Fundamental Journals
IJFPS
International Journal of Fundamental Physical Sciences
ISSN : 2231-8186
Full Length Research Paper
http://fundamentaljournals.org/ijfpss

The effect of reflux process on the size and uniformity of FePt nanoparticles

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(*Received June 2011; Published Sep 2011*)

ABSTRACT

FePt nanoparticles attract great research interest for possible application to ultrahigh density magnetic recording media. In this paper, FePt magnetic nanoparticles were synthesized by superhydride reduction of $FeCl_2$ and $Pt(acac)_2$ at high temperature. Adding superhydride (LiBEt3H) to the phenyl ether solution of $FeCl_2$ and $Pt(acac)_2$ in the presence of oleic acid, oleylamine, and 1,2-hexadecanediol at 200 °C, followed by refluxing at 250 °C, led to monodisperse 3 nm FePt nanoparticles. The effect of reflux process on the size and uniformity of FePt nanoparticles has been investigated. TEM images showed that the size of FePt nanoparticles increase to 4 nm with reflux process and the standard deviation of FePt nanoparticles increase to 10 % which lead to improve the uniformity of FePt nanoparticles. The results of EDS analysis, showed that the Fe shell around FePt nanoparticles increase with increasing reflux time and the composition of FePt nanoparticles gives $Fe_{68}Pt_{32}$ after 10 min reflux..

Key words: Chemical Synthesis, Reflux, FePt Nanoparticles, Uniformity

INTRODUCTION

The study of magnetic nanostructures is attracting much interest from both fundamental and applied point of views. Iron Platinum (FePt) nanoparticles have been a subject of intense study for many years due to their ease of synthesis, chemical stability, and potential applications in high density data storage (Chen , Nikles, 2002). However, as-synthesized nanoparticles exhibit a low Curie temperature, low saturation magnetization, and low coercivity. In order to correct the mentioned variables, the particles must undergo a high-temperature annealing, after which, through solid state diffusion, the Fe and Pt atoms rearrange from the chemically disoriented face-centered cubic (fcc) phase to the highly ordered face-centered tetragonal (fct) phase, in which Fe and Pt atoms are arranged in alternating layers. In the face centered tetragonal phase, the crystals exhibit a high Curie temperature, high saturation magnetization, and high coercivity, making them ideal candidates for data storage applications, with recording density attainments as high as 1 Tbit/in2 estimated. In recent years, L10 type materials have attracted more and more attention for ultra-high-density magnetic recording media (McCurrie, Bolzoni, Weller), which needs smaller particle size and higher anisotropy materials to maintain thermal stability. Chemical synthesis is a better way to fulfill this compared with physical ways since particle size, shape and size distribution can be better controlled in chemical synthesis. Self-assembled FePt nanoparticle arrays have been the subject of much interest due to their potential use as ultrahigh density magnetic storage media (Sun, Murray et al. 2000).

A key advantage of the self-assembly approach is the high degree of uniformity in the grain size and position. In bulk phase FePt, the chemically ordered high magnetocrystalline anisotropy $L1_0$ phase is thermodynamically more stable. In direct synthesis of $L1_0$ phase, FePt nanoparticles has been reported (Jeyadevan, K. Urakawa et al. 2003), but so far this

method leads to poly-disperse particles that cannot self assemble into arrays. Monodisperse FePt nanoparticles as made are FCC, and must be annealed to high temperatures to induce the transformation (650°C and up). Unfortunately this annealing process sinters the particles together, destroying the key advantage over existing magnetic recording media. Therefore, Sintering occurs because the surfactant coating around the particles decomposes at 400 ^{o}C. In this paper, as-synthesis FePt nanoparticles were first synthesized by chemical reduction of $FeCl_2.4H_2O$ and $Pt(acac)_2$ at 250 °C under N_2 atmosphere and then were dispersed in hexane solution after purification according to Sun and coworkers method (Sun, Anders, et al. 2003).The effect of reflux process on the size and uniformity of FePt nanoparticles has been investigated by TEM and EDS analysis.

EXPERIMENTAL DETAILS

FePt nanoparticles with size of 3 nm were synthesis using the synthesis described by Sun et al. [7]. Preparation of the FePt nanoparticles involves the reduction of $Pt(acac)_2$ and $FeCl_2{\cdot}4H_2O$ in phenyl ether solvent in the presence of 1,2-hexadecanediol. Oleic acid and oleylamin surfactants were added to the solvent at 100 ^{o}C as a protective agent, in order to prevent agglomeration and oxidation. By adding LiBEt3H superhydride under a blanket of N_2 at 200 ^{o}C, followed by refluxing, the FePt nanoparticles were formed. The refluxing temperature was fixed at 250 ^{o}C. The black reaction mixture was cooled to room temperature and then combined with ethanol to remove the impurity.

