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Size Controlling of $L1_0$-FePt Nanoparticles During High Temperature Annealing on the Surface of Carbon Nanotubes

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Abstract Mono-size FePt nanoparticles with particles size about 2.5 nm have been prepared by polyol method on the surface of carbon nanotubes (CNTs). The CNTs functinalization time and the mass ratio of nanoparticles to CNTs affects on the CNTs surface coating. The as-synthesis nanocomposites have a superparamagnetic behavior with chemically disordered fcc structure at room temperature and they can be transformed into chemically ordered fct structure after thermal annealing above 600 °C. Their magnetic behavior changes from the superparamagnetic to the ferromagnetic with a large coercivity up to 0.83 T for the nanocomposites which annealed at 800 °C. The CNTs surfaces as a substrate prevent the agglomeration of nanoparticles during high temperature annealing and the FePt nanoparticles after annealing at 800 °C have finite size with an average about 10 nm. The structure, composition and magnetic properties of nanocomposite were characterized by X-ray diffraction, transmission electron microscopy, Fourier transform infrared spectroscopy and vibrating sample magnetometer.

Keywords FePt nanoparticles · Carbon nanotubes · Nanocomposites · Ordering parameter · Superparamagnetic · Ferromagnetic · fcc and fct $L1_0$ structure

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1 Introduction

In the recent years, use from metal particles such as iron and its alloys instead metal oxides has increased in ultra high density magnetic recording media. The coercivity of iron particles increases with reducing of their size. Alloying of single-domain iron particles with cobalt, nickel and platinum will increase their magnetic properties because of their shape anisotropy. So, in the recent decades, the capacity of magnetic recording media has increased exponentially and it's exceeded from G bit/in^2 range. For such capacities, the coercivity should be near the one Tesla [1, 2].

To achieve a high density storage media, the size of nanoparticles must be small to the extent that the particles converted from multi-domain magnetic state to single-domain state, because their coercivity will be increased in this state [2–4]. The size of nanoparticle could not be less than a critical size, because the superparamagnetic phenomenon appears in this size and their anisotropy energy (K_uV) becomes less than their thermal energy (k_BT). Due to thermal fluctuations, the orientation of nanoparticles magnetic moment is completely random and makes them unsuitable for using in the magnetic recording media. To solve this problem, the nanoparticle must be fabricated in a critical size until the nanoparticles became single-domain with a thermal stability [5, 6].

Thus in the recent years, researchers are using the materials with maximum thermal stability and high anisotropy energy. Hence, CoPt, FePt with $L1_0$ phase and SmCo nanoparticles are the best candidate for using in the magnetic recording media in T bit/in^2 range [7]. The FePt nanoparticles for their unique properties such as ultra high magnetocrystalline uniaxial anisotropy, high coercivity, high saturation magnetization and chemical stability are

very suitable for using in the magnetic storage, permanent magnets and biomedical applications [8]. Their superparamagnetic size is about 2.4 nm in the room temperature and the suitable size of spherical FePt nanoparticles to be used in a high density magnetic storage is about 4.4 nm [5, 9]. The chemical methods are suitable for synthesis of mono-size nanoparticles with a size between 2 and 10 nm. Also, we can organize regular nanostructures with this method.

Synthesis of mono-size FePt nanoparticles was carried out with Polyol methods including simultaneous reduction of $Fe(acac)_2$ and $Pt(acac)_2$ salts in the presence of Diol reduction agent at the boiling temperatures of solvents such as benzyl ether, phenyl ether, and octyl ether. In this method, some alcohols such as polyethylene glycol (PEG) or ethylene glycol (EG) could be used instead of Diol agent for reduction of metal salts to metal ions. But 1,2-hexadecanediol is the best Diol agent for reduction of precursors mentioned above [10–13].

The as-synthesis FePt nanoparticles have disordered fcc structure with A1 phase and shows superparamagnetic behavior at room temperature. An additional thermal treatment under reduction atmosphere (10 % H_2 + 90 % Ar) at the temperature above 550 °C is required for obtaining the ferromagnetic properties. After annealing at high temperature, the FePt nanoparticles transformed from the disordered chemically A1 phase to the ordered chemically $L1_0$ phase. With respect to the aggregation of nanoparticles at high temperature, the nanoparticles' size increases and makes them unsuitable for use at storage media [7]. Different strategies have been proposed to overcome this problem. From a decade ago, many efforts were performed to obtaining the fct-FePt nanoparticles with uniform distribution after heat treatment. One of these methods is decrease the phase transition temperature of the FePt bimetalic alloy with the addition of a third metal such as Au, Ag and B_2O_3 [14–16]. Rapid thermal annealing is another method for preventing of FePt nanoparticles from aggregation [17]. Deposition of FePt nanoparticles on the surface of polymer wafer (such as FePt-PEI method) [18] or putting an oxide shell around each nanoparticle (core–shell method) are other methods for avoiding of FePt nanoparticles agglomeration [19–21].

Carbon nanotubes (CNTs) have great attention because of their electrical, optical and mechanical properties. The CNTs have high thermal stability and they can be used as support and substrate. The carbon nanotubes have more advantages than conventional supportive materials and create a large area for reaction because of their conventional crystal structure with more conductivity, low impurities and three-dimensional structures. The CNTs have high chemical and thermal stability. So, the nanotubes are suitable for using as a substrate [22–24].

