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Influence of graphene oxide doping on the morphology and the magnetic properties of $Ni_{0.8}Gd_{0.2}Fe_2O_4$ nanofibers prepared by electrospinning

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Abstract

Nickel-Gadolinium ferrite nanofibers doped with graphene oxide (GO) were prepared for the first time by electrospinning technique. The structure and morphology of the as-obtained composites were characterized by scanning electron microscopy, energy-dispersive X-ray spectroscopy (EDX), transmission electron microscopy, X-ray diffractometer, Fourier transform infrared spectra, and Raman spectroscopy. The magnetic properties were analyzed by Quantum design MPMS-XL magnetometer. A transition from the 'fiber' morphology to 'ribbon' morphology was detected for the samples doped with GO. Also, an increase in the magnetic saturation was found when the GO amount increases. Therefore, doping with GO influences both the morphology and the magnetic properties of the Nickel-Gadolinium ferrite nanofibers prepared by electrospinning. These results provide a promising lead for the future synthesis of a variety of GO doped ferrites nanofiber morphologies with controllable magnetic properties.

Keywords

Nanofibers; Graphene Oxide; Nickel Gadolinium Ferrite; electrospinning.

1. Introduction

Nanostructures have received an important interest, particularly the one-dimensional (1D) morphology nanofibers because of their high surface area, anisotropic shape and potential technological applications in many fields such as ultrahigh density storage, magnetic sensors, spintronic devices, electromagnetic wave absorber and photo catalysis [1-3]. An easy and low cost method for generating 1D nanostructures is the electrospinning technique [4-7]. This technique has been applied to elaborate spinel ferrite nanofibers displaying specific magnetic properties. Spinel ferrite oxides have the general AB_2O_4 structure, in which the A and B cations are distributed in one-eighth of the tetrahedral (A) sites and half of the octahedral (B) sites. The properties of ferrites are strongly dependent on their chemical composition, cation distribution and method of preparation. The inverse spinel type is particularly interesting due to its high magnetocrystalline anisotropy, high saturation magnetization, and unique magnetic structure [8, 9]. As a very known spinel ferrite, the $NiFe_2O_4$ has an inverse spinel structure and is a soft ferrite with lower magnetic coercivity but high electrical resistivity, which makes it an excellent core material for power transformers in electronic and telecommunication applications. Compared to the 3d transition metal ions, the rare-earth lanthanides elements, such as Gadolinium, have large magnetic moments, large magnetocrystalline anisotropy and very large magnetostriction. They can play an important role in the electrical and magnetic properties of the ferrite due to the localized nature of 4f electrons [8, 10, 11].

On the other hand, graphene is the form of carbon that has superior physical properties [12, 13] whilst graphene oxide (GO) is a very welcomed derivative of graphene. As opposed to graphene, GO is hydrophilic [14], so it could easily form a colloidal suspension of sheets that is easily mixed with the electrospun solution [10, 15].

Like in graphene, all kinds of defects can exist in GO. Also, the binding of O-atoms to the carbon network can give rise to magnetic moments [16]. Therefore, it becomes very interesting to study the effect of the incorporation of graphene oxide in the nickel-gadolinium ferrite nanofibers. In fact, several studies about nickel-graphene prepared by the solvothermal and hydrothermal route showed an enhancement of the structural and magnetic properties of the ferrites [1, 3, 8, 9].

However, to our knowledge no literature is found for any study about nickel-gadolinium ferrite doped by graphene oxide by electrospinning. Therefore, in this study, the structural and magnetic properties of the electrospun nickel-gadolinium nanofibers will be investigated while varying the GO amount.