The product was precipitated and separated by centrifugation (8000 rpm, 10 min). Any undissolved material was removed by centrifugation. Then FePt nanoparticles were dispersed in hexane solution in the presence of surfactants. To determine the composition of FePt nanoparticles reflux stage, energy dispersive spectroscopy (EDS) analysis (15 kV) was carried out. The specification of the size and shape of FePt nanoparticles were examined by transmission electron microscopy (TEM) analysis using a Philips EM 208 TEM (100 kV) with a resolution of 200 kX.

RESULTS AND DISCUSSION

Figure 2 show the TEM images of the as-synthesized fcc FePt nanoparticles. Figure 1(a) shows the 3 nm FePt at 245 °C in the beginning og the reflux process. The size of FePt increases to 3.5 nm after 10 min reflux in figure 1(b) and finally, after 30 min the size of FePt nanoparticles reach to 4 nm (Fig. 1(c)). As you can see from TEM images, with increasing reflux time the size of FePt nanopartcles increase. Because the Fe atoms are released from $FeCl_2{\cdot}4H_2O$ precursor and attach to FePt which lead to increase the size of nanoparticles. The results of TEM observations confirm the EDS analysis (Fig. 3)

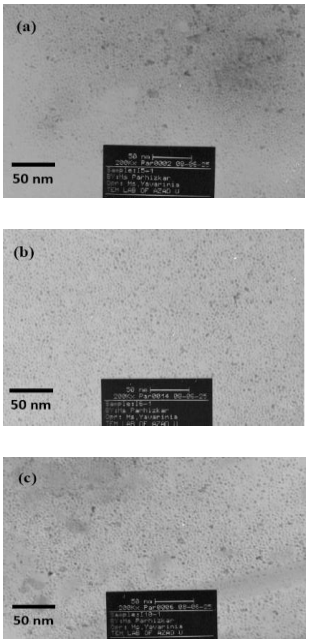

Figure 1. TEM images of as-synthesis FePt nanoparticles at 250 ^{o}C, (a) in the beginning; (b) after10 min. (c) after 30 min reflux.

Figure 3 shows the size measurement of 100 randomly selected particles fitting with a log normal curve. It indicates the 4 nm FePt nanoparticles histogram after 30 min reflux with standard deviation about 10 %.

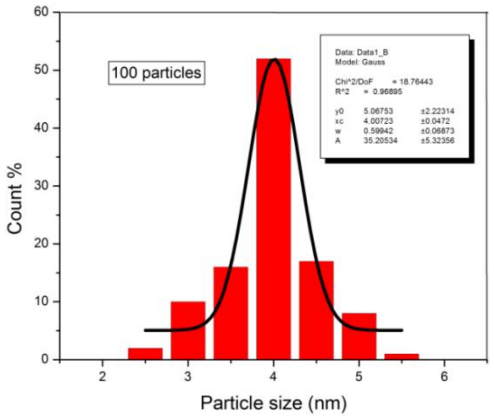

Figure 2. Particle diameter histogram of 4 nm FePt nanoparticles.

Figure 3 shows a characteristic spectrum collected by EDS analysis. By comparing the area under each peak to a set of standards with known element concentrations, the concentration of the elements could be quantified, and the compositions gave $Fe_{60}Pt_{40}$, $Fe_{68}Pt_{32}$ stoichiometery.

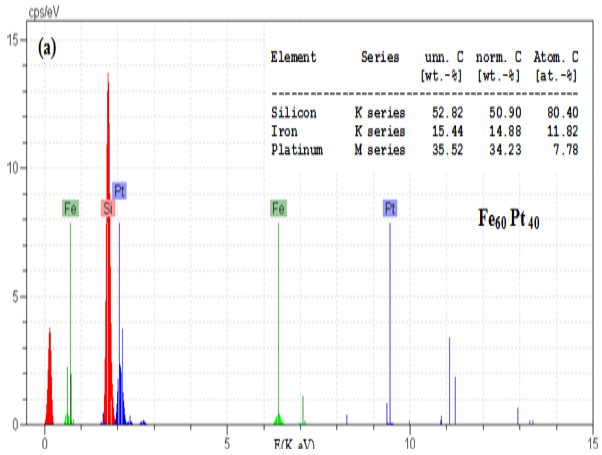

Figure 3. Energy dispersive spectroscopy (EDS) pattern of nanoparticles.

CONCLUSION

FePt nanoparticles were successfully synthesis with mean diameter of 3 nm by polyol method. The study of reflux process on the size and uniformity of FePt was investigated. The TEM results demonstrated that the size of FePt increase to 4 nm with increasing reflux time. The standard deviation of FePt nanoparticles increase to 10 % which indicate the increasing in uniformity of FePt nanoparticles. EDS analysis show that with increasing reflux time the Fe shell around the FePt nanoparticles increase because the monomers are released from precursor. The results show that the compositions of the nanoparticles change from $Fe_{60}Pt_{40}$ to $Fe_{68}Pt_{32}$ after 10 min reflux.

ACKNOWLEDGMENTS

The author is thankful for the financial support of Physics Research Center, Science and Research Campus at Islamic Azad University for analysis and the discussions on the results.

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