For growth of nanoparticle on the surface of CNTs, they must be purified and functionalized. For functionalization of CNTs, they should be first oxidized at high temperature (500 °C) and then oxidized with a mixture of HCl, HNO_3 and H_2SO_4 acids [25, 26]. These acids can be cause the formation of different functional groups such as hydroxyl (–OH), carbonyl (–C=O) and carboxyl (–COOH) on the surface of the nanotubes. The sites of this functional group are an appropriate place for growth of FePt nanoparticles in the reaction media. In this paper, before the growth of FePt nanoparticles on the surface of nanotubes, the outer surface of CNTs must be functionalized and finally the resulting nanocomposite must be annealed at high temperature in order to phase transition.

The morphology of nanotubes and their coating with FePt nanoparticles during synthesis process and agglomeration of FePt nanoparticles and their structure and magnetic properties after annealing treatment were studied with FE-SEM, TEM, XRD and VSM analysis.

2 Experimental Details

2.1 Synthesis of FePt/CNT Nanocomposites

In this work, the pure multi wall carbon nanotube (with a diameter between 20 and 30 nm and length about 5–10 μm) prepared with chemical vapour deposition (CVD) method were immersed in a mixture of HCl: HNO_3: H_2SO_4 (3 M) acids with the ratio of 1:1:3 for functionalization of the CNTs surface with carboxylic functional group [27]. The resulted solution was stirred (1600 rpm) at 90 °C for 4 h. Oxidized nanotubes were washed several times with distilled water until its PH was reached to 7 and finally they were dried under N_2 atmosphere at 150 °C for 12 h.

For synthesis of FePt nanoparticles on the surface of functionalized CNTs, they were immersed in 40 mL phenyl ether and ultrasound was performed for 20 min to obtain a uniform dispersion of CNTs in the reaction soloution. $Pt(acac)_2$ (0.5 mmol) and $Fe(acac)_3$ (0.8 mmol) salts and also 1,2-hexadecanediol (5 mmol) as reducing agent were added into above solution. After flowing of N_2 gas for 20 min under stirring (1000 rpm), the solution temperature was increased to 100 °C during 20 min to starting the reduction of precursors and nucleation of FePt clusters. The temperature of solution was increased to the boiling point of phenyl ether (259 °C) for 90 min and kept at this temperature for 30 min. Finally, the solution temperature becomes cool down to room temperature.

For washing the products: first, 40 ml Ethanol was added to the mixture and it was stirred for 20 min at room temperature. After 12 h, the black products were separated

with filtering and centrifugation (8000 rpm, 10 min). After separating Phenyl Ether and other impurities, the black discarded was dispersed in Hexane and stirred it for 10 min. Then, the black products were separated with centrifugation. This process was repeated for several times until removing all impurities. Finally, they were dried at 200 °C under (10 % H_2 + 90 % Ar) flow for 12 h.

2.2 Characterization of Nanocomposite

Synthesized nanocomposite before and after annealing were investigated with TEM (LEO-912 AB) and FE-SEM (HITACHI S-1460) for determination of purity, morphology and size of FePt nanoparticles. To study the functionalization of nanotubes, FT-IR spectrum (Thermo Nicolet system) was used. To determination of structure properties, the XRD measurement was performed on the as-synthesis and annealed FePt nanoparticles (X'Pert MPD Philips). The coercivity and magnetic properties of FePt/CNT samples were measured using a VSM analysis (Lake-Shore model 7400). The annealing of FePt/CNTs nanocomposite was carried in tube furnace under reduction atmosphere (%90 Ar + %10 H_2) at the several temperatures.

3 Results and Discussion

Figure 1 shows the FT-IR spectrum of raw CNTs and functionalized CNTs for different time. Figure 1a denotes the CNTs that are not functionalized. Since, there's no peak relating to carboxylic functional groups. Figure 1b–d related to the CNTs, which they were functionalized for 1, 2 and 4 h, respectively. According to the figures, broad peak at 3423 cm^{-1} relates to O–H bonds and the one at about 1633 cm^{-1} relates to C=O bonds that shows the carboxylic functional group on the CNTs.

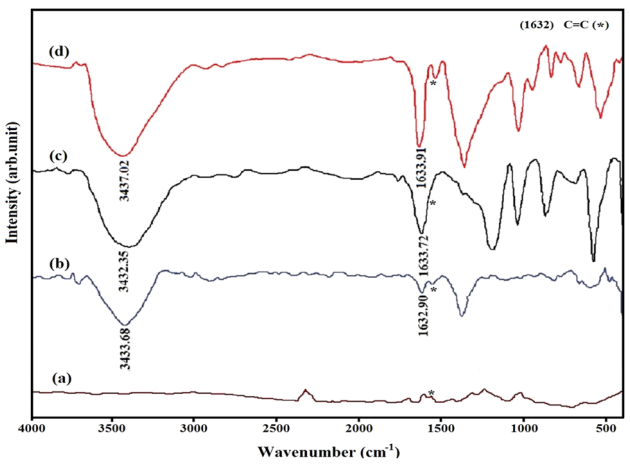

Fig. 1 FT-IR spectrum related to CNTs: *a* Non functionalized and after functionalization for *b* 1 h, *c* 2 h, and *d* 4 h

According to FT-IR analysis, a lot of active sites are made on the surface of CNTs that are useful for nucleation and growth of FePt nanoparticles. The peaks intensity of carboxylic group increases with increasing of functionlization time, which shows great number of active sites on the surface of CNTs. Therefore, the quality of functionalization can be significantly improved with increasing the reaction time.