2. Materials and methods

2.1. Synthesis of Graphene oxide

GO was synthesized using an improved Hummers method as reported by Marcano *et al.*[17, 18], 3 g of graphite powder (SigmaAldrich, # 282863, powder) and 18 g of potassium permanganate (Alfa Aesar, # 30743) has been added slowly in a mixture of 40 mL of phosphoric acid (Sigma-Aldrich, # 30417) and 360 mL of sulfuric acid (ACS reagent, 95.0–98.0%). The solution was then heated to 50 ºC and stirred for 18 hours. After cooling to room temperature, the solution was poured onto ice (400 mL) with 3 mL of H_2O_2 (30 weight percentage in H_2O, ACS reagent). The solution was then filtered. The filtrate was centrifuged (6000 rpm for 5 min) and the supernatant was decanted away. The mixture was then purified using the protocol of sifting, filtering, centrifugation, decanting with multiple washes followed the procedure described elsewhere [18]. The final solution was vacuum filtered and the solid was placed in an oven for 12 hours to obtain the graphene oxide (GO).

2.2. Synthesis of $Ni_{0.8}Gd_{0.2}Fe_2O_4$/GO nanofibers

The first step consists of dispersing different weight amounts (15 mg, 30 mg and 40 mg) of GO in 2 ml of Deionized water by ultrasonic bath for 25 minutes. The amounts correspond to 6%, 12% and 15% doping of GO on the Ni-Gd ($m_{GO}/m_{(Ni+Gd)}$). Then, 0.5g of nitrate iron (Iron III) nine times hydrated ($Fe(NO_3)_3.9H_2O$, MW ~ 404, assay > 98%, Aldrich), 0.123 g of nickel acetate (Nickel II) four times hydrated ($Ni(CH_3COO)_2$ $.4H_2O$, MW ~ 248.84, Aldrich) and 56 mg of nitrate gadolinium (Gadolinium III) six times hydrated ($Gd(NO_3)_3$ $.6H_2O$, MW ~ 451.36, Aldrich) are added to the previous solution under vigorous stirring for half hour until a complete dispersion of the precursors. In parallel, 0.9 g of PVP (poly (vinyl pyrrolidone), $(C_6H_9NO)_x$, M.W ~ 1 300 000, Aldrich) is dissolved in 7.5 ml of absolute ethanol (EtOH). The obtained polymer solution is added along with 1 mL of acetic acid to the initial solution.

This final solution is filled in a syringe headed by a metallic needle. It is ejected under an applied electric field between the needle tip and the metallic collector. The voltage was maintained at 25 kV. The distance between the needle tip and the collector was kept at 10 cm. The flow rate was 1 mL/h. After 4 hours of spinning, the nanofibers were collected, and then calcined at 600 ^{o}C for 3 hours with an annealing rate of 1^{o}C/min. Table 1 summarizes the names of the fibers prepared with different GO weight amounts after thermal treatment.

Table 1: Samples ($Ni_{0.8}Gd_{0.2}Fe_2O_4$) prepared with different weight amounts of GO. Samples denoted A are annealed under air

Sample's name	Mass of GO (mg)	Doping rate (%)
A0	0	0
A1	15	6
A2	30	12
A3	40	14

2.3. Physical and chemical characterizations

Scanning electron microscopy was used to analyze the structure and shape of the samples (FESEM, Hitachi 4800, Japan). EDX measurements were done using a Zeiss EVO ED15 microscope coupled with an oxford X-MaxN EDX detector. The structural properties have been studied by transmission electron microscopy (TEM PHILIPS-CM 20) and Raman spectroscopy, functioning at 532 nm wavelength (HORIBA Xplora). The crystal structure of the nickel gadolinium ferrite doped by oxide graphene was investigated using PAN alytical Xpert-PRO diffractometer equipped with an accelerator detector with Ni-filtered Cu Kα radiation (0.15406 nm) at 40 kV and 20 mA. Infra-red characterization was done by Nicolet 370 FTIR spectrometer using an ATR system. The magnetic characteristics were analyzed by Quantum design MPMS-XL magnetometer.

