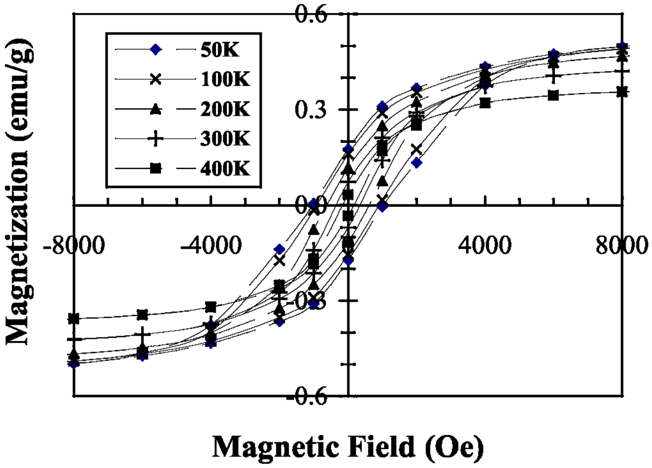


Fig 1. K.J.Sun et al.

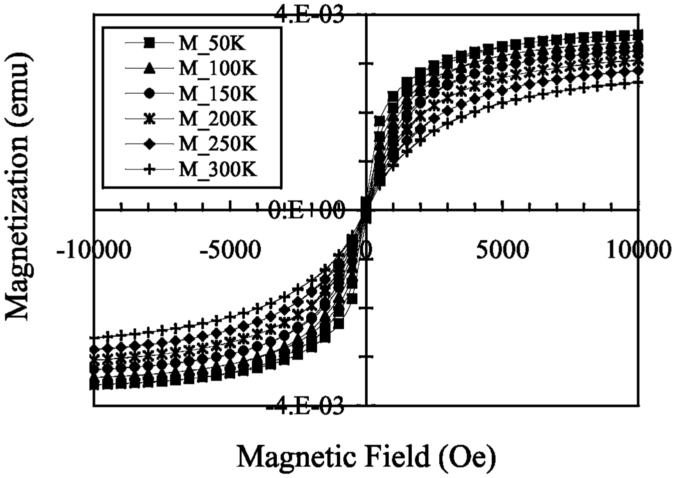
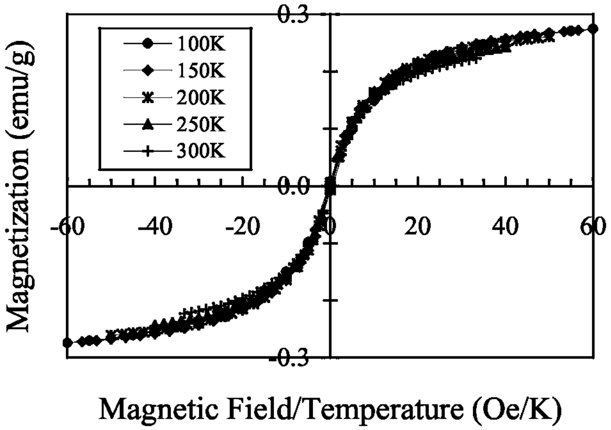


Fig 2. K.J.Sun et al.



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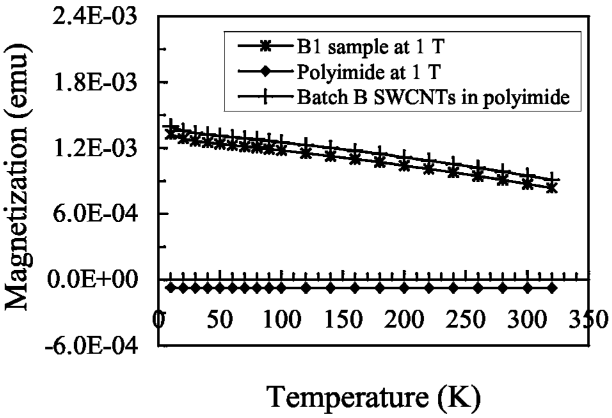