Figure 2 shows TEM images and histograms of synthesized FePt nanoparticles on the CNTs' surface that are functionalized for 1, 2 and 4 h [28], respectively. The black spots on the surface of CNTs are matched to FePt nanoparticles. By comparing panels (a), (b) and (c) in Fig. 2, we find that the average size (about 2.5 nm) of FePt nanoparticles will not change with increasing the CNTs functionalization time. The distribution of FePt nanoparticles size is very narrow in each plan so, their size is very uniform. But, the size of FePt nanoparticle synthesized without the presence of the CNTs is about 4 nm [29].

But, the coating percent of CNTs surface is very different and improves with increasing the functionalization time at the same reaction conditions. The average size of FePt nanoparticles is the same, about 2.47, 2.5, and 2.59 nm, respectively. Therefore, the number of actives site on the surface of the CNTs does not affect on nanoparticles size. But, the coating of CNTs surface enhanced with increasing the functionalization time which is entirely compatible with the FT-IR results. With increasing the functionalization time more than 4 h, the acids destroy the CNT walls and they had been unsuitable for use as a substrate.The values of nanoparticle size and coating of different planes represented at Table 1. According to obtained results, the suitable time for functionalization of CNTs is 4 h.

Another parameter which investigated in this paper is mass ratio of FePt nanoparticles to CNTs (FePt:MWCNT). Figure 3 shows TEM images of nanocomposite by different mass ratio (1:5, 1:4, 1:3, 1:2 and 1:1) and all of them were functionalized for 4 h. As can be seen, the coating of CNTs significantly increased with increasing the mass ratio of nanoparticles to CNTs. So, at the mass ratio of 1:1 the surface of CNTs is completely covered by nanoparticles and the exact size of nanoparticles is not measurable, because the nanoparticles have overlaps with each other. But, at the others mass ratios, the average size of nanoparticles increased with improves of their coating.

The values of nanoparticles size and coating percent of CNTs are presented at Table 2. The number of Fe and Pt ions enhanced in the reaction media with the increasing of mass ratio and more particles will begin to grow on the surface of the nanotubes. So, the more surface of CNTs are

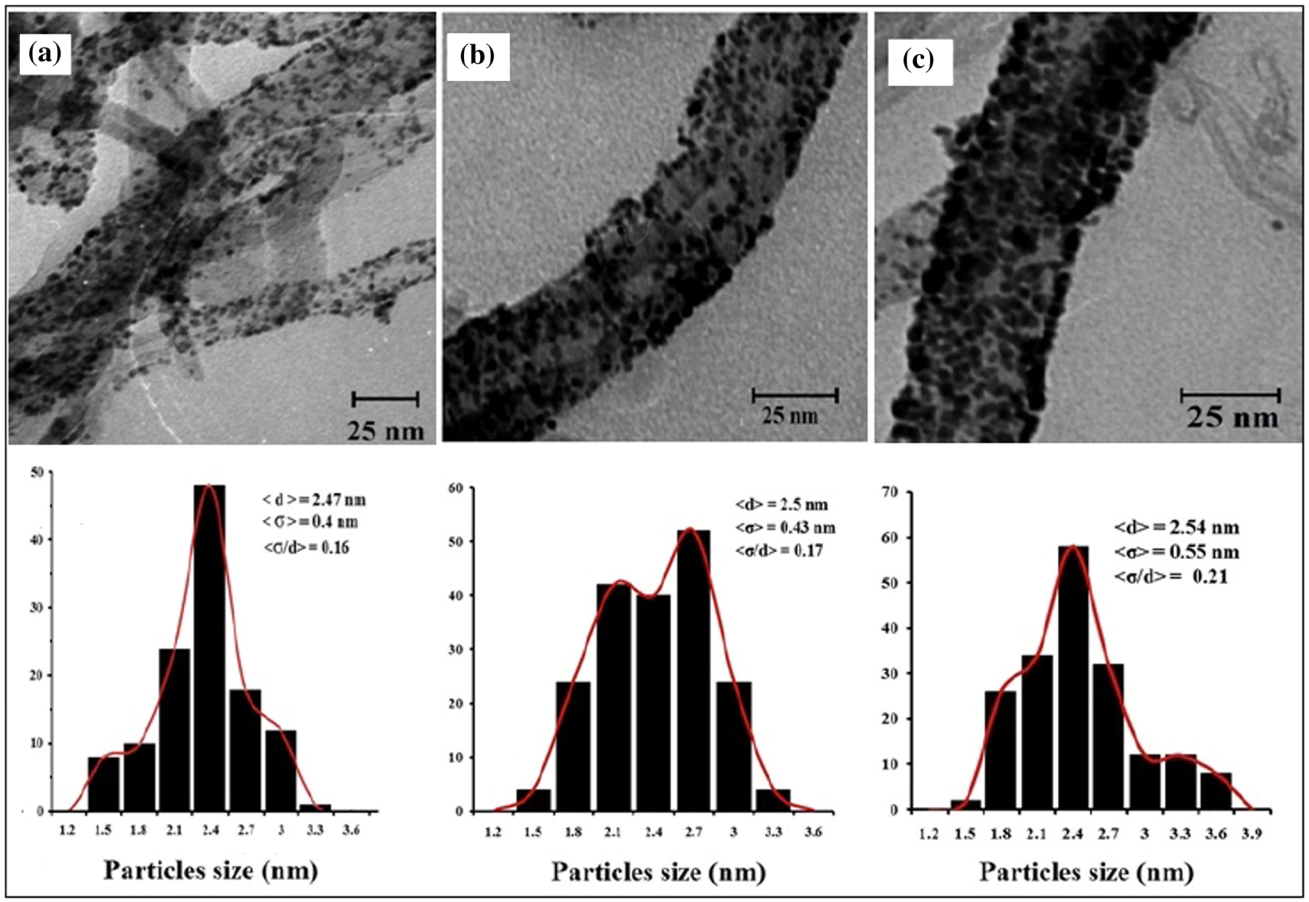

Fig. 2 TEM images and histograms of FePt/CNTs nanocomposite prepared with functinalized CNTs for: **a** 1 h, **b** 2 h, and **c** 4 h

Table 1 The values of nanoparticle size and coating of CNTs that functionalized for different time

Functional time (h)	Nanoparticle size (nm)	Standard deviation (nm)	Coating (%)
1	2.47	0.4	28
2	2.5	0.43	39
4	2.59	0.55	56

coated by FePt nanoparticles. The distance between the nanoparticles decreased and probability of nanoparicles collisions increases during growth process. Therefore, the nanoparticles size was increased.