3. Results and discussion

Scanning Electron Microscopy was used to study the structure of the Nickel-Gadolinium GO doped nanofibers. As shown in Fig. 1, the doping effect on the samples can be observed through the change of morphology. While maintaining the electrospinning parameters intact, the samples' shape is changing from fiber to ribbon-like shape. In fact, the flat form is clearly observed in figures 1.C and 1.D corresponding respectively to a doping rate of 12% and 14%. The sample A1 prepared with a doping rate of 6% (Fig 1.b) shows a mix of fibers and ribbons while the undoped sample A0 is only composed of fibers. The diameter and width of the fibers are given as a mean value (± standard deviation) after measuring 100 random nanofibers using the ImageJ software. The results are given as follows (89 ± 4) nm and (126 ± 4) nm for samples A0 and A1 respectively. Whilst for A2 and A3, an average width of (402 ± 72) nm and (467 ± 58) nm were found. EDX was used to study the chemical compostion of the samples. The EDX data reported in Table 2 of $Ni_{0.8}Gd_{0.2}Fe_2O_4$ and doped Nickel-Gadolinium ferrites shows that the nanofibers are formed without impurities. The atomic ratio is around 1:4 for Gadolinium and Nickel, 1:2 for (Nickel-Gadolinium) and Iron. Also given by table 2, the percentage of carbon in increased after the addition of GO. This result confirms the insertion of GO within the nanofibers. It may be the reason of the conversion of the morpholgy from nanofibers to ribbons observed by SEM. Further investigations on the GO effect on the morphology should be performed in the future.

Table 2: EDX results for different ferrites samples

Atomic percentage					
Sample	Ni	Gd	Fe	O	C
A0	16.29	4.44	33.79	39.80	5.67
A1	16.37	4.00	30.31	41.36	7.97
A2	15.84	3.28	29.12	44.71	9.10
A3	11.41	3.16	27.25	33.79	24.39

The nature of the fibers was further studied by Raman spectroscopy (Fig. 2). The nickel ferrites crystallize as cubic spinel in the space group Fd3m where group theory based calculations result in five Raman active bands namely $A_{1g}+E_g+3\ T_{2g}$ [19, 20]. Raman peaks over the region of 660-720 cm^{-1} represent the modes of tetrahedral group (T-site) involving symmetric stretching of oxygen atoms with respect to metal ion and those in the 460-660 cm^{-1} region correspond to the modes of octahedral group (O- site) related metallic ions involved in octahedral void of ferrites [21, 22]. The spectrum shown in Figure 2 insures the inverse spinel structure of all the samples. In fact, Raman peaks at 198.7 cm^{-1}, 330.9 cm^{-1}, 482.9 cm^{-1}, 571.4 cm^{-1} and

696.1 cm^{-1} correspond to the five active Raman modes T_{2g}, E_g, $2T_{2g}$ and A_{1g} respectively [23, 24]. Thus, it announces the insertion of the Gadolinium in the Nickel ferrites structure without inducing any change.

To confirm the Raman results, a crystal phase analysis was performed. The X-ray diffraction patterns of pure and doped $Ni_{0.8}Gd_{0.2}Fe_2O_4$ are shown in Figure 3. The samples demonstrate high crystallization. They show the characteristics peaks of ferrite material with the most intense peak (311) confirming the formation of cubic spinel structure [25]. Crystal size is calculated using the Debye-Scherrer's equation [26]. They are found to be 9.81, 9.94, 10.5 and 9.63 for samples A0, A1, A2 and A3 respectively. It seems there is no significant influence of the GO doping on the crystal size.

Fig. 4 show the TEM images of samples A0 and A3. They reveal the improvement of the structure with higher doping amounts of GO. In fact, more tidy crystal chains can be distinguished. Besides, the fiber mophology is well observed for sample A0 while the ribbon shape is assigned to the doped sample A3.

FT-IR characterization was used for the Nickel gadolinium to show the retained bonds after calcination for 3 hours at 600°C. The FT-IR spectra of the calcined samples are shown in Figure 5. The broad band observed at 3600−3000 cm^{-1} is ascribed to the presence of hydrogen bonded O-H [27]. The band situated at 1640-1625 cm^{-1} is related to the presence of H_2O bending. A weak band appearing at 1112 cm^{-1} corresponds to the C-OH bonding [28]. The sharp intense band at 1375−1365 cm^{-1} is due to the antisymmetric stretching of interlayer carbonate, which is shifted to a lower frequency compared to the free CO_3^{-2} band (due to strong hydrogen bonding of carbonate with hydroxyl sheets and H_2O molecules) [29].We noticed that the peaks corresponding to the functional carbon group at 1112 and 1392 cm^{-1} are arising with higher doping amounts of GO indicating the retained GO in the fibers after calcination [28].