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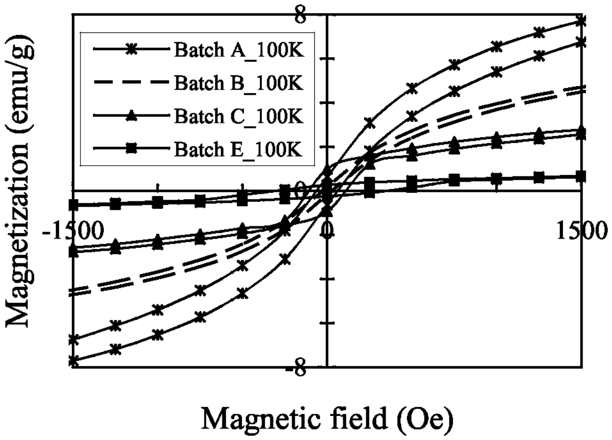

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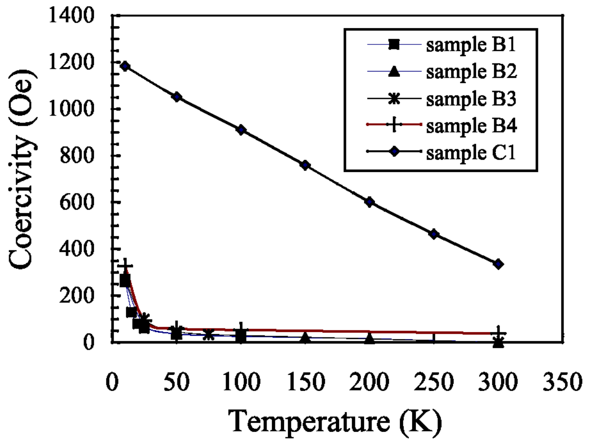

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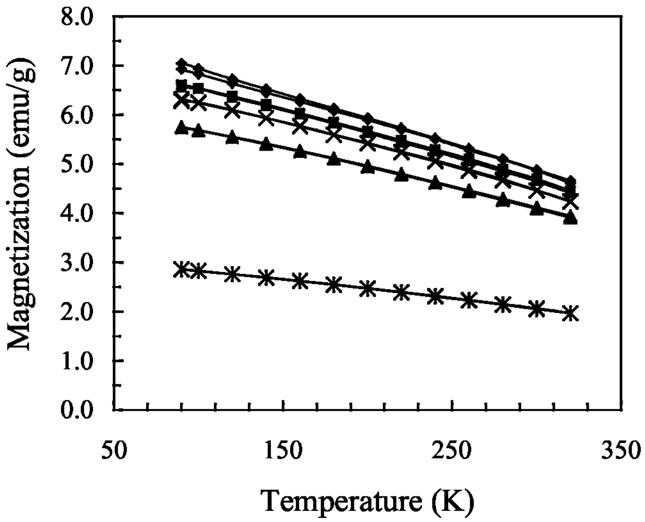

Fig 7. K.J.Sun et al.

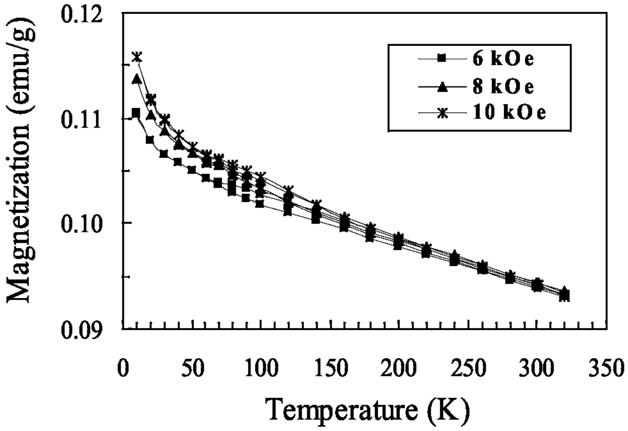

Fig 8. K.J.Sun et al.