The XRD patterns of as-synthesis FePt/CNTs nanocomposite and after annealing at different temperature for 2 h under reduction atmosphere (Ar 90 % $+H_2$ 10 %) are presented in Fig. 4. The mass ratio of used FePt/.CNTs nanocomposites is equal with 1:2. According to Fig. 4a, all peaks ((1 1 1), (2 0 0) and (2 2 0)) related to fcc structure of as-synthesis FePt nanoparticles. In addition, the (0 0 2) peak at $2\theta = 25.48$ related to graphite structure of CNTs. After annealing at 500 °C (Fig. 4b), the peaks of (1 1 0), (0 0 1) and (1 1 2) are appears and the position of (1 1 1) peak shift to larger angles that denote a change in the structure of FePt nanoparticle from fcc to fct structure.

But, $L1_0$ phase of nanoparticles has not been formed at this temperature, because the separation of peaks (2 0 0) and (2 2 0) is not observed and complete separation of Fe and Pt crystal planes has not been occurred. The separation of (2 0 0) and (2 2 0) peaks has been appeared after annealing at 600 °C. So, the separation of Fe and Pt crystal planes started at this temperature and increased with growth of temperature and the (1 1 2) peak arise at the temperatures above 600 °C. As shown on Fig. 4d, e, the phase transition of nanoparticles to $L1_0$ phase to be more completed with increasing the annealing temperature.

Crystal structure constants for FePt nanoparticles are given by:

$$a = \frac{\lambda\sqrt{h^2 + k^2 + l^2}}{2\sin\theta} \quad (1)$$

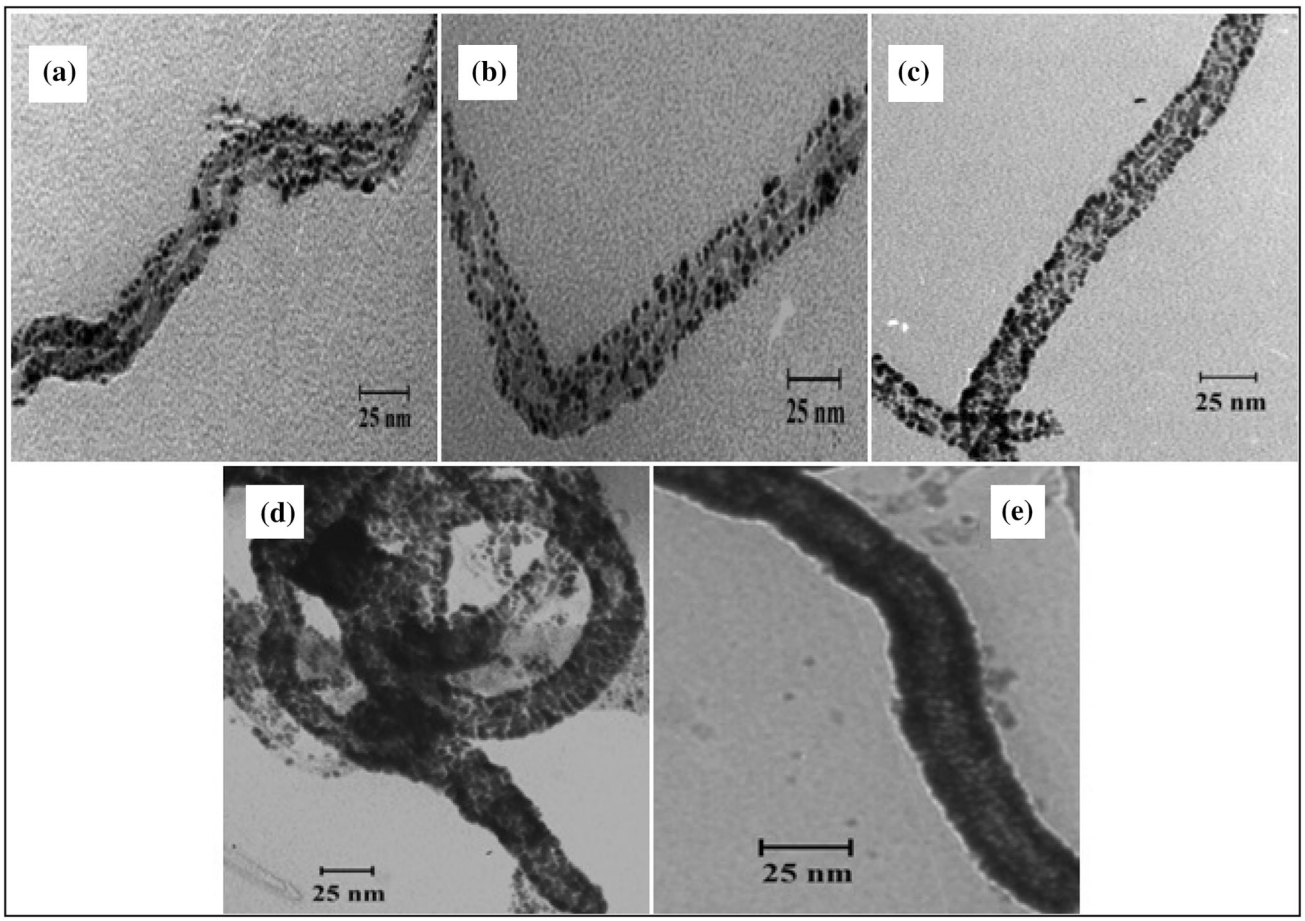

Fig. 3 TEM images of as-synthesis FePt/CNTs nanocomposites with different mass ratio of FePt:MWCNTs; **a** 1:5, **b** 1:4, **c** 1:3, **d** 1:2, and **e** 1:1