The magnetic hysteresis measurements of the samples were performed at 300 K under an applied magnetic field sweeping from -50 to 50 kOe. Magnetic hysteresis loops are shown in Figure 6. Table 3 show the coercivity, the remnant magnetization along with the saturation magnetization of the samples. It can be noticed that the Nickel-Gadolinium ferrite fibers exhibit lower magnetic properties than pure Nickel ferrite [11]. This confirms the insertion of the Gadolinium in the spinel structure and its effect on the magnetic and structural properties. The net magnetic moment M_s is M_A - M_B, where the M_A and M_B are the magnetic moments in A and B sites, respectively in the spinel structure. Considering that $Fe^{3+}{}_A$ (Fe^{3+} ions in A sites) and $Fe^{3+}{}_B$ (Fe^{3+} ions in B sites) magnetic moments compensate each other, since they are antiparallel, we expect the magnetic saturation to come from the Ni^{2+} in B sites. But due to the incorporation of Gd^{3+} ions a decrease in the saturation is expected [30-32] because Gd^{3+} prefer to settle in A sites in an opposing magnetic moment direction to B sites, so in an opposition direction with Ni^{2+} magnetic moment.

Consequently, the addition of GO in the fibers seems to have implications on their magnetic properties. In fact, with higher GO amounts, higher magnetic saturation was obtained. This behavior was previously observed in GO doped $CoFe_2O_4$ nanofibers [33]. This was due to the improved crystallization with the doping level. In our case, no significant change in the crystal size was detected by XRD. However, the TEM study showed improvement in the fibers' structure in the doped samples unlike the non-doped ones. The variation of coercivity and magnetic remanence seem to be random and unpredictable. This could be probably due to the formation of aggregates that could alter the magnetic properties [34, 35]. Besides, the magnetization of materials is very sensitive to the morphologies of the samples [36, 37].

In this study, the fibers' conversion into ribbons when the samples are doped with GO may have influenced the values of H_c and M_r.

Table 3: The saturation magnetization, the remnant magnetization and the coercivity of the samples doped with different GO amounts.

Sample	M_s(emu/g)	M_r(emu/g)	H_c (Oe)
A0	29.45	1.21	31.32
A1	32.57	0.82	22.8
A2	36.01	1.93	33.73
A3	36.71	0.52	8.7

Conclusion

In conclusion, $Ni_{0.8}Gd_{0.2}Fe_2O_4$/GO nanofibers were successfully prepared by electrospinning. The GO incorporation seems to affect the morphology of the samples. In fact, a total conversion from the fiber morphology to ribbon morphology was observed starting from a doping rate of 12 %. On the other hand, an increase in the magnetic saturation was detected when the amount of GO increased in the samples. These results are similar to the ones obtained for the cobalt ferrite fibers doped with GO. We believe that GO influences both the morphology and the magnetic properties of the spinel structure. Further studies are to be conducted on the addition of graphene oxide onto fibers for applications in functional materials and textiles.

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Figure captions

Fig.1 SEM images of the annealed samples doped with different amounts of GO: a) A0 ;b) A1; c) A2; d) A3; e) A4

Fig.2 Raman spectra for the pure and doped Gadolinium substituted nickel ferrites

Fig.3 XRD patterns of the different prepared samples (A0, A1, A2 and A3)

Fig.4 TEM and HRTEM images of samples: A0 (a and b); A3 (c and d)

Fig.5 FT-IR of the pure and GO doped Nickel-Gadolinium samples

Fig.6 Magnetic hysteresis loops of the pure and doped Nickel-Gadolinium ferrites