Table 2 The values of nanoparticle size and coating of CNTs with different mass ratio

Mass ratio (FePt:MWCNT)	Nanoparticle size (nm)	Standard deviation (nm)	Coating (%)
1:5	2.5	0.48	18
1:4	2.54	0.55	24
1:3	2.6	0.55	56
1:2	4.14	0.71	88
1:1	–	–	100

where $\lambda = 1.54$ Å (wavelength of X-ray), (h k l) are Miller indexes of each peak and θ is position of peak. The (1 1 1) peak is used for calculation of structure constant in fcc structure (a = b = c). But, the (1 0 0) and (1 1 0) peaks are used for obtain the **a** and **c** structure constants in fct structure (a = b ≠ c), respectively. The values of structure constant related FePt nanoparticles with different structure and their average size (calculated with Sherrer' equation $\left(D = \frac{k\lambda}{\beta \cos\theta}\right)$) presented at Table 3.

As shown at the table, the average size of nanoparticle inhanced with increasing of temperature and reaches to 7.5 nm at 800 °C. The possibility of nanoparticle's collision to each other increased with the annealing temperature and metal bonds were created between the different nanoparticles. So, their size will increase after enhancement of annealing temperature.

The ordering parameter (S), which is a measure of the volume fraction of the fct structure, is given by [30]:

$$S^2 \equiv \frac{1 - \left(\frac{c}{a}\right)}{1 - \left(\frac{c}{a}\right)_*} \quad (2)$$

where $\left(\frac{c}{a}\right)$ and $\left(\frac{c}{a}\right)_*$ are the experimental and the theoretical axis ratios for the chemically ordered fct-$L1_0$ phase, respectively. S = 1 means that the FePt nanoparticles are fully in the ordered fct-$L1_0$ phase. Here, $\left(\frac{c}{a}\right)_*$ was

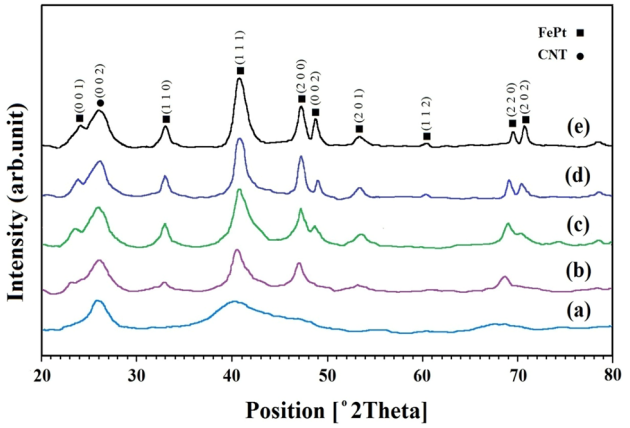

Fig. 4 The XRD patterns of as-synthesis FePt/CNTs nanocomposites and annealed at different temperature: *a* as-synthesis, *b* 500 °C, *c* 600 °C, *d* 700 °C, and *e* 800 °C

determined to be 0.96 according to [31]. The diagram of ordering parameter versus annealing temperature presented at Fig. 5. As shown in figure, the ordering parameter enhances with increasing the annealing temperature and it reaches to 0.9 at 700 °C. Also, it reaches to 0.94 above 800 °C that is very close to 1. It means that most of the FePt nanoparticles have transformed to the fct-$L1_0$ phase after 700 °C annealing and they have good magnetic properties.

Figure 6 shows the room temperature hysteresis loops of as-synthesis and annealed FePt/CNTs nanocomposites at different temperature. The as-synthesis FePt nanoparticles (Fig. 6a) have superparamagnetic behavior at room temperature because of, their chemically disordered fcc structure and low magnetic anisotropy.

But, the FePt nanoparticles after annealing at the temperature above 500 °C have been coercivity, which shows their structure transition. As shown in the Fig. 6b–e, the coercivity of nanoparticle increased with growth of annealing temperature. So, the coercivity of nanoparticle reaches to 7050 G after annealing at 800 °C, even though they have average size about 8 nm. In addition,coercivity, $H_{C,}$ for FePt nanoparticles with L10 phase was about 10 kG at 800 °C [29], while, in this work, it was about 7 kG for FePt/CNT nanocomposite with L10 phase. High coercivity after annealing process, like the XRD results, denotes that

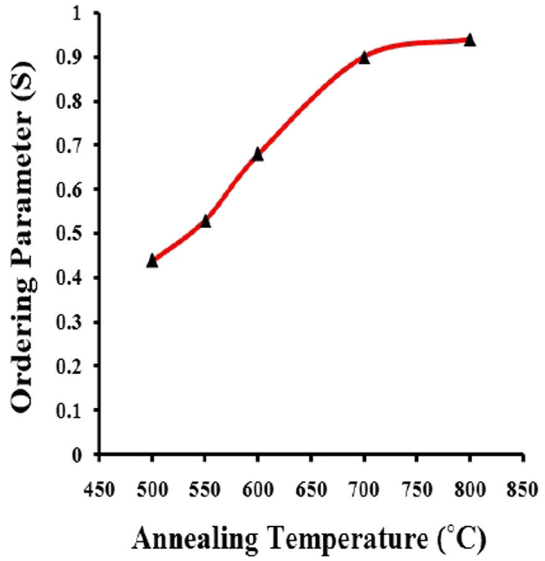

Fig. 5 The diagram of ordering parameter versus annealing temperature

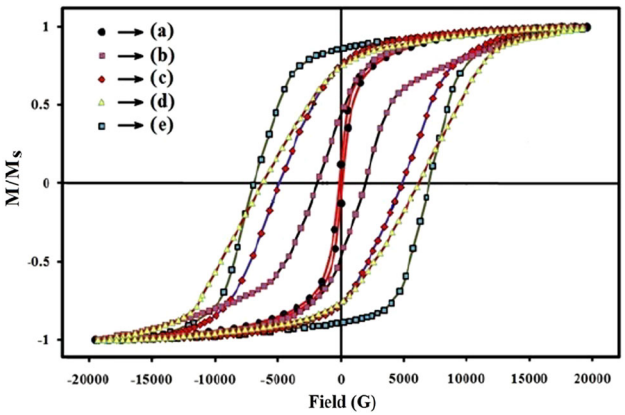

Fig. 6 The room temperature hysteresis loops of FePt/CNT nanocomposites: *a* as-synthesis, after annealing for 2 h in *b* 500 °C, *c* 600 °C, *d* 700 °C, and *e* 800 °C

chemically ordering of $L1_0$ phase improves at temperature higher than 600 °C.

The simultaneously diagram of size and coercivity of nanoparticle versus annealing temperature in room temperature are presented at Fig. 7. According to figure, the

Table 3 The values of structure constant and nanoparticles size related to FePt/CNTs nanocomposite before and after annealing at diferrent temperature

Annealing temperature	2 Theta(111)	**a** in fcc (Å)	**a** in fct(Å)	**c** in fct(Å)	$\frac{c}{a}$	Particle size (nm)
As-synthesis	40.23	3.878 A	–			2.6
500 °C	40.47	–	3.876	3.84	0.99	3.7
600 °C	40.52	–	3.856	3.80	0.985	3.9
700 °C	40.79	–	3.842	3.72	0.968	5.8
800 °C	40.80	–	3.838	3.7	0.964	7.4

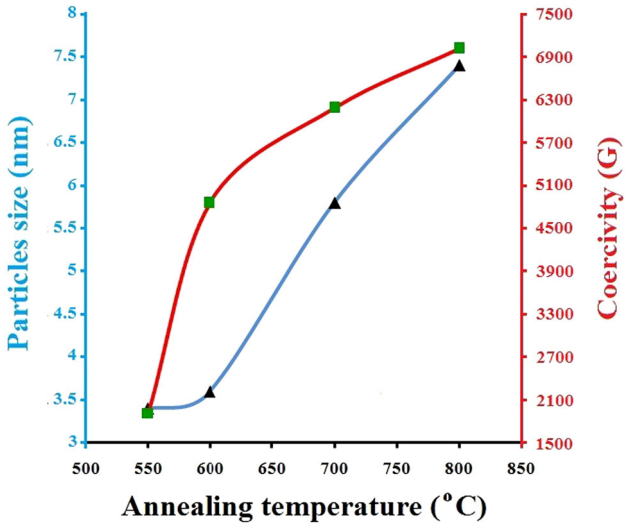

Fig. 7 Simultaneously diagram of size and coercivity of nanoparticle versus annealing temperature in room temperature

coercivity and size of FePt nanoparticles increased with increasing the temperature.

These results are entirely compatible with SW theory [5]. Because according to this theory, the magnetic anisotropy energy of single domain particle is directly related to their volume (k_uV). The anisotropy energy of single-domain particles enhanced with increasing their size and the required magnetic field for reverse the magnetization (H_C) will be increased. So, annealed nanocoposite at 800 °C shows the maximum coercivity, because they have maximum size.

TEM images of FePt/CNT nanocomposites (with mass ratio 1:4) that were annealed at 600 and 700 °C for 2 h have been shown in Fig. 8 [28]. According the figure, the FePt nanoparticles after annealing at 600 and 700 °C have average size about 3.47 nm and 5.6 nm, respectively. It is fully compatible with the XRD results. But, annealed FePt nanoparticles without the presence of CNTs shows very heterogeneous structure comprising particles with sizes ranging from 5 to 100 nm [28]. So, the CNTs as a suitable substrate for growth of FePt nanoparticles can be prevent them from agglomeration and coalescence during the annealing process.

Figure 9 shows, TEM images of nanocomposite with different mass ratio (1:4, 1:3 and 1:2) that annealed at 700 °C for 2 h. With increasing the mass ratio and the coating of nanotubes at the same annealing temperature the average size of nanoparticle enhanced and their size are about 5.6, 7.9, and 10.5 nm for mass ratio 1:4, 1:3 and 1:2, respectively. Because, the distance of nanoparticles on the surface of nanotube decreases with increasing of nanoparticles mass, and the probability of a collision between the nanoparticle and establish of metal bond increases. So, the nanoparticles are aggregated to each other and their size is increased.

The hysteresis loops of nanocomposites in room temperature with different mass ratio after annealing at 700 °C for 2 h have been shown in Fig. 10. These nanocomposites with mass ratio 1:4, 1:3 and 1:2 have coercivity about 6340, 7180, and 8340 G, respectively. Hence, the coercivity of nanoparticles enhanced with increasing of mass ratio nanoparticles to CNTs. Because the size of nanoparticles after annealing at specified temperature enhanced with increase of their number on the surface of nanotubes. So that, the coercivity of FePt nanoparticles after annealing process reaches to 0.83 T for the nanocomposite with mass ratio 1:2, while their size is about 10 nm.

The small size nanoparticles ($\approx$10 nm) with the coercivity from order Tesla are very good for building the magnetic recording media with a capacity about 1 Tbit/in^2. Also, we can use from coated nanotube with $L1_0$ phase FePt nanoparticles as micro or nano magnetic storage.

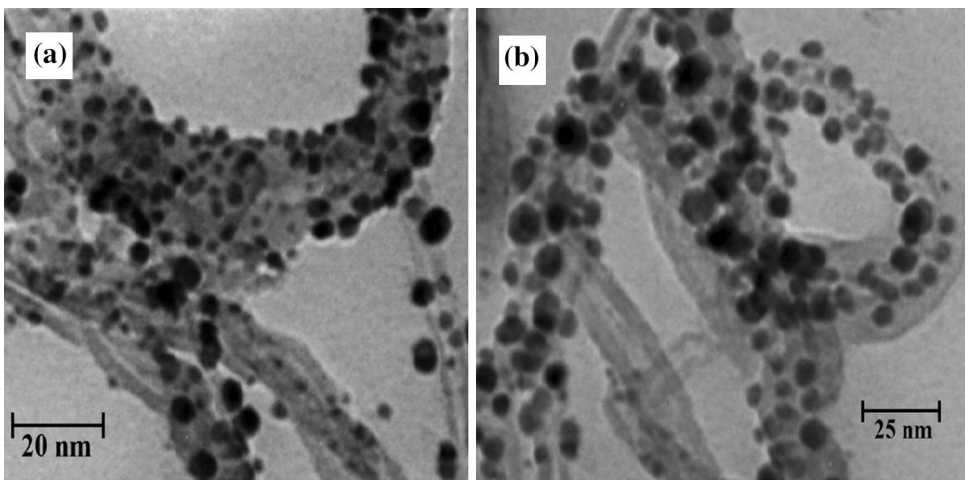

Fig. 8 TEM images of FePt/CNTs nanocomposites that annealed for 2 h at: **a** 600 °C and **b** 700 °C

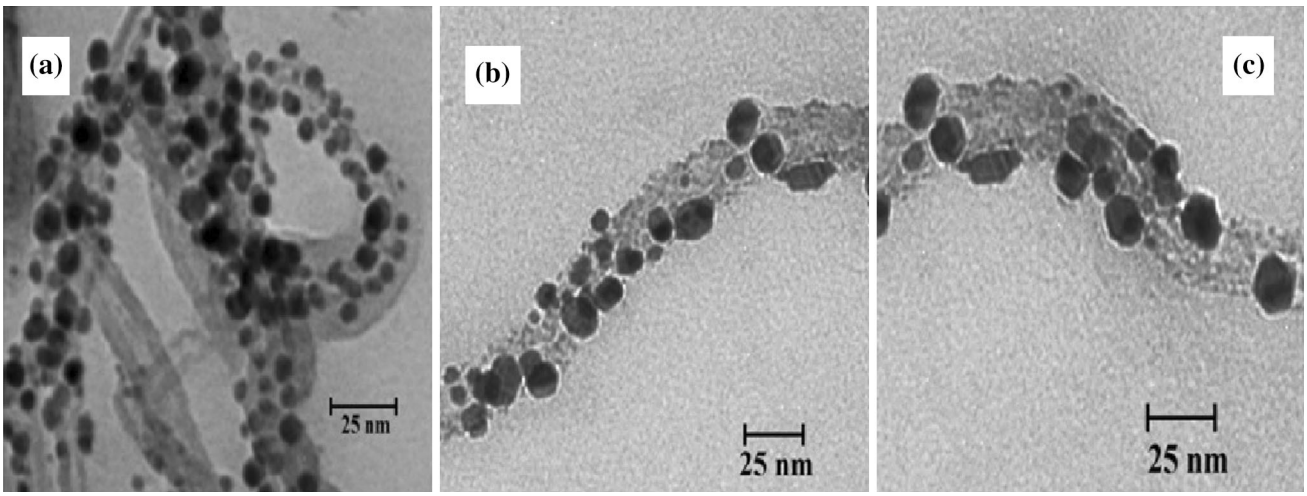

Fig. 9 TEM images of FePt/CNTs nanocomposites that annealed at 700 °C for 2 h with mass ratio: **a** 1:4, **b** 1:3, and **c** 1:2

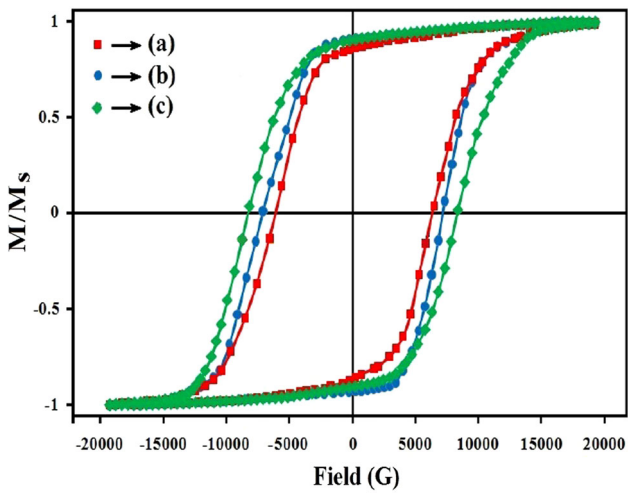

Fig. 10 Hysteresis loops of FePt/CNTs nanocomposites in room temperature that annealed at 700 °C for 2 h with mass ratio: *a* 1:4, *b* 1:3, and *c* 1:2

4 Conclusions

In this paper, the mono-size FePt nanoparticles with an average size about 2.5 nm on the surfaces of CNTs were synthesized by a Polyol process, without any surfactant agent. Their coating percent was related to functionaliztion time and mass ratio of nanoparticles to nanotube. The optimum situation in coating of CNTs occurred in the functinaliztation for 4 h and the 1:2 mass ratios. The resulted FePt nanoparticles from reaction media have a chemically disordered fcc structure and they have super-paramagnetic behavior at room temperature. The phase transition of nanoparticles from disordered A1 phase to the ordered fct-$L1_0$ phase occurred with annealing the nanocomposite at a temperature above 600 °C. The CNTs as a suitable substrate for growth of FePt nanoparticles used to prevent agglomeration of nanoparticles during the annealing treatment. Resulted nanoparticles have finite size less than 10 nm even after annealing in high temperature (800 °C). According the different coating situation, they obtained different sizes after annealing process and their coercivity were various amounts. Their maximum coercivity reaches to 0.83 T for nanoparticle with the size about 10 nm. Therefore, they are suggesting for magnetic recording media with a capacity about T bit/in^2.

References

1. K.J. Klabunde, *Nanoscale Materials in Chemistry* (Wiley, New York, 2001)
2. S. Liu, D. Tao, L. Zhang, Powder Technol. **217**, 502 (1999)
3. M.P. Morales, C.J. Serna, F. Bodker, S. Morup, J. Phys. **9**, 5461 (1997)
4. Y. Jun, J. Seo, J. Cheon, Account **41**, 179 (2008)
5. B.D. Cullity, *Introduction to Magnetic Materials* (Addison-Wesley, Readings, 1972)
6. R.C. Handly, *Modern Magnetic Materials* (Wiley, New York, 2000)
7. D. Sellmyer, R. Skomski, *Advanced Magnetic Nanostructures* (Springer, New York, 2006)
8. S.N. Piramanayagam, T.C. Chong, *Developments in Data Storage Materials Perspective* (Wiley, New York, 2010)
9. S. Nicola, *Magnetic Materials: Fundamentals and Device Applications* (Cambridge University Press, Cambridge, 2003)
10. F. Bonet et al., Solid State Ionics **126**, 337 (1999)
11. H. Grisaru, O. Palchik, M.A. Slifkin, A.M. Weiss, A. Gedanken, Ingor. Chem. **42**, 7148 (2003)
12. H. Zeynali, S.A. Sebt, H. Arabi, H. Akbari, J. Cluster Sci. **23**, 1107 (2012)
13. G. Viau, F.F. Vincent, F. Fievet, Solid State Ionics **84**, 259 (1996)
14. S. Kang, J.W. Harrell, D.E. Nikles, Nano Lett. **2**, 1033 (2002)
15. Z. Jia, S. Kang, S. Shi, D.E. Nikles, J.W. Harrell, J. Appl. Phys. **97**, 10J310 (2005)

16. H. Zeng, R. Sabirianov, O. Mryasov, M.L. Yan, K. Cho, D.J. Sellmyer, Phys. Rev. B. **66**, 184425 (2002)
17. K. Yano, V. Nandwana, N. Poudyal, C.B. Rong, J.P. Liu, J. Appl. Phys. **104**, 013918 (2008)
18. D. Weller, A. Moser, IEEE Trans. Magn. **35**, 4423 (1999)
19. Y. Tamada, S. Yamamoto, M. Takano, S. Nasu, T. Ono, Appl. Phys. Lett. **90**, 162509 (2007)
20. L.C. Varand, M. Imaizumi, F.J. Santos, M. Jafelicci, IEEE Trans. Magn. **44**, 4448 (2008)
21. G.S. Chaubey, V. Nandwana, N. Poudyal, C.B. Rong, J.P. Liu, Chem. Mat. **20**, 475 (2008)
22. H. Karimi-Maleh, P. Biparva, M. Hatami, Biosens. Bioelectron. **48**, 270 (2013)
23. Y. Li, R. Yang, J. Am. Chem. Soc. **128**, 8136 (2006)
24. S. Park, B. Kim, Y. Lee, M. Cho, Int. J. Hydrogen Energy **33**, 1706 (2008)
25. A. Peles, C. Van de Walle, Phys. Rev. B **76**, 4101 (2007)
26. R. Zacharia, Y. Kim, A. Kibria, K. Nahm, Chem. Phys. Lett. **412**, 369 (2005)
27. A.G. Osorio, I.C.L. Silveira, V.L. Bueno, C.P. Bergmann, Appl. Surf. Sci. **255**, 2485 (2008)
28. R. Moradi, S.A. Sebt, H. Arabi, M. Mojtahedzadeh Larijani, Appl. Phys. A. **113**, 61 (2013)
29. H. Zeynali, S.A. Sebt, H. Arabi, H. Akbari, S.M. Hosseinpour-Mashkani, K.V. Rao, J. Inorg. Organomet. Polym. **22**, 1314 (2012)
30. Y. Liu et al., Powder Technol. **239**, 217 (2013)
31. S. Iwasaki, IEEE Trans. Magn. **20**, 657 (1984)